Book of Abstracts

Symposium: Injection molding and molds

Keynote (S01-114, Time: Thursday 10:15, Room: St. Gallen)

Pelletization and Injection Molding of Ultra-high Molecular Weight Polyethylene (UHMWPE)

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This presentation reports some of recent developments in pelletization and injection molding of ultra-high molecular weight polyethylene (UHMWPE). The extremely high molecular weight (> 106 g/mol) of UHMWPE has rendered it superior properties such as excellent mechanical and lubricious properties, biocompatibility, chemical resistance, and the highest impact strength among all thermoplastics. However, the ultra-high molecular weight also presents processing challenges due to the high degree of molecular entanglement, lack of melt fluidity, and poor interparticle fusion. Traditionally, complex UHMWPE products have been machined from annealed stocks made by ram extrusion or compression molding, resulting in low throughput and high energy and material consumptions. To overcome these deficiencies, a novel tapered extrusion die with air cooling was developed to extrude and pelletize neat UHMWPE raw powders and convert them into regular pellets suitable for various mass-production polymer processing methods. The combined effect of enhanced molecular chain diffusion, improved consolidation, and lower oxidation using the tapered die are the keys to continuously extruding and pelletizing UHMWPE. The UHMEPE pellets were injection molded with the aids of a supercritical fluid (SCF), which acts as a dual-purposed polymer plasticizer and physical foaming agent that reduce the viscosity and processing temperature of UHMWPE while successfully producing parts with tunable microstructures. Alternatively, the UHMWPE could be processed by injection compression molding that minimizes the injection pressure requirement and improves the packing efficiency and mechanical properties of the UHMWPE molded parts. Finally, a mold insulation layer that delays the rapid cooling of UHMWPE could reduce the shear stress and improve polymer chain "interdiffusion" across the entangled chain bundles, thereby, eliminating the common delamination layer defects within the injection molded UHMWPE parts.

Keynote (S01-120, Time: Thursday 13:45, Room: St. Gallen)

Evaluation of Molding Behaviors and Micro-/Nano-structure Replication of Liquid Silicone Rubber by Injection Molding

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Recently, many types of research attempt to transfer microstructures to the surface of the liquid silicone rubber (LSR) for the improvement of its surface features. In this study, we have investigated the effects of micro/nanostructure on the optical and surface wettability of the LSR in the injection molding process. Moreover, the physical properties of molded products were investigated. The curing time of LSR at various temperatures was predicted by the Kamal model. It was found that the LSR was rapidly cured due to excessive mold temperature, and the replication ratio was significantly reduced. Flow analysis was also carried out using Moldex 3D (CoreTech System). As the first step, the factors responsible for the change in replication properties on a flat plate without micro/nanostructure were investigated in order to analyse the replication behaviour on the micro/nanostructure. Experimental results show that the replication ratio decreased as the mold temperature was increased. This is considered to be due to the rapid curing reaction of the LSR at high mold temperatures, which prevented the LSR from filling the micro/nanostructure. It was also observed that increasing the injection speed improved the surface replication properties for micro/nanostructure. It is considered that the filling of the micro/nanostructure before the curing of the LSR progressed improved surface replication when the injection speed was increased.

Keynote (S01-363, Time: Friday 10:15, Room: St. Gallen)

Negative Impact of Viscous Dissipation in Polymer Processing

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The standard way of polymer processing with a three-zone screw would not work without viscous dissipation. The work done by the processing machine induces shear that is transformed into the heat necessary to melt the polymer. Shear-rates and stresses increase in narrow slits and fine geometries like the flight between the screw and the barrel, in sprue- and hot runner-systems as well as in the tools for extrusion. Furthermore, more shear is induced into the material by using smaller machines with higher through put rates to increase productivity by saving on machine size and therefore cost and energy demand. In polymer processing it is well known that these circumstances can lead to a degradation of the polymer as a result of the induced heat, leading to faulty products. Furthermore a degradation of the polymer can lead to harmful byproducts for human and machine as it for example is the case in PVC processing. In recent years, our ongoing research showed that not just the polymer can be damaged, but so can even the mold steel that is used for screws, barrels and tools, even if it is of a high grade. The data presented will prove, that due to the viscous dissipation a drastic rise of the metal surface temperature can lead to a loss of hardness and thus damage the steel although the polymer does not degrade! The loss of hardness decreases the resistance against abrasive wear significantly. The detected reason for this loss of hardness is a growth of the secondary carbides. These carbides bind chromium and therefore reduce the resistance against corrosion leading to even higher wear. This development would in turn again cause faulty parts, more energy consumption and overall decreased productivity. Beside the shown research results a guideline for wear related damage analysis and preventive measures will be presented.

Keynote (S01-616, Time: Friday 13:45, Room: St. Gallen)

Prediction and Optimization of Blush Defect in Plastic Injection Molding using Machine Learning Methods

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Injection molding plays a vital role in the mass production of high-quality plastic products. Many researchers have conducted studies to predict the injection defects and optimize the products' quality. Although many of the defects of injection molded parts (such as weld lines, sink marks, flashes, etc.) have been substantially investigated, a research gap is found in prior studies about the effect of process parameters on the blush defect. Accordingly, this study aims at investigating the influence and extent of effect of eight process parameters (injection flow rate, holding pressure, melt temperature, mold temperature, runner diameter, gate diameter, gate angle, and included angle) on the occurrence and intensity of the blush defect. For this purpose, digital twin, meta-heuristic algorithm, statistical (i.e., design of experiments and analysis of variance) and machine learning methods (i.e., artificial neural networks (ANN), ANN combined with genetic algorithm, and ANN combined with particle swarm optimization) have been employed. As a result, artificial neural networks showed a higher prediction accuracy of the blush defect with an average deviation error of 1.3% from reference data based on experimentally validated multi-physics numerical simulations. Finally, an additional optimization step was conducted using both analysis of variance optimization and genetic algorithm. Results showed that the genetic algorithm provided an improved optimization in terms of process parameters that allowed for a further reduction of the blush defect area.

Oral (S01-029, Time: Wednesday 16:00, Room: St. Gallen)

Over-moulding of dissimilar polymers with optimized interfacial bonding strength enhancement due to interfacial surface modification

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Over-moulding process is an enabling technique to bond dissimilar polymer material with various material properties into an integrated plastic product without using mechanical fasteners or chemical joining. However, due to high chemical inertness for most polymers, polymer material is characterized under low surface energy which leads to poor compatibilities and interfacial bonding strength. This paper investigates the optimum interfacial bonding mechanism design by introducing mechanical interlocks between two dissimilar hard-soft material combinations. A series of interlock designs are created on base layer of hard polycarbonate (PC) substrate. Soft thermoplastic urethane (TPU) melt flows into various interlock designs during over-moulding process study to form a strong grip onto PC substrate. The authors quantified the bonding strength enhancement with respect to various interlock designs through both finite elements modelling and over-moulding experimental studies.

Oral (S01-034, Time: Wednesday 16:20, Room: St. Gallen)

Monitoring and control of plasticizing quality in regrind polymer injection molding

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To meet the demand for net-zero carbon emissions in manufacturing, it is critical to use regrind polymers to efficiently manufacture plastic parts and maintain the same quality consistency as virgin polymer materials in injection molding. However, due to the effects of temperature, pressure, and shear stress on the polymer during polymer processing, the material quality of regrind polymer is different from that of virgin polymer. As a result, the quality, such as weight and geometry, of molded parts made from regrind polymers differs from parts made from virgin polymers. Although proper setting of packing conditions can compensate for this deficiency, it is of limited benefit for injection molding parts with high flow length to wall thickness ratio (L/t ratio) due to poor pressure propagation. Instead, this study improves the quality consistency of molded parts by controlling melt quality during plasticization in injection molding of regrind polymer. Based on the pressure profile measured in mold cavity, the changes in extracted quality properties during injection molding of regrind polymer are monitored and the plasticization parameters are adjusted to approximate the molded part quality between the regrind polymer and the virgin polymer. The experimental results show that the pressure integral index can reflect the quality change of the molded part during the injection molding of regrind polymer. In particular, by adjusting the back pressure, after six shots, the quality of the molded part is close to that of the part made from the virgin polymer.

Oral (S01-059, Time: Wednesday 16:40, Room: St. Gallen)

Investigation of surface quality of talc and rubber contained in PP material for automotive plastic parts

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The material of the automobile plastic parts contains talc and rubber in PP in order to satisfy rigidity and impact property. At the part where the resin merges, minute ridges forms along the weld line and remain even after painting, causing a problem with the appearance that looks like a linear scratch. This research was conducted on technological measures to reduce ridges and improve the surface quality. First, we observed the appearance and size of ridges on the painted surface after injection molding. Next, in order to evaluate the influence of talc and rubber on the ridges, four materials including talc and rubber, materials including only talc, materials including only rubber, and materials including only talc and rubber and materials including only PP were used for evaluation. As a result, It was found that rubber had influence on the ridges. Therefore, by quantitatively measuring the degree of elongation and direction of the rubber in the flow process of the resin in the mold, it was clarified that the rubber was stretched in the flow process and orientated in the plate thickness direction at the confluence part, and a relative shrinkage difference was generated at the general part and the confluence part, and it became the ridges. Two technological measures were devised to examine measures to reduce the ridges. The first method was to evaluate the effect on the ridges using rubber, which is different hardness. The second method focused on the thickness of the solidification layer of the skin layer in contact with the mold and evaluated the effect of the mold coating on the ridges. From the above experimental results, it was confirmed that the use of hard rubber reduced the ridges. In addition, it was confirmed that the mold coating slows the cooling and solidification of the surface layer part, reduces the orientation of the rubber in the thickness direction of the joining part, and reduces the ridges.

Oral (S01-064, Time: Wednesday 17:00, Room: St. Gallen)

Mechanism Analysis of Rib Sink Depression Using Internal Pressure

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Thinning is one of the lightening measures for exterior resin parts in automobiles. However, since rigidity is insufficient, reinforcement by setting a rib can be considered as a measure, but sink marks on the back surface of the rib become an issue. In this study, we focused on the use of internal pressure for sink marks in ribs. Concretely, by supplementing the volume shrinkage component with bubbles by chemical foaming, the rib sink is suppressed. It was considered that maintaining heat at the base of the rib was one of the rib sink mark control mechanisms to promote foaming. When multiple rib shapes were prepared and the effect of sink mark improvement was confirmed, the difference in foaming effect was confirmed depending on the shape. Next, it was considered that the balance between the contraction of the skin layer and the expansion of the bubble was one of the mechanisms of the suppression effect of the rib sink in the foaming. The kind of foaming material was changed, and the visualization observation was carried out on the difference of foaming effect by the rib shape and foaming behavior in the resin flow to the rib part actually. The difference in the effect of the foaming agent was confirmed. And, the figure in which contraction of the skin layer and expansion of the bubble were balanced was able to be observed from the hardening behavior in the rib division. On the basis of these results, the suppression mechanism by applying the internal pressure to Rib moss is summarized and reported.

Oral (S01-082, Time: Thursday 10:40, Room: St. Gallen)

Datadriven prediction of quality characteristics of injection moulded parts

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In the digitalization process, there are many interesting use cases that are intended to optimize injection molding as a production process. One of the most frequently discussed use cases is quality prediction. The aim here is to predict the quality or the quality characteristics of the manufactured parts based on process data. If a decision on the quality of the parts can be made directly after production, this is a decisive advantage. Deviations or trends in the quality characteristics can be detected at an early stage and direct good/bad part sorting becomes possible. Mathematical models of artificial intelligence offer great potential here. The basis for the use of these mathematical models is a proper database. This includes not only the various parameters based on which the quality is to be predicted, but also the quality characteristics of the parts themselves. These must be available so that the mathematical models can be trained. Investigations have shown that with the help of selected process parameters, promising predictions for the quality characteristics can be made with different regression models. Therefore, the data sets are being divided into training data and test data. Already with a simple linear model a R2 of approx. 0.85 can be achieved. The model is improved by adding interaction terms, which allows a R2 greater than 0.9. Increasing energy costs and the careful use of resources strengthen the desire for zero-fault production. Through smart real-time data analysis and the use of artificial intelligence, any process instabilities can be detected at an early stage and eliminated through the targeted introduction of countermeasures. To be able to reduce the experimental effort required for training the models in the future physically based prognosis models should be integrated as well.

Oral (S01-085, Time: Thursday 11:00, Room: St. Gallen)

Properties of polypropylene/polyamide 6/multiwalled carbon nanotubes micromoldings

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Microinjection molding (μIM) is considered as a convenient technology for mass fabrication of polymeric microparts. However, the electrical conductivity of the microparts was always inferior to those reported for conventional injection molding and compression molding counterparts, and the prevailing high shear rates in μIM were believed to be responsible for such phenomenon. In this work, the concept of adopting immiscible blends as the host matrix for conductive fillers was employed with an aim to reduce the percolation threshold and increase the electrical conductivity of resultant microparts for potential applications in industrial sectors. Polypropylene/polyamide 6 (PP/PA6) blend was employed as the model system to study the influence of adding carbon nanotubes (CNT) on the properties of microparts. Results showed that the percolation threshold of PP/PA6/CNT microparts was lower than that of PA6/CNT and PP/CNT counterparts. The selective localization of CNTs in PA6 and the in-situ formation of CNT-enriched PA6 phase were believed to be crucial for increasing the electrical conductivity of corresponding moldings. Moreover, unlike compression molded samples, the blending sequence of PA6, PP and CNTs showed little dependence on the electrical conductivity of PP/PA6/CNT microparts when the concentration of CNTs was 7 wt%. This study provides a perspective to improve the electrical conductivity of microparts at lower filler concentrations which sheds light on preparing conductive microparts for potential applications in industrial sectors.

Oral (S01-110, Time: Thursday 11:20, Room: St. Gallen)

Study on the Fiber Bending Phenomena in FRP Injection Molding Parts

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Fiber-reinforced thermoplastics (FRP) is one of the most effective lightweight technologies in automobile industry. Indeed, the fiber orientation distribution (FOD) is one of the most important microstructures to provide the reinforced capability in FRP injection molding. However, even today, it is still very difficult to obtain the accurate FOD either from numerical prediction or experimental observation. In this study, the FOD of the ASTM D638 injection specimen with 30 wt% of 3 mm short fiber polypropylene (30SFPP) is analyzed by using numerical simulation (Moldex3D) method. The FOD is also validated through micro-computerized tomography (micro-CT) and image analysis technology based on Avizo. Results show that the flow direction orientation tensor component A11 is increased from 0.7 to 0.78 at skin-layer then decreased to 0.6 at core-layer. Overall the fiber orientation is dominant along the flow direction at near gate region (NGR). On the other hand, the A11 component from experimental observation present very close trend (with average error of 7% only). To further study what causes the difference in FOD between simulation prediction and experimental observation. The NGR is divided into twenty-one zones. In each zone, a single fiber is selected randomly. Each fiber is then regarded as two type of shapes: one is bent with original curved shape, and the other is assumed as a straight rod. Based on the shapes of fibers, the average deviation of the FOD between two types of fibers is about 4%. In addition, the difference of the fiber length between those fibers is about 6.5%. Obviously, even for short fibers, the fiber bending can be one of the major reasons to cause deviation for numerical prediction of FOD.

Oral (S01-133, Time: Thursday 11:40, Room: St. Gallen)

Optimization of injection moulding parameters for thermoplastic composter bottom

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Nowadays, it is necessary to educate people about the separation of waste depending on the type of the material. In the Republic of Croatia, more and more work is being done on proper composting of fruits, vegetables and other food in the households. Decomposition of organic matter is sensitive to air flow, humidity and temperature, thus apropriate conditions are needed for effective composting. This, includes the correct selection of materials, design and the process of production for the composter. In this paper, the simulation analysis will be carried out during injection moulding of the bottom of the composter. Contemporary injection moulding machines require fine tuning for succesfull mould filing and satisfactory product quality. Plastic products are often approached from design for manufacturing standpoint to ease the challenges related to injection moulding process and mould design. Injection pressure, injection time, packing pressure, packing pressure holding time, melt temperature, mould cavity temperature and cooling system features are common variables of choice in process optimization. Achieving consistency in product quality, process parameters should be optimized for a specific case since there is a lot of variability among injection moulding input parameters. To recognize locations of critical values of defects, such are shrinkage and warpage, Moldex3D software is used. Warpage and shrinkage defects are minimized by DOE optimization method. Results show that simulation can be implemented with optimization techniques to successfuly improve product quality and process efficiency.

Oral (S01-169, Time: Thursday 12:00, Room: St. Gallen)

A comparative study of different color sensors for inline color measurements in the injection moulding of recyclates

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Achieving a uniform product color plays an important role in injection moulding, particularly when it comes to the direct processing of recycled polymers. Due to the inhomogeneity and contamination of these materials there is a need for control strategies for the product color, which requires application of inline color sensors. There are two types of color sensors that can be industrially applied, including spectrometers and True Color Sensors. While both systems can be used for inline measurements, True Color Sensors are usually cheaper, show a smaller chip area and higher operating speeds. In this work, we compared the inline applicability of two True Color Sensors in the injection molding of artificial material mixtures including virgin and recycled materials. First, a systematic experimental study was carried out to measure the color of injection moulded parts in an offline environment. To this end, the proportion of masterbatch in the part was varied to evaluate the accuracy of the measuring systems and to collect data with regard to an automatic color controller for masterbatch. Next, the sensor closest to the spectrometer results was used for inline measurements in the injection moulding machine and for further measurements investigating thermochromism. In the latter the behavior of the product color was examined after cooling from demoulding to room temperature. The results indicated a low temperature influence on the measured product color.

Oral (S01-170, Time: Thursday 14:10, Room: St. Gallen)

Inline optimization for injection molding processes for abrupt and gradual process behavior alterations

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Inline optimization in injection molding mostly relies on rigid statistical process control to discover irregularities in the process and manual reoptimization of the process. This occupies a considerable amount of time of experienced workers. Therefore, a methodology is required which autonomously detects changes in the process (concept drift, CD) and automatically reoptimizes the machine settings with a flexible model, preferably in a machine manufacturer agnostic platform. Several drift indication algorithms are used to evaluate a multitude of process and quality parameters. These are comprised of moving averages over a varying time span and complex methods such as adaptive windowing, drift detection method, early drift detection method and a Kolmogorov-Smirnov test derivative. The analyzed parameters are part weight, maximal injection pressure, dosing time, melt cushion and cavity pressure. If a CD is identified, recent process data is used to adapt a machine learning model which is supplied for a genetic algorithm to reoptimize the machine settings. The model is initially trained with data from the injection molding setup process. Two datasets for the development of CD algorithms are generated, both with polypropylene. The first dataset is comprised of data with varied machine settings to induce drifts in the process parameters. Machine settings are defined with, inter alia, 30 °C mold temperature and 450 bar packing pressure. After the processing conditions reached equilibrium, the packing pressure is raised to 700 bar and then, after return to the standard conditions, the mold temperature is raised to 60 °C to provoke both an abrupt and gradual CD. For the second dataset, four material batches recycled material are processed with fixed settings to provoke CD due to batch fluctuations. Abrupt changes are identified rapidly after 5 cycles in initial results. Underperforming results for gradual changes will be improved by data filtering and preprocessing.

Oral (S01-176, Time: Thursday 14:30, Room: St. Gallen)

Experimental Analysis of Transient Behavior of Melt Filling Through Gate Based on Polymer Compressibility

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The rheological properties of polymer melt significantly affect the quality of injection molded components. However, during polymer melt filling through a restricted gate, a corner vortex structure is induced by the viscoelastic behavior of the polymer in the contraction flow. Furthermore, a choking effect is produced by the compressibility flow. The choking effect leads to a sharp reduction in the volumetric flow rate, which induces flow instabilities and results in defects on the surface of the molded component. Accordingly, this study derives an analytical expression for the transient flow rate of the polymer melt through the gate based on a consideration of the melt compressibility in contraction flow. The effects of the molding conditions on the melt filling behavior are then examined in experimental trials using acrylonitrile butadiene styrene (ABS) and high-density polyethylene (HDPE). The experimental results show that the specific volume (S.V.) of the polymer melt during transient contraction flow through the gate is dominated by the injection speed. As the injection speed increases, the choking phenomenon induced by the melt compressibility increases and reduces the volumetric flow rate to a value less than the nominal value calculated for a Newtonian fluid. As the melt temperature increases, the S.V. of the melt also increases and leads to a reduction in the transient flow rate. However, the melt temperature has only a minor impact on the choking effect compared to the injection speed. The variation of the transient flow rate of the ABS feedstock material is more significant than that of the HDPE due to a larger free volume and compressibility. However, the ABS flow rate increases rapidly after choking. Further experiments will be conducted in future studies to investigate the effects of the gate geometry and polymer material on the choking effect in order to optimize the gate design. Keywords: Compressibility, choking effect, transient flow rate, restricted gate, injection molding

Oral (S01-200, Time: Thursday 14:50, Room: St. Gallen)

Mechanical properties of injection-moulded glass fibre reinforced Polyamide with flowing weld-lines

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The negative impact of weld-lines (WLs) on the mechanical behaviour of injection-moulded short fibre reinforced polymers (SFRPs) is caused by various reasons, such as poor bonding between the polymer chains at the interface, unfavourable fibre orientation at the WL, and the presence of V-notches and voids in the vicinity of the WL. This study aims to investigate the influence of each of these factors on the WL strength of Polyamide 6 (PA6) reinforced with 50 weight percent (wt%) glass fibre (GF), which is injected in a double-gated plate mould in order to generate a flowing WL. Tensile tests and rheological measurements showed that the WL in neat PA6 samples can be fully healed. Thus, in the absence of V-notches, the significant drop in the mechanical properties is a consequence of unfavourable fibre orientations at the WL region. The fibre orientation distribution at the WL is scrutinised using the Autodesk Moldflow simulation as well as the micro-computed X-ray tomography (micro-CT) scans of the part. As it has been shown that the WL strength could be partially recovered by optimising the injection moulding process parameters, the effect of the packing pressure, melt temperature and mould temperature on the WL strength is also studied. This showed that increasing the packing pressure can produce a more favourable fibre orientation and hence improve the mechanical properties at the WL, while the other two parameters had less influence on the WL strength. ACKNOWLEDGEMENTS: The work leading to this abstract has been funded by the ICON project “ProPeL”, which fits in the MacroModelMat (M3) research program, coordinated by Siemens (Siemens Digital Industries Software, Belgium), and funded by SIM (Strategic Initiative Materials in Flanders) and VLAIO (Flemish government agency Flanders Innovation & Entrepreneurship). The authors would like to thank Domo Chemicals for providing the material.

Oral (S01-218, Time: Thursday 15:10, Room: St. Gallen)

Efficiency of extrusion of polyethylene nanocomposite with a new generation extruder

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New polymeric materials, characterized by more and more new properties, including rheological ones, in many cases are difficult to process and inefficient, which results in the application of proper processing techniques and conditions. One of the ways to make their processing possible is to look for new structural solutions of processing machines, especially plasticizing systems of extruders and injection moulding machines that ensure their proper processing. This work presents various constructional solutions of the plasticizing system of a single-screw extruder equipped with innovative, original elements: an active grooved feed section (AGFS), a rotating barrel segment (RBS) and a special screw (SS), structurally adjusted to a fundamentally changed barrel. The methodology of selecting the best constructional solution of the mentioned vital elements of the plasticising system has been demonstrated. Such an innovative extruder with an independent screw and rotating barrel segment drive has been constructed and equipped with appropriate pressure and temperature sensors and an automatic control system. Then, the process of extrusion of polymeric material with nanofiller in the form of halloysite nanotubes HNT was carried out using a conventional extruder and an extruder with the mentioned innovative elements. The extrusion was carried out for two different screw speeds: a low speed of 50 rpm and a high speed of 150 rpm and using a smooth barrel and a barrel with four longitudinal grooves with a maximum depth of 6 mm, width of 5 mm and length of 100 mm. Each extruder used a screw dedicated to it. The efficiency of the extrusion process was checked by determining the mass flow rate of the polymer, specific energy consumption supplied to the extruder, efficiency of the extrusion process, energy supplied to the plasticating system and energy transferred by the processed material. Appropriate relations were prepared and conclusions drawn.

Oral (S01-298, Time: Thursday 16:00, Room: St. Gallen)

Effects of in-process wear of laser induced mould surface features within an injection moulding process

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The integration of micro- and nano-features on polymeric surfaces has become increasingly popular over the last decades, as these features can add a wide range of functionalities to the product. A promising process technology to manufacture such products is injection moulding. By applying the negative geometry of the surface features on a mould cavity, millions of products with functionalised surfaces can be produced in a single step. However, for the injection moulding process to be economically viable, it is essential that the micro surface features within the mould can successfully withstand several thousands of injection moulding cycles. In this work, the in-process wear of laser induced mould surface features is determined within an injection moulding process. Moreover, the effect of the subsequent mould wear on the functionality of the replicated surface features is investigated. Three different surface features are studied, being solid microneedles, hollow microneedles, and hydrophobic micro-bumps. A total of 30,000 injection moulding cycles are conducted. After every 10,000 cycles, the in-process wear of the mould surface is characterized using a confocal microscope and the functionality of the replicated parts is assessed. The solid and hollow microneedles are evaluated using penetration tests with artificial skin, while the hydrophobic surface is assessed using contact angle measurements.

Oral (S01-307, Time: Thursday 16:20, Room: St. Gallen)

Development of a Self-Adjusting Injection Molding Production Cell: A Step towards Autonomous Sampling

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Injection molding is a polymer processing technique with potential for a high degree of automation. Skilled technicians are always required for the successful operation of an injection molding production facility. They determine basic settings of injection molding processes, optimize part quality and adjust setting parameters if process variations lead to poor part quality. Under the conditions of skill shortage, these various tasks lead to organizational challenges. Research and development in industry and universities has been addressing automatic process adaptation to deal with process variations for more than 10 years. This frees up skilled workers and gives them more time for tasks that are not automated. An injection molding machine that automatically determines the basic process setting can provide further work relief. The automatically determined basic setting can then be used as a basis for optimization of the part quality by the skilled operators. In order to develop a self-adjusting system, a production cell, consisting of an injection molding machine and an external computer, was set up. Communication within the production cell takes place via OPC UA. To determine the basic setting of the injection molding process, the set-up procedure is divided into multiple subtasks. To solve the subtasks, the external computer predefines movements for the injection molding machine. The sensor data of the injection molding machine are analyzed by the external computer and suitable setting parameters are calculated. The self-adjusting production cell does not require any information about the injection mold or the polymer used. No sensors are required in the injection mold. In addition, it is not necessary to carry out an injection molding simulation in advance. The manufacturing cell developed was validated with two injection molds.

Oral (S01-362, Time: Thursday 16:40, Room: St. Gallen)

Sample preparation and thermal conditioning of PC and PP on thermal and mechanical properties

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Over the years, the use of polymers in household appliances has steadily increased. Polymers have an excellent strength-to-weight ratio and are easy to process. Injection molding is usually used to manufacture parts that are further used in household appliances. Polycarbonate (PC) has high Tg, thermal stability, and good mechanical properties, while polypropylene (PP) is highly wear-resistant and does not absorb water. Consequently, they both are used for production of many parts in home appliances. To design and develop sustainable parts and products, where even the smallest change in design can drastically change their performance, we need to be able to predict the time-dependent mechanical properties of polymer materials under different environmental conditions and loads. To evaluate time-dependent mechanical properties of selected polymers (PC and PP), rectangular and cylindrical samples were prepared by injection molding. Rectangular samples were used for determination of extensional, while cylindrical samples were used for determination of shear time-dependent mechanical properties. To optimize the annealing procedure time, the samples were annealed for 1.5–6 h, at 0.1°C/min. The influence of annealing procedure was observed using DSC and DMA temperature sweep measurements, which are used to determine the effect of temperature on the mechanical response of the material before and after annealing procedures. The results showed profound influence of annealing on viscoelastic properties, while no significant difference was observed when using longer annealing times. It was confirmed that the annealing procedure is a critical step in the determination of long-term behavior of polymer products. Furthermore, only properly determined polymer material properties lead to optimal predictions when simulating polymer behavior using the finite element method (FEM). The authors acknowledge financial support from Slovenian Research Agency (Research project No. L2-3165).

Oral (S01-365, Time: Friday 10:40, Room: St. Gallen)

Mechanical Properties of Hot Runner and Cold Runner Injection Molded Bioplastics

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Biopolymers have become increasingly interesting due to their petroleum-free sourcing and end-of-life disposability compared to conventional plastics. Hot-runner injection molding is characterized by lower scrap compared to the cold-runner alternative, thus making it more environmentally sustainable. However, in a hot runner mold, polymers are kept at high temperatures for a longer time, which could lead to thermal degradation for biopolymers. Consequently, the processing window required to maintain mechanical properties is inherently narrower for biopolymers than for conventional plastics. In this work, different biopolymers, including PLA and PBS, were injection molded using a hot-runner and a cold-runner tensile bar mold. Materials were tested at the low and high melt temperatures of their respective processing windows on each mold to evaluate how the biopolymers behave differently in a hot-runner versus a cold-runner system. The mechanical evaluation was performed utilizing tensile and Izod impact test methods on post-fabricated samples. DSC was performed to see how manufacturing changes alter the crystalline behavior of the materials.

Oral (S01-367, Time: Friday 11:00, Room: St. Gallen)

Development of a Micro Injection Molding Machine for Small to Medium Series

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The advancing miniaturization of components in numerous areas is increasing the need for micro injection molding technologies. These include the production of a wide variety of components for medicine, bionics, and electrical engineering. Micro components are produced on cost-intensive multi-cavity molds. These molds can only be operated economically for large quantities. At the same time, the demand for the individualization of components linked to small batches is increasing. Regarding raw material consumption, the sprue's weight is often several times the weight of the part. Therefore, only a reduced profit margin can be achieved, or the part costs are getting more expensive. Small shot weights are only feasible to a limited extent on conventional plasticizing and injection units. The residence time of the plastic melt becomes so high that material degeneration occurs. Therefore, injection molding machines must be adapted to the required shot weights. The downsizing of a classic injection molding screw is only feasible to a certain extent for mechanical reasons. The developed injection molding machine aims at the known problems in micro injection molding, such as long residence times, inhomogeneous melt quality, insufficient reproducibility, and excessive machine or production costs. In order to make small series production of micro components profitable, the machine enables the production of components from thermoplastic parts in a single cavity mold. The prototype, which has already been manufactured and tested, enables the production of sprueless components with a shot weight between 0.05 g and 0.25 g. No additional shut-off nozzle is required, as is the case with conventional systems. For an economic consideration of the developed plasticizing and injection unit, the performance is validated using a demonstrator part. Properties such as energy consumption, reproducibility, and part quality are considered.

Oral (S01-378, Time: Friday 11:20, Room: St. Gallen)

Comparison of the Mechanical and Crystalline Properties of Injection Molded Virgin and Recycled Polypropylene

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The mechanical properties of injection-molded polypropylene parts are affected by polymer macromolecular characteristics and processing conditions. The high temperature, pressure, and shear rate, required for injection molding control the mechanical properties by affecting the formation of different crystalline structures. The effects are more accentuated for recycled polymers, typically characterized by wider molecular weight distribution and possible contamination. In this work, injection molding experiments were carried out to study the relationship between processing, mechanical properties, and morphology. The analysis focused on virgin and recycled polypropylene resins, characterized by different properties. Tensile and Izod impact testing were performed on parts molded at different temperatures and pressure. The crystallinity of the molded parts is analyzed using Differential Scanning Calorimetry. The results show that processing parameters, mainly melt temperature, affect mechanical properties differently between virgin and recycled PP. The analysis of the processing and mechanical data allows correlation of the results with the formation of α-phase and β-phase.

Oral (S01-379, Time: Friday 11:40, Room: St. Gallen)

In-Mold Rheology and Automated Process Control for Injection Molding of Rheologically-Modified Polyolefins

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Injection molding of recycled polymers is inherently characterized by higher variability of the polymer states due to their wider molecular range distribution and compositional variability. When compared to virgin polymers, lack of consistency in the quality of the molded parts can arise from processing variability. In this work, in-mold rheology is utilized to assess the processing variability of recycled film-grade polyolefins and define strategies for automated process control. Different grades of the same recycled resins are produced by high-speed extrusion to modify their molecular weight. The experiments are carried out using a pressure-controlled injection molding system, which maintains the nozzle pressure constant during the injection while adjusting the screw velocity. The viscosity of the polymer within the cavity is adjusted by modifying the screw velocity in response to cavity pressure signals. Different quality metrics, such as the maximum cavity pressure or melt flow front velocity, are assessed to control the processing stability. The experiments are designed to investigate the automated process control as a function of different processing parameters, part wall thickness, and polymer rheology. The efficacy of the approach is validated by checking the weight and mechanical properties of the molded parts.

Oral (S01-411, Time: Friday 12:00, Room: St. Gallen)

Weld line studies on polyolefins and post-consumer recyclates

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Though highly investigated, the collision of two or more melt fronts is still inevitable in most injection moulded parts. These applications have to deal with more or less intense defects due the resulting weld lines. Therefore, the aim of this work was to learn about the mechanisms and influence parameters, as well as possibilities to reduce the resulting defects like reduced mechanical performance and visible flaws. The growing re-use of plastic products enhance the material variability and add new influence parameters. In this paper we investigate the hot and cold weld line behaviour of polyolefins (PP and PE) under different processing parameters in comparison with PP and PE post-consumer recyclates. Two special moulds were used to produce test specimens along different positions in a flowing weld line area and one for cold weld line specimens. With this moulds, virgin polypropylene and polyethylene materials were processed into test specimens, as well as two different post-consumer recyclates. To gain information about the best processing parameters, three different mass temperatures and two mould temperatures were systematically varied. The so produced specimens were tested with a universal test device for their tensile behaviour. The optical defects were evaluated by means of an optical microscope and scanning electron microscope. It showed, that hot weld line defects are more intense at position 1 near the obstacle that divides the flow front. Interestingly, the virgin PP material shows a higher weld-line effect than the PP recyclate and the influence of the processing parameters is higher. In general, the best values were gained with medium processing temperatures (200 °C) and higher mould temperature (50 °C). This is true for cold as well as hot weld lines.

Oral (S01-435, Time: Friday 14:10, Room: St. Gallen)

Competitive study of injection moulded and 3D printed conductive thermoplastic elastomer structures

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In this study, we will compare the performance of flexible conductive structures produced by additive manufacturing (AM) and injection moulding (IM). For the fabrication of the 3D-printed conductive structures, pellets were introduced in a screw type extruder printing head. For injection moulded conductive structures, 3D-printed moulds were fabricated with thermoplastic material extrusion (MEX-AM) with filament printing heads. On one hand, AM is a powerful method to produce free-forming structures. With this method, it is possible to rapidly change the stucture design, and therefore achieve high design flexibility without increasing the cost per part significantly. On the other hand, IM is a widespread method to produce structures time-efficiently on a large scale. In general, the advantage of IM is the high surface quality and dimension precision. To reduce mould costs and manufacturing time, researchers have started to combine both manufacturing methods. Good precision of the moulds printed by MEX-AM can be already achieved, however, the surface quality needs to be improved. In our study, we observed that good printability of different commercially available filaments and thermal stability during IM could be achieved. For this study natural polyamide (PA), PA carbon fiber-reinforced filament and polypropylene glass fiber-reinforced were used. To optimize the surface quality of the moulds, printing parameters and post-treatment methods were investigated and analysed by optical microscopy. Conductive disc structures for tactile sensing applications were fabricated by 3D printing and IM. The investigation was performed for composites with different filler contents of the conductive filler. The effect of the manufacturing method of the flexible conductive structures were compared for the surface quality and the sensor response during compression. Due to orientation effects, the initial conductivity of the 3D printed tactile sensors and thickness slightly differed.

Oral (S01-510, Time: Friday 14:30, Room: St. Gallen)

Thermography in injection molding process for in-line defect characterization of molded parts

Müller Dennis[1], Puch Florian[1]

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To preserve energy, resources and postprocessing worktime, the use of in-line embedded sensors to monitor product quality is becoming a growing field of research. In this investigation several defects common to injection molded parts are purposely produced by choosing the process parameters accordingly. Theses defects are inspected by in-line thermography and the detection accuracy is examined. Hence, different installation positions of the thermal imaging camera were investigated by considering possible sources of thermal interference and comparing the results with parts showing defects such as short shots, vacuum voids, or flashes. Furthermore, the influence of the time interval for taking the thermal image was investigated for the installation positions to further narrowing down the recording window and to approximate the loss in detection accuracy over time. These aspects are important to implement thermography sensors for industrial scale cycle times. One of the defects investigated, the vacuum voids, were not fully detectable even with the most optimal camera position and recording time. Initial investigations showed that the depth, shape and positioning of the vacuum voids influence the detection accuracy. Future experiments with multiple thermal imaging cameras should provide further information.

Oral (S01-601, Time: Friday 14:50, Room: St. Gallen)

Tuning the performances of injection molding polypropylene parts by in-mold annealing.

Salomone Rita[1], Speranza Vito[1], Liparoti Sara[1], Pantani Roberto[1]

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To obtain plastic products 100% recyclable, thus, reducing the environmental impact of plastic, industries are looking to switch to parts made of just one material (mono-material). This work focuses on the possibility of controlling the properties of isotactic polypropylene parts obtained by injection molding. One of the main operating parameters that play a key role in the final characteristics of the part, in other words, the performance of the part, is the mold temperature. It is generally recognized that high temperature treatments, namely annealing, influence the microstructure and the morphology of molded part. Injection molding tests coupled with in-mold annealing are conducted on an isotactic polypropylene. The in-mold annealing is characterized by two steps: the polymer is injected in a mold at high temperature (413 K or 433 K), which is kept for 5 minutes (first annealing step); afterwards, the mold temperature is cooled down at 403 K and held at that temperature for a time compatible with the crystallization half time at that temperature (second annealing step). The morphological characterization of the molded parts is carried out by optical and electronic scanning microscopy. Furthermore, atomic force microscopy coupled with HarmoniX tool allows detecting simultaneously local morphology and mechanical properties. The part morphology is characterized by a fibrillar layer close to the sample walls and a spherulitic core; a transition zone is also detectable in between. Final morphology distribution along the sample thickness is significantly influenced by the adopted in-mold annealing conditions. Morphology greatly affects mechanical properties: the transition zone shows the minimum value of elastic modulus; additionally, the elastic modulus increases with spherulite dimensions. Eventually, the in-mold annealing results to be crucial in determining the part properties and can be adopted as a robust method to tune properties through the process.

Poster (S01-132, Time: Thursday 17:00, Room: Foyer)

Influence of Oscillating Surfaces on the Markedness of Weld Lines by Investigating Mechanical Properties

Bruchmüller Matthias[1], Vincent Raphael[1], Puch Florian[1]

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In this investigation the shear thinning effect of an oscillating surface in a mold is used to minimize the influence of weld lines on the mechanical properties of the produced test specimen. Therefore, simulations were used to calculate the appropriate process conditions for a test specimen, containing weld lines. The processed with weld lines showed a significant decrease in Young’s modulus and tensile strength, compared to weld line free specimen. Subsequently, a piezo actuator was used during the production of parts with weld lines to reduce the decrease in the mechanical properties with the help of oscillation.

Poster (S01-370, Time: Thursday 17:00, Room: Foyer)

Analysis Methodology of the Effect of In-mold Conditions on the Quality of Injection-Molded Parts Using Explainable Artificial Intelligence

Turng Lih-Sheng[1], Gim Jinsu[1]

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In-mold conditions measured by in-mold sensors in the cavity have been regarded as the most relevant information to the quality of injection-molded parts because they include and reflect the details of molding conditions and polymer properties. Hence, process monitoring, control, and optimization has been attempted by utilizing certain types of in-mold conditions such as peak cavity pressure and/or cycle-averaged mold surface temperature. However, finding influential features in the in-mold condition profiles and quantifying their effects on part quality are difficult because of the complex relationship between the in-mold conditions and part quality. As a solution, the characteristic features of in-mold conditions have been predetermined using prior knowledge and understanding on the process and polymer material, and the correlation between the features and part quality has been investigated. This work proposes a novel analysis methodology concerning the effect of in-mold conditions on part quality using explainable artificial intelligence (XAI). Interpretation of machine learning model for quality prediction using in-mold conditions can pinpoint relevant features and their respective effect on quality based on the experimental data. Point-wise and section-wise approach for feature extraction and model interpretation was compared and section-wise interpretation offered better explanations on the effect of features on part weight. The methodology not only provides reasonable explanations of the effect of in-mold-condition features on various quality factors like shrinkage, and warpage, but reduces the dependency of process analysis and optimization on knowledge and understanding, thereby, realizing intelligent injection molding that is centered on in-mold conditions.

Poster (S01-392, Time: Thursday 17:00, Room: Foyer)

Study on Injection Molded Direct Joining between PA66 and A5052

Enomoto Kazuki[1], Kurosaka Kousuke[1]

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Injection molded direct joining (IMDJ) is one of the metal-plastic direct joining processes and is based on a combination of a special surface treatment of a metal piece and an insert molding. The advantages of IMDJ are high design freedom and high productivity, then it is a technique that contributes to weight reduction through multi-materialization. In this study, A5052 plates were used as the metal pieces and mechanical surface treatments such as blasting and drilling were applied. IMDJ was performed by inserting A5052 piece into the cavity and injection molding PA66. Joined single-lap specimens were subjected to tensile shear tests to determine their fracture strength, and the effects of hole diameter and number of holes in the drilling process, and the presence or absence of blasting treatment on strength were experimentally evaluated.

Poster (S01-418, Time: Thursday 17:00, Room: Foyer)

Investigation of experimental validity of varying resin flow length

Sugino Naoto[1], Fujii Takuro[1], Kaneko Atsushi[1], Hashimoto Miyuki[1], Kameda Takao[1], Yao Shigeru[2]

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In our previous reports, we have obtained experimental results for molding polypropylene resin at a single point gate for small and large flat plates, where the difference in tensile rupture elongation within the molded product tends to increase as the flow length increases. It is important to homogenize the distribution of physical properties (tensile rupture elongation) within the molded product for use as a product. Therefore, in this study, an attempt was made to make the physical property distribution in a flat molded product constant by matching the resin flow length. The method used was to change from a single-point gate to a two-point gate to make the flow length uniform. The mold was a flat plate with dimensions of 389 mm in the flow direction and 180 mm in the width direction. Because the mold flows from the edge of the molded product with a single fan gate, it is possible to conduct experiments in which the flow length is made uniform by placing the other gate near the center of the molded product. However, due to the mold structure, only a pin gate can be installed in the center of the molded product, and it is not possible to create the same fan gate shape as in one-point gate molding. One possible problem is that if different gate types are used, the flow will not be the same and may not be evaluated based on resin flow length alone. Therefore, using resin flow analysis software, we examined whether there would be any change in resin flow even if the gate shapes were different at one or two points. The results of the resin flow analysis showed that even if the gate shapes at the two points were different, the resin flow was the same for the one-point and two-point gate molding in the area where the specimen was taken. This result suggests the validity of the experiment to change the resin flow length.

Poster (S01-427, Time: Thursday 17:00, Room: Foyer)

Effect of Resin Flow Length on Tensile Breaking Elongation in PP Injection Molded Products

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In our previous reports, we have obtained experimental results for molding polypropylene resin with a single point gate, where the difference in tensile breaking elongation within a molded product tends to increase as the flow length increases for small and large flat plates. It is important to homogenize the distribution of physical properties (tensile rupture elongation) within the molded product for use as a product. In previous research, attempts were made to make the physical property distribution uniform by placing two gates in the mold (one at the edge of the molded product and the second at the center of the product) to equalize the flow length of the resin. To verify the validity of the experiment, resin flow analysis was conducted, and it was suggested that even if the gate shapes at the two points were different, the resin flow in the specimen sampling area was the same for the one-point gate molding and the two-point gate molding. In this study, based on those results, a two-point gate mold was fabricated to investigate the effect of resin flow length on tensile rupture elongation In two-point gate molding, the gate at the end of the molded part was first opened, and after resin reached the gate in the center of the molded part, the gate in the center was opened to prevent weld from occurring. Then, dumbbell-shaped specimens were punched out separately at different locations. Tensile test results showed that the molded product with a single point gate showed variations in tensile rupture elongation depending on the location of the molded product, but the difference in tensile rupture elongation between locations was smaller for the molded product with a two-point gate. The experimental results suggest that the tensile elongation at break can be homogenized within a molded product by maintaining a constant resin flow length within the product.

Keynote (S02-183, Time: Thursday 10:15, Room: Monosuisse)

Evaluating the electromechanical performance of dielectric elastomer actuators in uniaxial tension state by Smart Rubber Analyzer

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Dielectric elastomers have gained a considerable attention as promising electromechanical transducer materials for soft sensor and actuator technologies. A dielectric elastomer actuator (DEA) consists of a thin elastomer layer separating two compliant electrodes and undergoes large deformations in geometric shape when subjected to electric fields. Such electrically induced deformations are persuaded for the development of shape changing structural components of soft robotic systems and, according to the Maxwell's equation, relates to the dielectric constant, elastic modulus and thickness of elastomer at an applied electric field strength. In this study, the influence of elastomer structure and barium titanate filler on dielectric characteristics i.e. voltage-induced electrostatic pressure (Maxwell stresses), polarizability and permittivity is investigated by electro-rheological measurements. Application-specific tensile, electrical and dynamic properties of actuator materials are also determined and compared. The dielectric and tensile characters of polar nitrile butadiene and polyurethane elastomers are found to influence the electromechanical performance of DEAs as determined by means of an indigenously designed tabletop Smart Rubber Analyser (SRA) device. According to the working principle of SRA, a rectangular strip actuator under uniaxial loading state of fixed strain is subjected to a voltage pulse and a decrease in applied deforming force is recorded on actuation as a function of applied voltage (see Figure 1). The effect of elastomer composition, pre-strain level and sequence of applied voltage on actuation is tested and discussed.

Keynote (S02-236, Time: Thursday 13:45, Room: Monosuisse)

Self-repairable and reprocessable thermoset polyolefin elastomer based on dynamic covalent bonds prepared by reactive melt blending

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Thermoset elastomers are widely used in industry, but the presence of permanent crosslinks in the thermoset elastomers limits their recyclability and the waste products cause environmental problems. Recently, polymer networks linked by associative dynamic covalent bonds exchangeable each other at elevated temperature, called vitrimers, have been paid great attentions as new materials demonstrating both good performance properties of thermoset and thermal reprocessability of thermoplastics. Numerous thermoset elastomer vitrimers have been reported, but studies on polyolefin based elastomeric vitrimer prepared by a melt blending method are relatively rare. Herein, elastomeric polyolefin vitrimers were prepared through dynamic crosslinking of commercially available thermoplastic maleated polyolefin elastomer using proper crosslinkers in Haake internal mixer to introduce different type of dynamic covalent bonds in the network. Effects of the type of dynamic covalent bonds in the network as well as the presence of inorganic nanofillers on mechanical properties, recyclability, stress-relaxation behavior and self-healability of the vitrimers of polyolefin elastomer are discussed.

Keynote (S02-280, Time: Friday 10:15, Room: Monosuisse)

Influence of binary graphene/carbon black hybrid fillers on thermal and mechanical properties of natural rubber nanocomposites

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In this study, we focused on the synergistic effect between carbon black (CB) and graphene nanoplatelets (GNPs) of various aspect ratios and specific surface areas as hybrid fillers in natural rubber (NR) nanocomposites. Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, and scanning electron microscopy (SEM) were carried out to characterize the GNPs properties, while dynamic mechanical analysis (DMA), tensile properties, hardness, thermal conductivity, swelling behavior in toluene and SEM were performed on the NR nanocomposites. A synergistic effect between GNPs and CB was observed for this GNPS/CB hybrid filler system mainly on the thermal and mechanical properties of their corresponding NR nanocomposites. This synergistic effect is mainly attributed to the high surface area and aspect ratio of the GNPs that help in the creation of a thermally conductive hybrid fillers' network. Moreover, the GNPs/CB hybrid fillers' synergy increased with increasing their concentration up to 5 phr (parts per hundred of rubber) due to their good dispersion, as confirmed by SEM characterization. The tensile strength and elongation at break were slightly improved for each of the three GNPs used. On the other hand, it was observed that the incorporation of GNPs into the NR reference sample increased the Payne effect and the GNPs with higher aspect ratio and higher surface area had more influence on the swelling ratio by generating more restrictions against solvent diffusion into the compound. Finally, the thermal conductivity was improved with the substitution of CB by GNPs. At 2 phr GNPs, the highest thermal conductivity improvement (49%) was obtained with the highest surface area GNPs.

Oral (S02-009, Time: Thursday 10:40, Room: Monosuisse)

Fuel-efficient Green Tire tread compound: Optimization of mixing parameters

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Towards a greener environment, demand for a fuel-saving tire with low rolling resistance emerges; thereby, the application of silica filler is governing the tire industry day by day. Recently, carbon black has been replaced by silica as a reinforcing filler for the development of "green tires" in the tire industry. The "green" claim is that silica-reinforced materials help further reduce rolling resistance, save vehicle fuel and reduce CO2 emissions to the environment. Silica-silane technology dramatically contributes to the field tire industries by saving fuel. However, unlike carbon black mixing, silica mixing is complicated as it involves several reactions inside an internal mixer. The key factor for better silanization in silica and silane rubber system is to efficiently devolatilize and reduce the condensation of ethanol inside the mixing chamber. In this paper, we have evaluated the individual and synergic effects of mixing parameters and optimized them in the silica-filled elastomer compound using the design of experiments (Taguchi and Response surface method). Silanization temperature and time are found to be crucial parameters for silanization. Compounds mixed using optimized mixing parameters show superior performance properties compared to the control. Filler dispersion in the optimized batch was better and characterized using a scanning electron microscope (SEM). Tan δ @60 °C as an indication of rolling resistance is improved by 23%, indicating the better fuel economy of this system. The derived optimum condition for a better degree of silanization is 60% fill factor, 155 °C silanization temperature, 240 s silanization time, and 80 °C of Temperature Control Unit.

Oral (S02-046, Time: Thursday 11:00, Room: Monosuisse)

Solid-phase orientation for stiff and tough thermoplastic vulcanizates

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Die drawing is a processing technique that has been used to produce polymeric products with superior mechanical properties by orientation of polymer chains. Although this process has been studied for a variety of thermoplastics and composites, no publications have previously explored its efficacy to enhance thermoplastic vulcanizates (TPVs). TPVs combine the elasticity and high elongation at break of thermosets while maintaining the processability of a thermoplastic. For the first time, TPVs have been die drawn and the effect upon their mechanical and thermal properties has been investigated. TPVs based upon Ethylene propylene diene monomer (EPDM) and polypropylene (PP) as well as ground tyre rubber (GTR) and PP were prepared by dynamic vulcanization using a twin-screw extruder. In all cases, die drawing led to an increase in tensile strength and modulus of the material along the drawing direction, often at the cost of elongation at break. At a draw ratio of 2, EPDM/PP TPVs exceed the strength and modulus of neat polypropylene whilst maintaining an elongation at break in excess of 200%. For EPDM/PP, tensile strength increased from ~21 MPa to over 90 MPa using a draw ratio of 4, whilst for GTR/PP this increased from ~15 MPa to ~57 MPa. Keywords: Solid-phase deformation, thermoplastic vulcanizates, die drawing, recycling

Oral (S02-081, Time: Thursday 11:20, Room: Monosuisse)

Microwave heating of rubber extrudates

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The increasing demand for energy-efficient methods for vulcanizing rubber extrudates requires the optimization and further development of existing vulcanization processes. Vulcanization by microwave irradiation makes it possible to introduce the energy required for heating directly into the material by means of dielectric losses. Microwave heating requires the polarity of the rubber compound so that the electromagnetic wave can vibrate the polar components in the compound. This oscillation results in internal friction, which leads to an increase in the temperature of the rubber compound. However, the polarity of rubber compounds is reduced due to corrosion protection, especially in the automotive sector. This requires optimization of the process parameters in the microwave crosslinking process. For this purpose, it is necessary to specifically adapt the setting variables of the electromagnetic wave as well as the geometry of the heating chamber to the different rubber compounds. The investigations of these process parameters showed a material-specific excitation frequency and heating chamber geometry at which the absorption behavior of the wave reaches its maximum. As a result, 90 % of the electromagnetic energy applied has already been converted into material heating in laboratory tests.

Oral (S02-146, Time: Thursday 11:40, Room: Monosuisse)

Influence of the blowing agent type on the foam properties of high consistency silicone rubber

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High-consistency silicone rubber (HCR) is typically processed by extrusion or injection molding. Due to its excellent properties in terms of flexibility down to low temperatures, physiological harmlessness, and wide operating temperature range, there are a large number of applications for silicone rubber; especially in the automotive industry, the food sector, or medical technology. Due to the high material costs, foaming HCR makes sense for a large number of applications, such as gaskets, dampers, insulation or similar. Nowadays, chemical blowing agents are used for the most parts in the foaming of silicone rubber. One problem with the use of chemical blowing agents, however, is that the decomposition reaction of the blowing agent must be coordinated with the crosslinking reaction, resulting in a narrow process window. In addition to the difficulty of process control, toxicological effects and the decomposition of products remaining in the product are also critical factors in the use of chemical blowing agents. In this study high-consistency silicone rubber was mixed with different types of blowing agents in a conical twin-screw mixer and then extruded in order to work out the advantages and disadvantages of physical blowing agents compared with chemical blowing agents. For the physical blowing agents, a mixture of silica and water in a ratio of 1:2, gas-filled microspheres, and a commercial available blowing additive based on crystalline water was used. The AZDN blowing agent from Pergan served as a reference for the chemical blowing agents. For the selected compounds, the vulcanization properties were first determined and then the foam structure, density and mechanical properties were evaluated on the extruded profile, with clear differences in the properties and foam structures between the individual blowing agents.

Oral (S02-185, Time: Thursday 14:10, Room: Monosuisse)

Process-induced anisotropy and its effect on swelling of elastomers: theory and experiments

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Most elastomer products exhibit process-induced anisotropy, whether they be compression-moulded, rolled or injection-moulded. This anisotropy is known to influence both the mechanical behaviour and the swelling response. Despite this, the anisotropy is generally ignored when determining the cross-link density from swelling experiments. In this paper we examine this experimentally and theoretically, and show that although the anisotropy influences the calculation of cross-link density, its effect is generally small in compression-moulded and rolled products. Careful measurements of linear, volume and mass swelling ratios were carried out on five cross-linked elastomers. Two of these, an EPDM and a natural rubber, were compression-moulded and showed transversely isotropic swelling. A further three, an EPDM, a nitrile butadiene and a chloroprene rubber, were rolled, and exhibited fully anisotropic swelling. This behaviour is consistent with the anisotropy observed in the response to uniaxial mechanical deformation on the same materials. We then extended the Flory-Rehner theory of swelling to account for general anisotropy of swelling, producing a more versatile form of the relationship between linear swelling ratios and cross-link density. The anisotropy leads to a correction to the calculation of average cross-link density, and we illustrate the possible magnitude of this effect. When applying the extended relationship to the experimental data, the effect on the cross-link density is visible, but relatively small in the compression-moulded and rolled specimens. The effect on components with greater anisotropy, such as may be produced by injection moulding is expected to be larger and more significant. The conclusion from this work is that, reassuringly, in most cases researchers can use the classical Flory-Rehner theory as a reasonable estimate of cross-link density without accounting for swelling anisotropy, which is more challenging and time-consuming to determine.

Oral (S02-192, Time: Thursday 14:30, Room: Monosuisse)

The Influence of Soft Segment on Physical Properties of Polyurethanes crosslinked by Polyrotaxanes

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We have already reported the physical properties of polyurethanes crosslinked by Polyrotaxanes consisting of poly(tetramethylene glycol) and diphenylmethanediisocyanate (PUPRX-PTMG-MDI). Here, we prepared polyurethanes crosslinked by polyrotaxanes consisting of poly(caprolactone) and diphenylmethanediisocyanate (PUPRX-PCL-MDI) (or hexamethylenediisocyanate (PUPRX-PCL-HDI)) to compare the influence of soft segment structures on the physical properties of PUPRXs. As a result, PUPRX-PCL-HDI showed the best elongation (strain is 5000) among our PUPRXs. We also carried out the hydrolysis test and found that the hydrolysis of PUPRX-PCL-HDI occurs easier than that of PUPRX-PCL-MDI).

Oral (S02-205, Time: Thursday 14:50, Room: Monosuisse)

Lifetime Prediction Method for Elastomeric Polyurethane Components based on Nonlinear Damage Accumulation and Finite Element Simulations

Rayer Moritz[1], Schiffers Reinhard[1]

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Elastomeric polyurethane components are used in many applications to solve problems of vibration damping or compensation of geometric misalignment. The material damage induced by thermo-mechanical stress collectives significantly influences the vibration behavior of the entire system. However, in product development, the consideration of progressive material damage is only possible to a limited extent. A possibility to face this problem is the coupling of a material model with a damage accumulation model to describe a damage progression in simulations. For this purpose, fatigue tests are performed on specimen level to generate damage parameters. The parameter sets are then used to calibrate a damage-dependent material model. A progress of material damage is calculated by a nonlinear damage accumulation model. The results are transferred to a demonstrator component using the finite element method to map the influence of progressive material damage on the entire component and finally on the component's lifetime.

Oral (S02-308, Time: Thursday 15:10, Room: Monosuisse)

Analysis of the center load influence of dynamically stressed elastomers on the lifetime in drive engineering applications

Ternes Sabrina[1], Mrzyk Pascal[1], Schiffers Reinhard[1]

[1]University of Duisburg-Essen, Duisburg, Germany

Elastomer components are used in numerous drive engineering applications to counter vibration-related problems. The lifetime of these elastomer components depends on many influencing factors. In particular, the type of stress influences the lifetime of the component. Despite the widespread use of elastomers in technical applications, research into elastomer-specific damage phenomena is currently lagging behind other materials. Preliminary tests have already shown that, in addition to the magnitude of the applied load amplitude, the center load position also has a decisive influence on the lifetime. This paper contains an investigation of the center load influence of dynamically loaded elastomers on the damage and lifetime of the material. For this purpose, material tests are carried out under dynamic loading and variation of the center load. Since elastomer components are used in drive engineering applications for different vibrational stresses, specific test specimens are used to represent the different types of stress. To eliminate the temperature influence due to the self-heating of the elastomer, the tests are carried out under damping power control with adaptation of the frequency. An evaluation is performed regarding the dynamic and damage-relevant characteristic values. These include the dynamic stiffness and the loss energy. Finally, the onset of damage and the lifetime are determined, and the influence of the center load is analyzed.

Oral (S02-314, Time: Thursday 16:00, Room: Monosuisse)

Calcium based layered double hydroxides as reinforcing fillers in natural rubber

Jones Louise[1], Labuschagné Frederick J. W. J.[1]

[1]University of Pretoria, Gauteng, South Africa

Layered double hydroxides (LDHs) have been shown to act as reinforcing fillers in various elastomers. Composites of natural rubber (NR) and various LDHs were prepared using a conventional sulphur cure system to investigate the effect of the LDH on the mechanical properties of the NR. The LDHs that were used are Ca-Al-NO3 LDH, Ca-Zn-Al-NO3 LDH and Ca-Fe-Al-NO3 LDH, either unmodified or modified with sodium stearate or sodium dodecyl sulphate. The LDH was added at a loading of 6 phr. The unmodified LDH was poorly dispersed. XRD, FTIR and TEM showed that organic modification improved dispersion of the LDH in the polymer matrix and allowed partial exfoliation and intercalation to take place. The improved dispersion improved the mechanical properties of the composites in comparison to when unmodified LDH was used. Mechanical testing showed that in comparison to the unfilled reference sample, the stearate modified LDHs all increased both the tensile strength and elongation at break while the other LDHs tended to decrease tensile strength but increase elongation at break. Ca based LDH can therefore be used as a reinforcing filler in NR when organically modified.

Oral (S02-403, Time: Thursday 16:20, Room: Monosuisse)

Investigation of the interaction between thermal conductivity and foam morphology of elastomers foamed by expanding thermoplastic microspheres

Frohberg Richard[1], Çelik Hakan[1], Hopmann Christian[1]

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The use of physical foaming agents enables both a reduction in density and an application dependent adjustment of the stiffness properties of technical rubber components. The focus of the presented research is on the application of expanding thermoplastic microspheres (TMS) as physical foaming agent for EPDM-based elastomer foams. TMS allow a two-step configuration of the foam morphology. In the preceding mixing process, a pre-configuration of the morphological parameters such as porosity, pore size, etc. can be achieved by selecting specific types of TMS. Whereas, in the second step of the manufacturing process, the final resulting foam morphology is determined by controlling the process temperature, since the expansion behaviour of TMS depends on the prevailing temperature. Therefore, it is necessary to have a good understanding for the local thermal conductivity. However, due to the expansion of the TMS and the resulting foam morphology, the thermal conductivity is a local, time- and temperature-dependent variable, which affects the expansion behaviour of the foam and vice versa. Hence, for the numerical modelling of the foaming process, it is required to describe the interaction between the foam morphology and the thermal conductivity. To investigate the interaction, the influence of morphological parameters on the thermal conductivity is determined numerically using Representative-Volume-Elements (RVE). The experimental space is defined by the porosity, pore size distribution and the pore distance. For validation, the thermal conductivity is determined experimentally on the pure elastomer and on specimens with different foam morphologies. The objective is the derivation of an analytical equation, which allows the determination of the foam structure dependent thermal conductivity and can thus be used for subsequently performed process simulations for the prediction of the resulting foam morphology.

Oral (S02-439, Time: Thursday 16:40, Room: Monosuisse)

Understanding the recycling of tyres by devulcanization and revulcanization

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Ground tyre rubber (GTR) is an abundant waste resource that has been undervalued. Tyres are a highly engineered rubber product that require significant resources to manufacture, with over 1.5 billion produced annually. Devulcanization selectively breaks sulfur crosslinks (rather than main chain C-C bonds) allowing conversion of GTR into a high-value recyclate. This powdered material can then be blended into virgin rubber and revulcanized. We evaluate how mechanical devulcanization by solid-state shear milling (S3M) reduces crosslink density and effects the sulfur bonds. The ratio of mono-/di-/poly- sulfidic bonds was measured by Soxhlet extraction using chemical probes. Additionally, devulcanized GTR/virgin natural rubber blends were prepared. The processing, curing and mechanical properties were measured and compared with CB-filled natural rubber. Devulcanized GTR prepared by S3M is shown to be elastically effective within the recycled rubber blend, matching the stiffness of the equivalent virgin material. This may allow incorporation into existing rubber products as a replacement for virgin rubber.

Oral (S02-441, Time: Friday 10:40, Room: Monosuisse)

Effect of dispersant on the mechanical properties of Fluoroelastomers/VGCF composite

Ishida Yuki[1], Takarada Wataru[1], Shioya Masatoshi[1]

[1]Tokyo Institute of Technology, Tokyo, Japan

Fluoroelastomers have excellent chemical resistance, heat resistance, and weather resistance, and are used in automotive O-rings. On the other hand, low tensile strength and adhesion of contaminants due to electrification have been cited as points to be improved. The addition of high-strength, conductive fillers has been considered as a way to improve these issues. In this study, we prepared VGCF-added FFKM using three dispersants with different chemical structures and investigated the effects of different dispersants on the physical properties of VGCF-added FFKM. FFKM (Daikin Industries, Ltd.) was used as the fluoroelastomer and VGCF®-H from Showa Denko was used as the VGCF. Three dispersants with different chemical structures were used. These dispersants were first mixed with VGCF to adhere to the surface of VGCF, and then the dispersant-attached VGCF was mixed with FFKM. The dispersion of VGCF was observed by optical microscopy and SEM. Dynamic viscoelasticity (DMA) measurements and tensile tests at 100 degree C were performed on the fabricated VGCF-doped FFKM to clarify mechanical properties. The volume resistivity was also measured as an electrical property. Optical microscope and SEM observation showed no improvement in dispersibility due to the dispersing agent. Volume resistivity was also less affected by the dispersant. These results indicate that the dispersibility of VGCF was determined by physical shear stress and that the effect of the dispersant was small. Results of tensile tests confirmed that the addition of dispersants improved the tensile strength of FFKM. DMA measurements showed that the addition of dispersants did not change the glass transition temperature, but improved the storage modulus in the rubbery plateau. These results indicate that the dispersant enhances the reinforcing effect of VGCF in FFKM though improving the adhesion at the VGCF-FFKM interface.

Oral (S02-460, Time: Friday 11:00, Room: Monosuisse)

Electro active polymers and soft dry electrodes – innovative applications based on elastomers

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Haptic feedback, noise-free actuation, HMI (human machine interphases), BCI (brain computer interphases), wearable sensors for bio-signal monitoring - these are the application fields for our innovative products based on elastomer technology: electroactive polymers (EAPs)and soft dry electrodes. You will hear about how we developed materials and surface technologies, process technology and product design for such applications and which properties and functional behaviour you can expect from the products. A) Working principle of stacked EAPs: Electroactive polymer transducers made of soft and elastic polymers exhibit a mechanical motion when stimulated by an electric field or vice versa. 1.Electrical power turns into mechanical motion by elastic deformation 2.Unique thin layer stacks allow tactile and sensitive actuation 3.Reliable electrostatic mechanism for multipurpose and efficient operation Up to 12 N force is achievable and the macroscale stroke is tunable by the number of layers. B) Soft dry electrodes: Soft dry electrodes are a solution for high-performance bio-signal monitoring (EEG,ECG,EMG or EOG) thanks to the combination of a dedicated electrically conductive rubber compound and a specific coating. The electrode material and design is optimized to ensure comfort during monitoring and to allow dry signal acquisition, eliminating the use of gels and signifantly decrease skin irritation. A specific brush design allows the electrode to work without hair removal. Properties and functionality of both products for the different applications will be explained in more detail during the presentation.

Oral (S02-502, Time: Friday 11:20, Room: Monosuisse)

Investigation of Self-Healing Properties of Bromobutyl Rubber in the Presence of Novel Hybrid Nanoparticles

Kodal Mehmet[1], Kavacık Öznur[2], Gökçesular Sezgin[3], Mert Olcay[2], Özkoç Güralp[1]

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Therefore, the attentiveness of smart materials having the ability to respond to environmental changes such as damage is significantly enhanced. Self-healing polymers can restore themselves spontaneously with the aid of stimuli when they are exposed to mechanical damage [1]. Generally, non-covalent interactions, such as hydrophobic interactions, and dynamic covalent bonds, such as Diel-Alder reactions, are used to obtain self-healing properties in polymer materials [2]. In this study, it was aimed to increase the self-healing properties of bromobutyl rubber (IIR), which is widely used in the inner liner part of a tire due to its lower gas permeability. For this purpose, a new generation of inorganic/organic hybrid nanoparticles that capability of interacting with IIR was used in this study. Hybrid nanoparticles were directly incorporated into the IIR using an innovative high-torque intermeshing Xplore model twin screw micro-compounder. The other ingredients are common additives such as processing oil and vulcanizing agents. After the compounding process, the extruded strands were cured in a hot press at 160ºC. To evaluate the self-healing properties of the samples, tensile test specimens were cut at the middle of their lengths by a razor blade. Then, they were joined together in an oven at different temperatures and times. Finally, the tensile tests were carried out to determine the mechanical properties. Moreover, the rheological, morphological, and dynamic mechanical properties of samples were determined. In addition, to evaluate the possible interactions between the components, Fourier transform infrared spectroscopy (FTIR) analyses were carried out. In general conclusion, the self-healing property of IIR compounds was significantly improved in the presence of hybrid nanoparticles. References 1- Xu et al. Applied Materials and Interfaces, 8, 27, 2016. 2- Zhai et al., Nano Today, 30, 100826, 2020.

Oral (S02-505, Time: Friday 11:40, Room: Monosuisse)

Understanding the influence of pressure and viscous heat on slippage for commercial styrene-butadiene rubbers during extrusion

Georgantopoulos Christos[1], Esfahani Masood[2], Pollard Michael[2], Ellwagner Felix[2], Zhang Ziyue[3], Naue Ingo[2], Causa Andrea[4], Kadar Roland[5], Hatzikiriakos Savvas[3]

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[5]Chalmers University of Technology, Göteborg, Sweden

During processing of elastomeric compounds significant pressure drops (10^6-10^8 Pa) are encountered. Such conditions affect significantly the flow behavior of elastomers due to compressibility of elastomers, wall slip and viscous heating. A typical method to determine the slip velocity of polymeric materials during capillary flow is the Mooney technique. However, the classical Mooney technique does not take into account the compressibility and viscous heating effects during the flow. An experimental methodology is developed in order to determine the slip velocity corrected for the effects of compressible and viscous heating effects. First, the rising temperature during the capillary extrusion is measured at the exit and correlated to the apparent shear rate/shear stress. By confirming the applicability of the Cox-Merz rule, that is complex viscosity is equal to the absolute viscosity at the same frequency/shear rate value, the time-Temperature-Superposition (tTS) principle is used in order to correct the experimentally determined flow curvefor the viscous heating. the modified Mooney method can then be applied in general to account for compressible and viscous heating effects in order to determine both the floe curve and slip velocity at the set operating temperature.

Oral (S02-555, Time: Thursday 12:00, Room: Monosuisse)

Synthesis Magnetic Core-shell Fe3O4@Al2O3 as material used to remove Protein from Natural Rubber Latex

Nghia Phan Trung[1], Mai Linh Khanh[1], Nguyen Thang Ngoc[2], Kawahara Seiichi[3]

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The presence of protein in the natural rubber latex for the medical device manufacture seems to be the most considered compound due to its allergic effect. Magnetic coated alumina (Fe3O4@Al2O3) has been known as an adsorbent for organic substrates due to its performance through physical and/or chemical reaction. In the current study, we try to figure out the potential of Fe3O4@Al2O3 to prepare low protein's natural rubber latex with and without the presence of surfactant through a continuous batch system. The dry rubber content, surfactant amount and time during the incubation were investigated to determine the effective protein removal natural rubber latex. The highly deproteinized natural rubber was confirmed using Kjeldahl method, proven that the utilization of Fe3O4@Al2O3 could reduce the protein content from 0.38% to 0.016% in optimum condition (dry rubber content of 10.00%, surtactant of 0.25%, Fe3O4@Al2O3 of 1.00 wt% and 15 min of incubation time). These results showed that Fe3O4@Al2O3 is a very good material to remove protein from natural rubber latex and this method can be performed using a continuous batch system.

Oral (S02-591, Time: Friday 12:00, Room: Monosuisse)

Preparation and mechanical property study of polyurethane/silicone rubber composites based on polymerization induced phase separation

Yu Bing[1], Tian Ming[1]

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Thermoplastic polyurethane (TPU) materials possess the advantages of high wear resistance, tear resistance, fatigue resistance, oil resistance, repeatable processing, biocompatibility, and so on, which play an irreplaceable role in industrial applications. However, compared with other traditional rubber materials, TPU were not vulcanized and cross-linked, resulting in a higher permanent deformation. Therefore, it has certain scientific and application value to blend polyurethane (PU) with silicon elastomer (SiR) to improve the human touch feeling and resilience of PU. Based on the method of polymerization induced phase separation, in this research, the SiR raw material was dissolved into polytetrahydrofuran (PTMG), which was the monomer to synthesize PU materials, and then diisocyanate (MDI) was added. During the process of PU prepolymerization, the polarity of the system gradually increased, resulting in gradually lower solubility for SiR, which slowly precipitated to form a dispersed phase, and then cross-linked via the crosslinking agent to obtain the PU/SiR composites. By tuning the prepolymerization time and temperature of PTMG and MDI in the system, the phase state fineness of the SiR phase in the composite can also be tunable, with the phase size as low as 0.37 μm. The deformation retention, heat resistance, and mechanical properties of the obtained material are also improved. The tensile strength and the elongation at break can reach 15.2 MPa and 474%, respectively, and the tensile permanent deformation can be as low as 11.2%. At the same time, the obtained material retains the reprocessing property of TPU. This research realized the reactive blending of immiscible PU/SiR system without the addition of solvent and compatibilizer, which provides an innovative and feasible solution for the preparation of PU/SiR composites with high strength, high elasticity, and comfortable human touch feeling.

Poster (S02-006, Time: Thursday 17:00, Room: Foyer)

The effect of carbon black crystallinity on dispersion in rubber compounds

Jana Sadhan C.[1], Wen Ming C[1], Valsadwala Abbas C.[1]

[1]University of Akron, Ohio, USA

This work assessed the influence of carbon black (CB) crystallinity on the quality of dispersion in SBR compounds. It is hypothesized that graphitic content and structures serve as deterrent to dispersion of CB for a given shear and elongational forces. In view of this, experiments are developed to relate the quality of CB particle dispersion in rubber compounds with graphitic content. The experimental data show that poor dispersion results in the case of high crystallinity of carbon black particles. Several scenarios are discussed to explain the experimental data.

Poster (S02-448, Time: Thursday 17:00, Room: Foyer)

Partial Replacement of EPDM with Devulcanized Rubber in Thermoplastic Vulcanizates

Oner Tugce[1], Pamukogullari Beste[2]

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With the increase in consumption frenzy in the world, the rapid consumption of polymeric materials has increased and therefore the problem of waste management has come with it. When it comes to applications that require flexibility, the first materials that come to mind are rubbers. However, the structure that provides these properties is the cross-linked structures in the rubber formed by the vulcanization process which is a reaction that prevents recycling. In rubber recycling, research studies on devulcanization are increasing day by day to break the cross-links formed by vulcanization. In this study, thermoplastic vulcanizates (TPVs) were produced with EPDM rubber obtained by devulcanization method from white goods wastes by making partial changes in the untreated EPDM at the rates of 5%, 10% and 20%. In addition, to examine the effect of compatibilizer on the samples, the change of properties with the addition of 2% and 5% by weight PP-g-MA was analyzed. Then, mechanical, physical and morphological analyzes were performed to examine the effects of devulcanized EPDM material and compatibilizer on the properties of TPV samples. As a result, it was determined that there was no significant change in material properties when the EPDM phase in the TPV material was partially replaced by devulcanized EPDM waste. It was also found that successful results were obtained when compared with commercial TPV material. It is observed that the material properties are improved in the use of compatibilizer, and this can be interpreted as PP-g-MA triggers a strong interfacial formation between the materials under the influence of the silanized silica in the devulcanized material. In addition, the thermal stability of TPV samples increases with the use of devulcanized EPDM material. This can be interpreted that the silica filler in the devulcanized material increases the thermal homogeneity in the EPDM-PP phase.

Poster (S02-554, Time: Thursday 17:00, Room: Foyer)

Study on Magnetic Core-shell CoFe2O4@Al2O3 to remove protein from Natural Rubber Latex

Bui Binh Thi Thanh[1], Dang Hue Thi Minh[1], Nghiem Thuong Thi[1], Nguyen Thang Ngoc[2], Kawahara Seiichi[3], Nghia Phan Trung[1]

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The presence of protein in the natural rubber latex for the medical device manufacture seems to be the most considered compound due to its allergic effect. It is good to be use alumina to remove protein, but it is difficult to recover alumina. Using magnetic core materials is a very suitable way to recover alumina. Furthermore, magnetic core-shell alumina has been known as an adsorbent for organic substrates due to its performance through physical and/or chemical interaction. In the current study, we try to figure out the potential of magnetic core-shell as CoFe2O4. Synthesis magnetic core-shell has been investigated. The X-ray of magnetic core-shell was checked. Besides, the SEM of the materials was also captured and analyzed. The results might be good for the aluminum coating to create the material needed for the removal protein from natural rubber latex.

Poster (S02-567, Time: Thursday 17:00, Room: Foyer)

Polybis(trifluoroethoxide)phosphazene based dielectric elastomer actuators

Zeytun Karaman Cansu[1], Nüesch Frank[2], Opris Dorina M.[2]

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According to 2019 Global Burden of Disease study, approximately 1.71 billion people suffer from muscle disorders all over the world. Muscles are complex biological actuators with excellent reversible shortening, elongation, and rotational performances. Therefore, industry and academia have paid tremendous attention to understanding the working principle of mammalian muscles. Many artificial muscles have been constructed with different actuation mechanisms like dielectric elastomer actuators (DEAs), ionic polymeric actuators, micro piezoelectric actuators, etc. DEAs are versatile and promising candidates since they are soft, lightweight, and can show large actuation with a quick response time. Nevertheless, the major weakness of the DEAs is the need for a high voltage for actuation. The voltage needed depends on the elastomeric film's thickness, and mechanical and dielectric properties. The biggest impact can be made using high dielectric permittivity material. Poly(organo)phosphazenes are one of the future promising, versatile materials which can be used in DEAs. They are inorganic-organic hybrid polymers with a skeleton of repeating single and double bonds between nitrogen and a pair of or-ganic units attached to the phosphorus atom. Depending on the substituents, these polymers can offer a wide glass transition temperature Tg window ranging from -105 C to 100 C. Besides, the big phosphorus atom volume shows high electron polarizability that can provide higher dielectric properties compared to silicone and carbon-based counterparts. Herein, polybis(trifluoroethoxide)phosphazene was synthesized and functionalized with 5% allyl alkoxide. A series of materials were prepared by cross-linking this polymer with different amounts of cross-linker. Cross-linking was performed via thiol-ene reaction under UV light. Prepared materials were mechanically tested via tensile test, and dynamic mechanical analysis (DMA). DEAs were constructed from the most promising materials.

Keynote (S03-054, Time: Wednesday 13:45, Room: Olma)

PVDF-based electrospun mats with exceptionally high dielectric properties for triboelectric nanogenerator (TENG) energy harvesting applications

Naguib Hani E.[1], Rastegardoost Mohammad Mahdi[1], Aghababaei Tafreshi Omid[1], Saadatnia Zia[1],
Ghaffari-Mosanenzadeh Shahriar[1], Park Chul B.[1]

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Recently, several nanoscale modification techniques have been developed to improve the properties and functionality of dielectric materials for energy harvesting applications. Among these techniques, electrospinning has been identified as one of the most effective strategies, mainly due to the enhancement in the surface area of the dielectric materials. However, the developed electrospun dielectrics have a high degree of porosity, which subsequently lower the dielectric properties and thus affect their performance in various energy applications. In an attempt to enhance the dielectric properties, various nanoparticle additives have been introduced into the porous dielectric materials. Despite the performance improvement, these techniques resulted in intensive synthesis and complex processes, which can limit the large-scale fabrication of high dielectric electrospun materials. To address this challenge, in this study, a novel dipole arrangement technique is proposed to improve the dielectric properties of porous PVDF mats for high-performance energy harvesting applications without the addition of additives. Further improvement in dielectric properties is realized through designing multi-layer porous structures that yield a dielectric constant of up to 10, which is significantly higher than single layer electrospun mat, and is comparable to solid PVDF film. In comparison with other techniques, the developed PVDF film exhibited five times improvement in the dielectric constant. The developed electrospun PVDF mat in this study possesses a high dielectric constant and high area-to-volume ratio which is an ideal candidate for energy harvesting applications such as triboelectric nanogenerator (TENG).

Keynote (S03-091, Time: Thursday 10:15, Room: Olma)

Development of Various Formulations for Active Polymer Flexible Packaging

Ajji Abdellah[1]

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The purpose of this research was to develop active flexible polymer packaging to scavenge oxygen and ethylene. For Oxygen, systems based on Hydroxyl terminated polybutadiene with a catalyser and for ethylene, systems based on pumice and KMnO4 were used. The research was performed step by step, testing first compounds and blends scavenging capacity, the LDPE based films and finally ending with multilayer films. The results obtained showed very good results on gas absorption. This research provides a feasible methodology and promising prospect for active food packaging.

Keynote (S03-520, Time: Thursday 13:45, Room: Olma)

Sustainability in Electrospinning - "Greener" industrial scale manufacturing and application

Herwig Gordon[1], Rossi René M.[1]

[1]Empa, St. Gallen, Switzerland

A particularly interesting way to produce uniform nanofiber nonwovens from virtually any kind of polymeric material is Electrospinning. Applications range from semi-permeable artificial skin in biomedicine, over long-term release or efficient encapsulation in agriculture and functional textiles, to membranes for liquid and gas filtration. For industrial applications, an efficient continuous needle-less setup has been developped by Elmarco Ltd. Unfortunately, this technique in general is often far removed from a green and environment-friendly process, as it employs solvents with issues such as high flammability, toxicity, difficult disposal, or energy-intensive synthesis. In order to render electrospinning more attractive as a commercial technique and further improve scalability, solvent/polymer alternatives and other green routes have to be established, which adhere to societal and legal restrictions especially in regards to the environmental and health impact. The focus of this presentation is set on the sustainability of polymers and solvents (or solvent mixtures) used in solution, emulsion, suspension, and in situ cross-linking electrospinning. While sustainability is often equated to biodegradability, alternative routes to achieving a less resource- and energy-demanding process and/or long-term stable composition are also presented.

Keynote (S03-549, Time: Friday 10:15, Room: Olma)

Polymer material developments for melt spinning technology and fiber applications

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The Leibniz Institute fuer Polymerforschung Dresden has a long tradition in the field of targeted polymer material development for melt spinning technology and diverse fiber applications [e.g. 1]. Thereby, the processability in the melt spinning process and the morphology development during the filament formation of new polymer materials are in the focus of our research. In this paper, selected results are presented starting from polymer material modification to bulk scaling of spinnability and special application properties of the fibers. The following topics will be presented: a) Additive-free electron-induced modification of PLA/PCL blends for melt spinning process [2], b) Melt spinning of biobased and biodegradable fibers for use in sustainable composites (e.g. bioactive glass and polylactid acid (PLA)) [3], c) Fibers for controlled drug release by process-guided structure formation in hybrid fibers or by integrated fillers as drug carriers [4,5], d) Polymer fibers (PP) for bridging microcracks in highly ductile concretes (within DFG GRK2250) [6]. Current demands on fibers with regard to sustainable textile applications and their recyclability as well as for higher performance of fibers in technical applications provide impulses for further interesting approaches to our fiber research, which we are already taking up in our current projects and on which we may also be able to report at the time of the conference. References: [1]Beyreuther,R.;Vogel R. Intern. Polym. Process. XI (1996)154-158 [2]Huang, Y.;Bruenig, H.;Boldt, R.;Mueller, M.T.;Wiessner, S. European Polymer Journal 162(2022)110895 [3]Eichhorn, J.;Elschner, C.;Groß, M.;Reichenbächer, R.;Herrera Martín, A.X.;Soares, A.P.;Fischer, H.;Kulkova, J.;Moritz, N.;Hupa, L.; et al. Appl. Sci.11(2021)7927 [4]Kutlu, B.;Meinl; J.;Leuteritz, A.;Bruenig, H.;Wiessner, S.;Heinrich, G. Macromol. Mater. Eng.299(2014)825–833 [5]Sibanda, M.; Focke, W.; Braack, L.; Leuteritz, A.; Bruenig, H.; An Tran, N.H.; Wieczorek, F.; Trümper, W.Materials Science & Engineering C91(2018)754-761 [6]Popa, M.-M.; Bruenig, H.; Curosu, I.; Mechtcherine, V.; Scheffler, C. in P. Serna et al.(Eds.) BEFIB 2021, RILEM Bookseries 36(2022)255–264

Keynote (S03-617, Time: Wednesday 17:00, Room: Olma)

Hydrogen bonds in copolyamide favor processing and properties of fibers

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The hydrogen bond is an important interaction between polyamide chains, which improves its mechanical and thermal properties as well as crystallization behavior, while hindering the movement and orientation of segments during fiber formation. Copolymerization is one useful way to adjust the hydrogen bond density due to the irregularity of the macromolecular main chain. In this study, ε-caprolactam was used as the main monomer, in combination with a series of copolyamide 6/x. Variable temperature in-situ total reflection Fourier transform infrared (ATR-FTIR) spectra technology was used to analyze the formation and release of the hydrogen bonds between C=O group and N-H group. It was also used to analyze the crystalline form transition in a highly oriented copolyamide nanoweb with different copolymerization ratios, as well as in a phase change nanoweb with enhanced thermal enthalpy and conductivity. Results showed that the second monomer in copolyamide 6/x destroyed the regularity of the main chain in copolyamide 6/x and decreased the hydrogen bond interaction in intermolecular chains. It also facilitated the formation of the γ crystalline form in as-spun fibers and high-speed roller nanowebs, as well as the transition from the γ to α crystalline form after the thermal heating process. Also, hydrogen bonds between an ion liquid and copolyamide were formed in respective liquid-core phase change fibers, which resulted in a significant effect on the thermal performance. This research provides a simple route to achieve high tenacity and highly functional polyamide fibers.

Oral (S03-066, Time: Wednesday 14:10, Room: Olma)

Photo-degradation of Surface Starch to Facilitate a Combined Pretreatment

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Massive demand for water and natural resources for textile manufacturing significantly impacts the environment. Pretreatment of cotton fabric includes desizing, scouring, and bleaching carried out with wet treatments and requires considerable water and energy. The yarn is sized with starch to prevent yarn breakage during weaving. However, the removal of the starch is complex due to its high molecular weight and adhesive nature. The possibility of using UVC to degrade surface starch and facilitate a combined pretreatment has been explored. An efficient UVC-assisted process that combines desizing, scouring, and bleaching has been developed for cotton fabrics to save time, water, and energy. Grey fabric was padded with a pretreatment solution, exposed to UVC, washed off and compared to the conventional process. The new pretreatment method saves more than 70% of water, time, and energy from the conventional technique.

Oral (S03-083, Time: Wednesday 10:40, Room: Olma)

The influence of aging on the viscoelastic behavior of plastic films

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When growing plants, it is important to ensure good protection from weather conditions (hail, frost, wind...) and from animals (birds, insects, wild pigs...) to improve plant growth and increase production and quality. For this reason, plants are grown in covering systems (greenhouses) where covering materials play a major role. The covering material can be in the form of film and as such has an extremely low price, easy application, resistance to cracking and low weight. However, films also have certain disadvantages such as a shorter service life due to atmospheric influences, reduction of transparency and water vapor condensation. Regarding the tendency to use film for a longer time, this paper analyzes the effect of aging on the viscoelastic properties of polyethylene as well as ethylene/tetraflour-ethylene (ETFE). Viscoelastic properties were analyzed using dynamic mechanical analysis (DMA). Film made of ethylene/tetrafluoro-ethylene (ETFE) has better properties but is in a higher price range. Key words: ethylene/tetraflour-ethylene (ETFE), dynamic mechanical analysis, polyethylene, viscoelastic behavior

Oral (S03-112, Time: Wednesday 11:00, Room: Olma)

Strengthening Polyamide 66 Interfaces by Grafting Polymerisation of 2-hydroxyethymethacrylate

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Polyamide is mainly produced and used as fibres with an economically viable polymer synthesis and economically viable processing methods as for instance extrusion, injection molding, and melt fibre spinning. In addition, polyamide fibre properties cover an excellent tensile strength, good elastic recovery, very good electrical resistance and good resistance to chemicals. Therefore, the polyamide fibres are not only used for the apparel industry, but also for technical applications in the automotive and areospace, smart textile, or medical sector, often in the form of hybrids and composites. However, the production of hybrids with polyamide often leads to interface stability problems, as polyamide usually shows a smooth surface with a low surface energy. Surface modification techniques are a common approach to overcome this challenge. In this study, the formation of an adhesion promotion layer involving a grafting process of 2-hydroxyethymethacrylate (HEMA) is used to change the surface properties of a polyamide 66 fabric. HEMA forms of an adhesion promotion layer that introduces polar oxygen-containing groups promoting the wetting behaviour of the coated surface. Firstly, the mechanism and the kinetics of the grafting process are investigated. Secondly, the impact of the grafting process at different grafting stages on the fabric's properties is investigated. These include the change in surface chemistry via FT-IR, the change in topography via SEM imaging, the change in wetting behaviour and the change in bulk properties such as melting enthalpy and rigidity. Third, an optimum of processing conditions is determined, with the goal of generating a fabric with increased oxygen containing groups and improved wetting behaviour, while altering its bulk properties at a minimum.

Oral (S03-126, Time: Wednesday 11:20, Room: Olma)

Development of Phase Change Material based Electrospun Fibers for Thermal Management at Low Temperature

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To date, the perfect design and development of phase change material (PCM) based smart textile is of paramount importance in our day-to-day life for thermal management and mitigation of energy crises across the globe. This research work aims to fabricate PCM-based electrospun fiber via a one-step approach for low-temperature thermal energy storage applications. Here, polyethylene glycol (PEG) is chosen as a potential PCM. The surface and internal architecture of the as-prepared fiber mat are investigated by SEM and confocal micrograph. The observed interior architecture of the fiber is explained by surface wetting and solution miscibility experiment. The prepared fiber possesses average latent melting and freezing enthalpy of 35.72 and 29.35 Jg-1, respectively. The surface wetting experiment reveals the hydrophobic nature of electrospun fibers, which is beneficial for the long-term environmental stability of the textile. Moreover, the heat transfer characteristics of pristine fiber and PCM incorporated fiber are analyzed from the thermal camera images. The transient dynamic temperature vs. time curve of the PCM loaded fiber demonstrates the delaying of surface temperature increment compared to pristine fiber. The obtained thermal camera results demonstrate the improved heat transfer and storage efficacy of the composite fiber for cold energy storage application.

Keywords: Phase change Fiber; Electrospinning; Hydrophobicity; Thermal management; Heat transfer

Oral (S03-127, Time: Wednesday 11:40, Room: Olma)

Strategies for controlling the piezoelectric properties of PVDF fibers with reduced graphene oxide (rGO) for energy harvesting application

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Among many piezoelectric polymers polyvinylidene fluoride (PVDF) found great research interest owing to its high piezoelectric coefficient. PVDF in the form of electrospun fibers has outstanding piezoelectricity, flexibility, thermal stability, lightweight, chemical resistance, and biocompatibility. Therefore, they are suitable candidates for energy harvesting as well as in portable, wearable, and self-powered electronics. The β phase exhibits the highest piezoelectric response among the different crystalline phases in PVDF. Therefore, the fabrication of PVDF plays a vital role in producing a high β phase fraction and crystallinity. In this regard, the electrospinning mats possess advantages over film. Because through electrospinning, a single-step mechanical stretching and electrical poling process promote the growth of the β phase. Furthermore, there are different strategies discussed to improve the piezoelectric property of PVDF electrospun fiber. Herein, we compared the effects of solvents, the addition of reduced graphene oxide (rGO) as a filler, and the post-processing of the PVDF fiber and composite to analyze their effects on the morphology, crystallinity, and piezoelectric coefficient of the fibers and mats. Fourier transform infrared spectroscopy (FTIR) and differential scanning calorimetry (DSC) results showed that PVDF/rGO fibers in dimethylacetamide (DMAc) solvent have the highest β phase fraction and crystallinity. The piezoelectric measurements on single electrospun fibers and mats were studied using piezo-response force microscopy (PFM) and piezometer, respectively.PVDF/rGO fibers in DMAc solvent exhibited the highest piezoelectric coefficient from the characterization studies. We demonstrate different strategies to compare and control the piezoelectric properties of electrospun PVDF fiber. The piezoelectric coefficient can be significantly enhanced from the investigation by using DMAc solvent and incorporating rGO for energy harvesting applications.

Oral (S03-145, Time: Wednesday 14:30, Room: Olma)

In-situ Polymerization Electrospinning of Amine-Epoxy/Poly(vinyl alcohol) Nanofiber Webs for Direct CO2 Capture from the Air

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Amine-Epoxy/poly(vinyl alcohol) (PVA) nanofiber webs with an average fiber diameter of ca. 400 nm were successfully fabricated by the hot air-blow assisted electrospinning process accompanied by the in-situ polymerization, i.e. the cross linking polymerization reaction between the amine and the epoxy groups. The cross-linked amine-epoxy/PVA nanofiber web exhibited high adsorption rate and high CO2 adsorption capacity; the adsorption half time < 40 min in CO2 (400 ppm) condition for the direct air capture (DAC) and the CO2 adsorption capacity of 1.6 mmol/g. The cross-linked amine-epoxy/PVA nanofiber web exhibited excellent thermal stability as well as good desorption property at low temperature (<65 °C) and low energy consumption conditions.

Oral (S03-155, Time: Thursday 15:10, Room: Olma)

Sustainable and green fabrication of PLA fibers spun by microfluidic wet spinning

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PLA (Polylactic acid) fibers have been extensively used in biomedical engineering, and in sensing, textile, and food packaging application, thanks for their biodegradability and biocompatibility. Traditionally, non-solvent-based spinning (melt spinning) and solvent-based spinning (wet spinning, dry spinning and electro-spinning) are used to produce PLA fibers. While the PLA fibers spun by non-solvent-based spinning are unable to encapsulate bioactive proteins or growth factors because of high process temperature, those spun by solvent-based spinning usually involve toxic solvents (Tetrahydrofuran, Dichloromethane, Chloroform, etc.). Hence, the development of green solvent-based dissolution of PLA and mild spinning condition for PLA fibers is in acute demand for high-value utilization of PLA fibers. Furthermore, the tunable mechanical property and dimension of PLA fiber are of great importance to biomedical application of PLA fibers. Herein, we propose a sustainable and green PLA dissolution method to prepare PLA spinning dope. Cyrene, a bio-based and none-hazardous solvent, was used to dissolve PLA. Microfluidic wet spinning was introduced for spinning PLA fibers from 10 wt% PLA/Cyrene solution as core flow and alginate aqueous solution as shell flow. PLA/Cyrene core flow was encapsulated by alginate hydrogel layer in 0.7 wt% CaCl2, where PLA solidification was induced by solvent exchange.

Oral (S03-222, Time: Thursday 14:50, Room: Olma)

Laser-Heated Melt Electrospinning of Poly(L-lactide-co-ε-caprolactone): Fiber Formation Behavior and Characteristics of Prepared Fiber Webs

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In this study, fiber webs of poly(L-lactide-co-ε-caprolactone) (PLCL) with the poly(L-lactide) (PLLA) content of 75 mol% were prepared via a laser-heated melt electrospinning (LES) process, i.e. solvent-free melt electrospinning with CO2 laser irradiation for heating. In situ observation of fiber formation behavior revealed that only a single jet was formed from the swelling region under the conditions of low laser power, applied voltage, and feeding rate, whereas multiple jets and shots were produced with increases in these parameters. The formation of multiple jets resulted in the preparation of thinner fibers. Under the optimum condition, an average fiber diameter of 0.77 ･m and its coefficient of variation of 17% was achieved without the formation of shots. The estimation of tension and stress profiles in the spin-line was also carried out based on the result of in situ observation of the thinning behavior. In this analysis, the forces which are originated from surface tension, electricity, air friction, and inertia were considered. The maximum values of tension and stress appearing near the apex of the swelling region decreased with the increase in the feeding rate and voltage, which resulted in the thicker fiber in the case of single fiber ejection. Analyses of the molecular orientation and crystallization of as-spun fibers revealed the formation of a wide variety of higher-order structures depending on the spinning conditions.

Oral (S03-232, Time: Wednesday 12:00, Room: Olma)

Investigating the influence of UHMWPE fibers as a toughening agent for PMMA in denture base

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Denture bases as a highly used material in dentistry have faced some problems including the brittleness which can cause cracks in denture base and reduce the life duration of the product. In this study, the influence of UHMWPE fibers and surface treatment of the fibers with different methods have been investigated on the toughness of denture base. Fibers have been treated with sulfochromic acid, corona discharge, and silanization to enhance the interaction between the fibers and the PMMA matrix. The samples included different percentages of fibers examined by fracture toughness test, FE-SEM to investigate the impact of fibers on the toughness of the denture base composite. The surface treatment properties has also examined with FTIR and contact angle test. Results have shown that the chemical treated fibers with sulfochromic acid showed the best interaction to the matrix which caused improvement in toughness of the manufactured Denture base.

Oral (S03-235, Time: Wednesday 16:00, Room: Olma)

The Influence of Zn/Al LDH nanoplatelets on the mechanical and gas barierity of PLA/PBAT packaging films

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This research have used a melt-compounding method to create a biodegradable novel PLA/PBAT/Zn-Al LDH nanocomposite films for food packaging applications. The sol-gel method was used to synthesize Zn-Al LDH with four different amounts of LDH nanoplatelets (0.25, 0.5, 0.75, and 1 phr). The samples' morphology, mechanical properties, thermal behavior, and water vapor permeability were thoroughly investigated. Images obtained by Field Emission Scanning Electron Microscopy (FE-SEM) revealed that the use of Zn-Al LDH nanoparticles increased the interfacial interaction between the PLA and PBAT phases and also enhance their compatibility. In comparison to others, the sample miscibility in the presence of 0.5 phr of Zn-Al LDH was better. The XRD test was used to examine the structure of LDH crystals, and the results were presented. Tensile tests confirmed that the presence of Zn-Al LDH nanoparticles increased tensile strength. According to the results, tensile strength increased from 25.14 to 34.83 and 31.26 Mpa for samples containing 0.25 and 0.5 phr of Zn-Al LDH, respectively, comparing to samples without Zn-Al LDH. Differential scanning calorimetry (DSC) of the nanocomposite films revealed an improvement in the polymer matrix's crystallization ability as well as an acceleration of the cold crystallization phenomenon. Water Vapor Transmission Rate (WVTR) measurements revealed that Zn-Al LDH nanoparticles reduced WVTR content. PLA/PBAT with 0.5 phr Zn-Al LDH demonstrates the lowest WVTR, with a 53% downturn compared to the pure PLA/PBAT film. This is consistent with the morphological findings and demonstrates that the Zn-Al LDH nanoparticles improved the crystallinity of the samples. Overall, the results prove that the nanocomposite films that were created have a high potential for use as food packaging films.

Oral (S03-277, Time: Wednesday 16:20, Room: Olma)

Development of sustained controlled release of chlorine dioxide films for food active packaging applications

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Compared to other active packaging types, gas emitting packaging offers many advantages. Due to its strong antimicrobial properties, gaseous chlorine dioxide (ClO2) is an ideal candidate for antimicrobial gas-emitting packaging to prolong the shelf life of food products. Polymers commonly used in the food packaging industry were used in this study to develop a ClO2 emitting system without premature release. Two-layer films made from ethylene vinyl acetate (EVA) and low-density polyethylene (LDPE) containing citric acid and sodium chlorite (NaClO2) as precursors of ClO2 generation were produced. The generation of ClO2 was triggered by hot pressing the two layers together. The effects of relative humidity, temperature, and concentration of precursors on the release of ClO2 were evaluated. Relative humidity above 90% in the packaging triggered the generation of ClO2 faster and led to significantly higher gaseous ClO2 production. Furthermore, the active films released more ClO2 as the temperature increased. As compared to other labels, the EVA incorporated with citric acid and the LDPE incorporated with NaClO2 provided the longest sustained ClO2 release (93.83 ± 1.67 mgClO2 L-1) in a 28-day period. In conclusion, these two-component systems can be used as active labels on food packaging that release ClO2 over a longer period without premature release.

Oral (S03-282, Time: Wednesday 16:40, Room: Olma)

Effect of Material Modification on Structure Development and Characteristics of Syndiotactic Polystyrene Fibers

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Syndiotactic polystyrene (sPS) has good thermal properties, high chemical resistance, and low water absorbance. Therefore, sPS is expected to be applied to fiber products for apparel and industrial uses. On the other hand, it is known that polystyrene tends to exhibit brittleness regardless of its stereo-regularity. Accordingly, it is considered that the improvement of mechanical properties is indispensable for manufacturing fiber products. In this research, a high-speed melt-spinning process was applied for the production of sPS fibers. Along with the analyses of the structure and properties of high-speed spun fibers through the wide-angle X-ray diffraction (WAXD), differential scanning calorimetry (DSC), and interference microscope analyses, on-line measurement of the spin-line was performed in the spinning process to investigate the crystallization (solidification) behavior. Several types of materials, i.e. pure sPS of different molecular weights, sPS copolymers, and sPS blended with atactic PS, were applied. We found that the spinnability of sPS was improved by decreasing the molecular weight, copolymerization, and blending aPS. From the measurement of the thinning behavior of the spin-line, it was speculated that the improvement of spinnability resulted from the shifting of the position of the neck-like deformation to downstream, which was probably caused by the suppression of orientation-induced crystallization. Furthermore, it was observed that the maximum tensile strength of as-spun fibers was achieved at an intermediate take-up velocity.

Oral (S03-291, Time: Thursday 10:40, Room: Olma)

The main factors influencing glass fiber shortening in the processing of bulk molding compounds (BMC)

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Plastics are growing in importance as substitutes for metallic materials and the associated resource efficiency. Due to their special properties, such as low density, low shrinkage and ceramic surfaces, BMCs have the potential to contribute to a significant reduction in component weight as a metal substitute in highly stressed applications. In particular, the mechanical properties depend on the fiber length achieved in the component. Latest investigations show that these fibers are significantly shortened due to high shear stress during injection molding process. As a result, the lightweight potential of the BMCs and the resulting resource efficiency are not fully exploited. Kunststofftechnik Paderborn (KTP) is analyzing the three main factors, such as material composition as well as plant technology and processing parameters influencing glass fiber shortening in the BMC injection molding process. The main objective here is the development of solutions for processing BMC in a fiber-friendly way. The investigations on the influence of the plant technology and the process parameters on the glass fiber length reduction show that the glass fibers already have an average fiber length of approx. 1 mm after the plugging unit. Along the processing path up to the component, the fibers are shortened from an initial state of 2.3 mm to less than 0.5 mm, which corresponds to a reduction of almost 80 %. Furthermore, the investigations have shown that some BMC additives have a significant influence on glass fiber damage during processing. In particular, calcium carbonate, which is usually used to reduce costs and shrinkage, produces clearly visible surface damage on the cladding surface of the glass fiber and thus contributes significantly to the shortening of the glass fibers in the injection molding process.

Oral (S03-292, Time: Thursday 11:00, Room: Olma)

Simultaneous polymerisation and wetspinning of thermosetting polydimethylsiloxane monofilament with circular section for transparent composite

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Polydimethylsiloxane (PDMS) is a soft, biocompatible, transparent, easy to cast thermosetting polymer which founds many applications in microfluidics or biomedical fields. Currently this material is not available on the market in filament form, due to the difficulty of processing it. Few literatures explore filamentary forming simultaneously with the polymerization of thermoset. One of them explores the "thermally induced wet spinning method" but in a non-circular cross section. The production of continous PDMS monofilament with a circular cross-section suitable for weaving process is investigated. The produced monofilament is inserted in a woven reinforcement, and is impregnated in PDMS matrix. With the optical index matching of the matrix and the produced monofilament woven in reinforcement, the resulting composite is transparent. To produce the monofilament, the PDMS solution is conveyed through a needle over a heating oil bath. The heating oil bath shapes and cross-links the PDMS solution into a circular monofilament. Monofilaments of several meters in length with diameters between 0.25 and 1.5 mm are produced. Different parameters are involved in the production of PDMS monofilament: the flow rate of the solution, the dimension of the needle, the temperature of the oil bath and the duration of stay in the oil bath. The impact of each parameter on the morphological and mechanical characteristics of the PDMS monofilament is studied.

Oral (S03-322, Time: Thursday 11:20, Room: Olma)

Fabrication of the colorimetric sheath/core type polyamide 6-RhYK/polypropylene bicomponent fiber sensor for acid gas detection

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Colorimetric sensors for hazardous gas detection are attracting a lot of attention due to their convenience, low cost, and green materials with non-power operation. In this study, the colorimetric sheath/core (S/C) type Polyamide 6 (PA6)-RhYK/Polypropylene (PP) bicomponent fiber for acidic gas detection was fabricated by melt-spinning process without any dyeing or printing process. The gas sensing performance of the S/C type PA6-RhYK/PP bicomponent fiber with various colorant concentrations and fiber drawing ratio was investigated under the exposure of HCl gas (1, 10, 100 ppm). The durable properties of the reused knitted fabric using our colorimetric bicomponent fiber were demonstrated by washing, cycle, and weather resistance test. From these results, we confirmed the applicability of the colorimetric bicomponent fiber-based sensor to clothing through reusability and reliability evaluation, and proved that it can be a useful method for detecting harmful gases in industrial sites and daily life.

Oral (S03-372, Time: Thursday 11:40, Room: Olma)

Polymeric core-shell and mono-material fibers for concrete reinforcement

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A reinforcement of concrete structures is needed as concrete provides a high compression strength but a low tensile strength. The usually used reinforcing material is steel. However, the corrosion of steel bars over time causes an issue e.g. weakening of the structural strength. One solution to counteract this problem is the use of non-corrosive materials like polymer fibers. A polymer fiber has to fulfill two requirements to be suitable as concrete reinforcement – high mechanical properties and a good bonding ability to concrete. The mechanical properties of the fibers can be influenced by polymer drawing and the resulting molecular orientation. Changes of the bonding ability can be achieved by the fibers physical structure e.g. mechanical anchoring due to crimping or embossment and its chemical structure e.g. the use of materials which allow a formation of hydrogen bonds. Two polymers, polypropylene and aliphatic polyketone are tested within this study, first mono-material fibers of each material are produced and tested, later the materials are combined in a core-shell fiber. All fibers are produced by standard extrusion or coextrusion and a later drawing process. The mechanical properties are determined by tensile tests. The calculation of interfacial shear strength from single fiber pull-out tests allows an evaluation of the bonding ability. Additionally, fiber surface before and after pull-out is examined using reflected light microscopy. Contact angle measurements are done to evaluate possible influences of the surface energy and polarity. Density measurements are used to compare weight potential of the different mono-material and core-shell fibers. The results show good mechanical properties for all fiber materials. The interfacial shear strength is higher for fibers with polyketone at the fibers surface, which can be explained by higher surface energy and polarity of the polyketone compared to polypropylene and different surface deformation during pull-out.

Oral (S03-373, Time: Thursday 12:00, Room: Olma)

Soft Multi-Material Magnetic Fibers and Textiles

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Magnetically responsive soft materials are employed, in principle, in minimally invasive procedures for their untethered, safe, and easy-to-engage in situ human intervention. However, constructing highly integrated magnetic fibers with extreme aspect ratios, that can be used as steerable catheters, endoscopes, and smart textiles still remain untapped. In this talk, we will present multi-material thermal drawing as a material and processing platform to manufacture 10s of meters long soft, ultra-stretchable, yet highly resilient magnetic fibers with a core diameter as low as 300 μm and an aspect ratio of 100,000. We maintain a high viscosity during drawing that enables the magnetic composites to sustain the large stresses and preserve structural integrity. Moreover, high viscosity (ranges between 10,000 to 100,00000 Pa.s) slows down thermal reflow driven by surface tension and conserves micro-scale geometrical features and textures during preform-to-fiber thermal drawing. We process magnetorheological elastomers with varied thermoplastic elastomer matrices indicating rheological traits compatible with thermal drawing but varying mechanical and magnetic properties, resulting in tunable actuation performance. Thanks to the computed tomography data, multi-material thermal drawing as a materials and processing platform is demonstrated to improve 30 times the distribution, percolation paths, and in particular the formation of magnetic particles into fibers than the preform stage. Thermally-drawn soft magnetic fibers can lift 370 times their own weight, withstand strains of >1000 %, and potentially be used for targeted drug delivery applications, while amenable to weaving into functional textiles. The magnetic textile can be programmed to selectively shape morph and can work optimally in extreme environments, for example, after running over by a car several times, and also can sustain after many machine-wash cycles. Magnetic textiles pave the way for assisting patients with rehabilitation.

Oral (S03-408, Time: Wednesday 14:50, Room: Olma)

Bicomponent spinning of biodegradable polymers: Melt-spun PHBV micro fibers

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Biodegradable polymers have gained lots of interest and publicity. However, narrow processing window limits the application of such novel polymers. Polyhydroxyalkanoates (PHAs) are member of biodegradable thermoplastic polyesters, biologically derived by bacterial fermentation. These biodegradable, compostable and biocompatible polymers are of great interest in the production of medical textiles, disposable materials and packaging. Slow crystallization rate and low thermal stability of PHAs limits the melt spinning of fibers and results in inadequate mechanical properties. Among PHAs, poly(3-hydroxybutyrate)-co-(3-hydroxyvalerate) PHBV is more flexible and easier to process due to lower crystallinity. Therefore, in this study, we have used bicomponent spinning method to produce melt-spun PBBV fibers. As a semi-crystalline polymer with slow crsytallization rate and low thermal stability, PHBV remains tacky during drawing in the spinline that hinders the spinnability. A very important advantage of the bicomponent spinning is that it could help a material that could not be spun into a fiber single form. For this study, the two polymers coaxially combined in a spinneret for bicomponent multifilament with core/sheath-geometry. The co-extrusion of a PHBV core and a polyvinyl alcohol (PVA) sheath having a higher thermal stability and faster crystallization enabled the stable winding of filaments. Following the filament production, the filaments were soaked in distilled water to remove PVA sheath and PHBV micro fibers were obtained. Thermal analysis obtained revealed that the complete removal of PVA was possible. Microscopic analysis showed that PHBV microfilaments (single filament diameter < 7 μm) have been successfully melt-spun.

Oral (S03-433, Time: Thursday 14:10, Room: Olma)

Sustainable manufacturing of fabric supercapacitors from biowaste for wearable applications

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Sustainable and environmentally friendly activated carbon from biomass is proposed to produce supercapacitors from banana peels, which has the potential to replace the unsustainable and cumbersome process from graphite or/and fossil fuels. To determine the potential of using banana peels for supercapacitors, the raw banana peels, a biowaste, were activated both mechanically and chemically to observe the actual differences between the activation processes. The sample was thermally treated at 700 °C and then chemically treated with KOH. The activated banana peels were characterized by molecular spectroscopy (FTIR and Raman), differential scanning calorimetry (DSC), dynamic light scattering (DLS), thermogravimetric analysis (TGA) and X-ray diffraction (XRD), scanning electron microscopy with electron dispersive X-ray spectroscopy (SEM/EDX) and atomic force microscopy (AFM). FTIR analysis verified the presence of hydroxyl, carbonyl and aromatic compounds on a banana peel cellulose-based carbon. The TGA results showed that 700 °C was sufficient to completely carbonize banana peels. DLS clearly showed the difference between the carbonized and KOH-activated material in particle size distribution. Meanwhile, surface area analysis by BET showed an increase from 553.862 m^2/g to 565.024 m^2/g when the carbon was activated with KOH with nitrogen isotherm at 77.350 K. The surface area analysis by BET showed an increase from 553.862 m^2/g to 565.024 m^2/g. The specific capacitance increased from 0.3997 F/g to 0.821 F/g, indicating that the specific capacitance increases by more than 100% due to KOH activation. The X-ray diffraction results showed the patterns of an activated carbon. The results indicate that banana peel waste can be used as low-cost and sustainable materials for the fabrication of flexible supercapacitor batteries.

Oral (S03-436, Time: Thursday 14:30, Room: Olma)

Soft electronic polymer fibers for mechanical sensing applications

Laperrousaz Stella[1], Leber Andreas[1], Dong Chaoqun[1], Richard Inès[1], Sorin Fabien[1]

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With the growing interest in soft robotics, smart textiles and wearables, arises the need to develop functional materials combining electrical conductivity with mechanical compliance to design soft electrodes and mechanical sensors able to conform to high curvatures and sustain large deformations. Nanocomposites polymers made of conductive fillers, such as metal nanoparticles or carbon nanotubes, dispersed in elastomer matrices embody the common strategy applied by researchers to meet this issue. However, it remains challenging to create devices combining such soft functional composites into complex 3D architectures, in a scalable and cost-effective way. Here we will present how we can leverage the unique attributes of the thermal drawing process, the approach used to fabricate optical fibers, to realize in a controlled way extended length of soft and stretchable electrically conductive composites embedded in a polymer fiber. By combining the thermoplastic elastomer poly[styrene-b-(ethylene-co-butylene)-b-styrene] (SEBS) to stretchable and conductive composites, 10s of meters-long soft and functional fibers with complex cross-sectional architectures are designed. Through a detailed investigation of the nanocomposites flow behavior, a novel rheological criteria to assess their compatibility with the thermal drawing process is proposed. In addition, by a careful control of the thermal drawing parameters, such as temperature, the nanocomposite resistivity can be adjusted to targeted sensing mechanisms such as resistive or capacitive based-sensing. In this talk, we will present our latest materials and designs, resulting in fibers sensitive to specific mode of deformations, such as bend, pressure or stretch, relevant for textile, soft robotics and biomedical applications.

Oral (S03-491, Time: Wednesday 15:10, Room: Olma)

Development of Mosquito repellent-UV protective polyester using a Disperse dye synthesized from 4-hydroxy coumarin

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The development of functional textile fibres using novel routes is an urgent requirement. Mosquito repellent textile is one of the best choices for manufacturing protective clothing. Polyester fibre is widely utilized in apparel and technical textile applications. In the present work, a novel disperse dye was prepared by reacting diazotized sulfadiazine with 4-hydroxy coumarin. The developed dye was characterized by Fourier-transform infrared spectroscopy (FTIR), 1H NMR,13C NMR, CHN analyzer, and UV-vis spectroscopy. The prepared disperse dye was utilized for the dyeing of polyester fabric. The exhaustion (%), fixation (%), and coloration properties (L*, a*, b*, C*, K/S) were studied. Wash-durability of dyed fabrics against repeated laundering treatments was additionally determined. The standard methods were used to measure the fastness and functional properties (mosquito repellency and UV protection) of dyed polyester. The dyed polyester indicated wash-durable mosquito repellency (100% after 20 washes) and excellent UV protection. X-ray diffraction (XRD) and thermogravimetric analysis (TGA) of dyed fabrics were also done to characterize the dyed polyester.

Oral (S03-531, Time: Thursday 16:00, Room: Olma)

Surface Replication and Mechanical Properties of Cellulose Nanofiber Composites by Thermal Imprinting Process

Ueda Tsubasa[1], Nemoto Akihiko[1], Ishigami Akira[1], Kobayashi Yutaka[1], Ito Hiroshi[1]

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The thermal imprinting process is one of the promising technologies for fabricating micro/nanoscale integration systems being currently applied in wide applications and various fields for fabricating fine patterns in large areas. However, several defect patterns in thermal imprinting have been reported due to polymer flow, demolding, and thermal expansion. One of the difficulties is the demolding process because defects are also influenced by polymer-to-mold adhesion. To overcome the mold adhesion problem, the mold's surface is treated with a fluorine monolayered polymer to decrease the polymer-to-mold adhesion energy. On the other hand, forest resources have been expected to reduce the environmental issue and be utilized as sustainable resources especially cellulose nanofibers (CNFs). CNFs have diverse and fascinating features, such as high strength with lightweight, large specific surface area, low thermal expansion, and eco-friendliness. Therefore, CNFs have a high potential as alternative materials to replace carbon or metal-based fillers for various industries and consumer applications. In this work, the effects of the CNF addition on the transferability of microstructure formation by thermal imprinting process were revealed. The defect of microstructure parts was not observed when the CNF compounding ratio was 5 wt%. Transferability showed an improving trend as increased in the amount of CNF. It is suggested that the addition of CNF can improve the processability of the thermal imprint process.

Oral (S03-561, Time: Thursday 16:20, Room: Olma)

Linkage Effect of Solution-Processable Arylamine-Based Polymers on the Electrochromic Energy-Harvesting Behaviors

Shao Yu-Jen[1], Liou Guey-Sheng[1]

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Nowadays, some researchers are focused on emerging electrochromic (EC) and supercapacitors (SC) to develop electrochromic supercapacitor (ECS) materials that can easily monitor the capacity content by color change. Metal oxides, small molecules, or conjugated polymers are primarily used to apply in ECS. For polymer systems, most of the research mainly utilizes the electrodeposition method to prepare the conjugated polymeric electrode because the resulting films can generate the mesoporous structure simultaneously. However, these polymers suffer from a low optical contrast ratio (ΔT), a waste of starting materials and struggle with large-scale manufacture. Recently, we have discovered a new class of ECS materials composed of solution-processable triarylamine-based polymers that exhibit remarkable charge-discharge capability with high specific capacitance and distinct color changes. Nevertheless, there is still room for improvement in terms of charging retention. In this work, we study a new class of solution-processable non-conjugated arylamine-based polymers with different linkages in the polymer backbone to evaluate the effect on energy-harvesting properties of specific capacitance (Csp) and retention capability, respectively. The results demonstrate that the triarylamine-based polymers with multi-electroactive nitrogen centers significantly promote the charging-discharging retention capability over the outstanding 5000 cycles both in the first and second oxidation stages with distinct color changes simultaneously during the charging-discharging procedure. Furthermore, introducing the electron-withdrawing linkage (such as sulfone or ketone) or intensive donor-acceptor charge transfer structure (such as polyimide) expect to substantiate the higher Csp and energy density for the supercapacitor application. Besides, it is worth mentioning that the Csp of these materials demonstrates superior performance to those of polymeric ECS materials to date.

Oral (S03-565, Time: Friday 11:00, Room: Olma)

Microfluidic wet spinning of soft polymer optical fibres

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Polymer optical fibres (POFs) processed from soft elastomer materials such as polydimethylsiloxane (PDMS) provides a versatile platform for sensory applications ranging from healthcare monitoring to soft robotics [1]. In a previous study based on moulding of POFs, we have demonstrated a soft POFs based textile integrated sensor for continuous pressure monitoring application [2]. Soft materials such as crosslinkable polymer systems cannot be spun into fibres with techniques such as thermal drawing, melt spinning or co-extrusion. Here, we propose a novel strategy called microfluidic wet spinning (MWS) for the spinning of crosslinkable soft polymer materials. A modular microfluidic chip with co-axial capillary design, consisting of core and sheath flow channels was developed for the MWS [3]. The flow rate optimization of sheath polymer solution (sodium alginate) and core polymer (PDMS) led to the formation of PDMS fibres encapsulated in sodium alginate shell inside the wet spinning bath. The crosslinking of PDMS inside the sodium alginate shell and further removal of the shell provided PDMS POFs. The swelling behaviour, optical, mechanical and surface properties of the produced soft POFs were studied to establish the processing-property relationship. The soft PDMS POFs depicted successful light transmission at the wavelength of 660 nm. The scope of this method is not only limited to the PDMS based POFs, but also can be applied to spin other crosslinkable polymer systems for sensing different stimuli such as pressure, strain, human body motions and others.
References [1] Bai, Hedan, et al. "Stretchable distributed fiber-optic sensors." Science 370.6518 (2020): 848-852. [2] Sharma.K et.al. PDMS based polymer optical fibres: From processing-property relationship to sensing applications (Submitted Manuscript). [3] Wei et al. Microfluidic-based wet spinning of individual solid polymer fibers. Patent application, EP22/17.

Oral (S03-593, Time: Friday 10:40, Room: Olma)

Silk Revitalization for Bactericidal Suture Applications

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Solid fibroin fibers (FFs) were directly cross-linked under visible light at room temperature for the first time. The chemical cross-link through dityrosine connection was confirmed by Fourier transform infrared (FTIR) spectroscopy, fluorescence spectra, and a solubility test. The resultant cross-link density of fibers was calculated based on their swelling ratio evaluation in LiBr solution. Further applying stretch to the fibers during irradiation increased the fiber strength to higher values. The break stress and Young's modulus of photo-cross-linked FFs reached a 60−90% increase in comparison to the original FFs in dry and wet conditions. This easy strengthening approach is scalable industrially to enhance FFs in a wide range of applications. Additionally, double network structure was fabricated in solid FFs via photo synthesis of bactericidal copolymers at room temperature. It was also effective in silk yarn suture processing, and successfully produced bactericidal silk sutures with greatly enhanced strength even in wet conditions. Keywords: silk fiber, photo crosslink, molecular orientation, bactericidal suture Acknowledgement We would like to thank the financial support General Research Fund PolyU152156/17E and PolyU152189/21E from the Hong Kong Research Grant Council.

Oral (S03-648, Time: Friday 12:00, Room: Olma)

Effects of extrusion variables on the crystalline structures and mechanical properties of the melt spun fibers from recycled Polypropylene copolymer

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High-speed melt-spun fibers from a 'model' recycled polypropylene (PP) copolymer material (ethylene content of 9%) were produced at two different extrusion temperatures (190 and 230 deg.C) and varied take-up velocities (0.5 to 5.0 km/min), and their structure and mechanical properties were compared with the fibers from virgin PP copolymer produced at the same spinning condition. For both extrusion temperatures, for the identical take-up velocities, mechanical properties for the fibers from virgin and recycled PP copolymers were very similar. At the identical lower velocities of 0.5 and 1.0 km/min, elongation at break and tensile strength for the extrusion temperature of 190 deg.C were similar to the extrusion temperature of 230 deg.C, whereas elongation at break were slightly higher and tensile strength were slightly lower for the identical higher take-up velocities for the extrusion temperature of 190 deg.C. Effect of extrusion temperature on initial tensile modulus was not clear. Structure analysis revealed that birefringence for the fibers from virgin and recycled PP were similar for both extrusion temperatures. Though birefringence for both extrusion temperatures were nearly similar for the take-up velocity of 0.5 km/min, the birefringence for the extrusion temperature of 190 deg.C were noticeably higher for the take up velocities of 1 and 2 km/min, than for the extrusion temperature of 230 deg.C. Crystalline structure analyzed through the X-ray measurement revealed that the crystallinities for the fibers from virgin and recycled PP were similar, however crystallinities for the extrusion temperature of 190 deg.C was slightly higher than for 230 deg.C for the identical take-up velocities. Relative amount of c-axis oriented crystals and a*-axis oriented crystals ('daughter' lamellae) was found more sensitive for the difference in structure development behavior. The relative amount of a*-axis oriented crystals were significantly high in the case of fibers melt extruded at 230 deg.C than for 190 deg.C, and more importantly it was found that the amount of a*-axis oriented crystals was noticeably higher for the fibers prepared from recycled PP copolymer.

Oral (S03-669, Time: Thursday 16:40, Room: Olma)

Viscoelastic flow simulation of film extrusion process involving flow-induced crystallization: Investigating role of radiation, natural and forced convection

Zatloukal Martin[1], Barborik Tomas[1]

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Current understanding of the role of flow induced crystallization and heat transfer during extrusion film casting process on the flow stability and resulting precursor film crystallinity is rather limited due to absence of relevant physical model, experimental data and methodologies allowing to reveal key structure–rheology–die design–process conditions–flow stability relations. This significantly limits material and process optimization during fabrication of polypropylene and polyethylene energy storage membranes, which are becoming increasingly more important due to their usage in rechargeable lithium-ion batteries. The situation is further complicated by flow phenomena (such as flow-induced crystallization, neck-in and associated flow instabilities), which are not fully understood yet and significantly narrow the processing window [1]. In order to understand the effect of molecular weight, molecular weight distribution, branching, rheology and process conditions on film crystallinity and flow stability, we have developed and validated viscoelastic non-isothermal model (utilizing modified Leonov constitutive equation, flow induced crystallization, temperature, temperature gradient and melt entropy). We have further generalized the model incorporating variable heat transfer coefficient to understand the role of radiation, natural and forced convection. It is believed that the developed numerical model together with stable numerical scheme as well as obtained research results will help to understand processing window for production of flat porous polymer membranes considerably. References: 1. Barborik, T., Zatloukal, M. Steady-state modeling of extrusion cast film process, neck-in phenomenon, and related experimental research: A review (2020) Physics of Fluids, 32 (6), art. no. 061302. Acknowledgments: The authors would like to acknowledge the financial support from the Grant Agency of the Czech Republic (Grant registration No. 21–09174S).

Oral (S03-682, Time: Friday 11:20, Room: Olma)

Surface-coated high-density polyethylene microfibers as reinforcement for mortar materials

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In the construction area, cementitious derivatives including cement paste, geopolymer, mortar, and concrete are by far the most utilized materials, due to their easiness to fabricate, low expenditure, good performance, and versatile applications. However, these cementitious materials (CMs) are quasi-brittle in nature and possess extremely low tensile and flexural features as compared to their compressive strength and high proneness to cracking. The use of polymer fibers in the mix design of the CMs appeared to be a promising way to tackle the aforementioned concerns over CMs. The correlation between the interface fibers/cementitious matrix and the mechanical performances of the resulting CMs have been established in a few early works. However, there is a clear lack of extensive studies on this aspect, and most of them rely on the use of invasive and costly surface modification procedures on the fibers, typically oxidations, and have resulted in mitigated results. Hence, this work aims to address the aforementioned limitations by considering the use of surface-modified high-density polyethylene (HDPE) microfibers as reinforcement for mortar matrix. The microfibers were produced using a melt-spinning spunbond system and the surface modification consisted of coating the microfibers with metakaolin particles, in a water medium, to improve the interface with the cement particles using a cost and environmental-friendly modification approach. The effect of the surface modification done on the HDPE microfibers was evaluated through bending, split tensile and compressive tests conducted on the reinforced mortar specimens. The correlation between the mechanical properties and the surface modification was investigated through structural analysis including FTIR, and XPS, as well as morphological visualization using SEM.

Oral (S03-686, Time: Friday 11:40, Room: Olma)

Antiplasticizing interactions between biobased small molecules and vinyl polymers for improved gel-spinning: A molecular dynamics simulation study

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Petrol and biochemical plasticizers are incorporated into polymers to aid processability. Plasticizers reduce polymer-polymer interactions increasing free volume allowing for better chain mobility at elevated temperatures. Upon cooling, the reduction of secondary bonding disrupts crystallization, reducing mechanical performance. This is problematic for the fabrication of high performing fibers as secondary bonding is crucial for molecular order. Antiplasticizers similarly improve processability by increasing segmental chain motion while 'strengthening' the polymer matrix for improved mechanical performance. To gain atomistic insights of antiplasticization in PVA, that is quantify interactions and structural changes previous theorized, amorphous models of PVA containing one of six selected lignocellulosic derivatives (glycerol, sorbitol, glucaric acid, mucic acid, tartaric acid, succinic acid) were studied over a 0.8-10wt% range using molecular dynamics simulations. Additive size and group functionality, interaction energies and radial distribution functions of PVA/additive systems were considered and correlated with the ability to form hydrogen bonds with PVA chains. Known plasticizers, glycerol and sorbitol, were observed to interact similarly to the known antiplasticizer, glucaric acid, at low concentrations (<3wt%), but did not coordinate with hydroxyl pendant groups at the same frequency. Di-carboxylic acids similar to glucaric acid in functionality but differing in size and polarity were also studied over a concentration profile of 0.8-10%, most notably mucic acid, the trans conformer of glucaric acid. This computational approach agrees with experimental work on PVA/GA fiber systems and further supports the concentration-dependent and interaction-driven phenomenon of antiplasticization discussed in the literature.

Poster (S03-285, Time: Thursday 17:00, Room: Foyer)

Development of regenerated cellulose nonwoven fabric from wet-type spun-laid spinning

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Most countries are striving to become an eco-friendly and more sustainable economies, resulting in a high level of interest in developing new materials from wood, pulp and other cellulose-based renewable raw materials. Among them, the demand for nonwoven fabric materials is particularly high. On the other hand, the method for manufacturing regenerated cellulose nonwoven fabric through the spun-laid process is very limited, and the only commercially successful product so far is the product name 'bemliese'. Since most cellulose-based raw materials are insoluble, reforming these raw materials into continuous fibers requires technical solutions to the entire process from raw material melting, extrusion, drawing and web-forming. In the case of cellulose, the solvent is dissolved in N-methyl-morpholine-N-oxide (NMMO), and then a direct spun-laid nonwoven fabric development technology based on solution spinning is being studied. In this study, a wet type spun-laid spinning system equipped with a single concentric spinneret was constructed to manufacture a direct spinning cellulose nonwoven fabric by modifying the wet-based lyocell producing method, and cellulose spinning nonwoven fabrics were produced under various process conditions. The effect of processing conditions such as temperature, melting concentration, and spinning distance on spinning was studied, and the fiber shape and physical properties of the lyocell nonwoven fabric were also studied. As a result, the higher the dope concentration and the shorter the spinning distance (DCD), the larger the fiber diameter. As the spinning distance increased, the fiber fineness progressed.

Poster (S03-503, Time: Thursday 17:00, Room: Foyer)

Investigation of the Effect of AcryloPOSS Loading Ratio on the Properties of LLDPE Fibers

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Polyethylene (PE) fibers have superior properties such as high strength, modulus, and lightweight. Due to its advantages, it finds applications in many fields, such as ballistic protectors, sports equipment, maritime, aviation, and medical supplies. Recently, polyhedral oligomeric silsesquioxane (POSS) nanoparticles have been frequently used as a reinforcing agent due to their potential to increase the thermal stability of polymers and improve their mechanical properties. In this study, it was aimed to investigate the effects of different loading ratios of acrylo POSS (AcPOSS) on the thermal, mechanical, crystallographic, and morphological properties of linear low-density polyethylene fibers (LLDPE). For this purpose, the AcPOSS loading rate was considered as 2, 4, 6, and 8 phr. LLDPE and AcPOSS were melt compounded at 145°C in an Xplore model laboratory twin-screw microcompounder. Then, they turned into fibers with the Xplore model laboratory scale fiber drawing system. Various characterization methods of LLDPE/AcPOSS fibers have been used, including tensile tests, differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), X-ray diffractometry (XRD), polarized optical microscope (POM), and atomic force microscopy (AFM). DSC and XRD test results revealed that the addition of AcPOSS increased the crystallinity of LLDPE fibers. The mechanical test results showed that the tensile strength decreased depending on the increasing AcPOSS ratio. Homogeneous distribution of AcPOSS in LLDPE was observed at lower content of AcPOSS from AFM analyses. However, some coarse dispersion of POSS molecules was determined as the POSS content increased.

Poster (S03-590, Time: Thursday 17:00, Room: Foyer)

Evaluating the sustainability of antibacterial activity of antibacterial polymer material using LCNF as a retainer

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With the increase in cleanliness and awareness of hygiene, the use of antimicrobial films is increasing due to the increased frequency of disinfection for items and places that are touched by an unspecified number of people. However, it is estimated that 8 million tons of plastic waste is discharged from land into the ocean annually worldwide due to improper disposal, and there is concern about the impact on the ecosystem. Under these circumstances, it is necessary to develop environmentally friendly antimicrobial materials. In this study, we investigated biomass-derived and biodegradable cellulose acetate butyrate (CAB) was used as a resin matrix, Hiba oil as a natural antimicrobial agent, and lignocellulose nanofibers (LCNF) as a retention agent to reduce environmental burden. The objective was to develop polymer films that reduce the environmental burden and improve the durability of the antimicrobial effect by adding Hiba oil as a natural antimicrobial agent and Ligno cellulose nanofiber (LCNF) as a retention agent. Various concentrations of Hiba oil and LCNF were added to CAB solutions prepared by dissolving them in chloroform as a solvent. After stirring, films were formed by a solvent casting method. The antibacterial activity was evaluated using the antibacterial activity test method (JIS L 902: 2015) for quantitative evaluation and the shaking method (antibacterial activity evaluation test method II: 2020) for qualitative evaluation. We will report detailed results on the antibacterial performance and antibacterial activity retention rate of the produced film.

Poster (S03-725, Time: Thursday 17:00, Room: Foyer)

Smart Fibers and Films for Sustainable Technical Textiles and Food Packaging Concepts

Lübben Jörn Felix[1], Gu Jiayan[1], Kloss Svenja[1], Bucher Matthias[1], Schmid Markus[1]

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The main objective of this work is to develop intelligent chemical, temperature and moisture-switchable functional and indicator layers on fibers and films that can be used in technical textiles, for example to improve wearing comfort. It is also possible to use them in intelligent packaging that can detect whether the food inside is still edible, thus helping to avoid unnecessary food waste. Suitable variable-volume fibers are to be developed in order to produce reversible and reproducible switchable filters and membranes with regard to heat and mass transfer. The thermoresponsive polymers (TRP) used for this purpose react in solution to temperature changes in their environment with conformational changes, which manifests itself in collapse or expansion behavior within a certain temperature and humidity range. TRPs were immobilized on synthetic metallized monofilaments using a linker to investigate whether the shape changes could be observed outside solvents under different thermophysiological temperature and humidity conditions. The functionalized fibers and films were characterized by molecular spectroscopy, differential scanning calorimetry (DSC), electron microscopy, atomic force microscopy, contact angle measurements, quartz crystal microbalance, and dynamic mechanical analysis. In order to produce fibers with optical indicator properties that could find application in packaging technology, synthetic fibers were functionalized with colored metal complexes. Suitable metal complexes were used to produce color indicators that change color in the presence of degradation products of protein-rich foods, allowing the resulting textile to be used as a freshness indicator in food packaging. The functionalized fibers and films were characterized by colorimetry, gas permeation measurements and correlated microbiological tests.

Plenary (S04-805, Time: Friday 09:10, Room: Sulzer Chemtech)

Innovative developments in the area of Polymer Processing with recent commercial impact

Altstädt Volker[1]

[1]University of Bayreuth, Bavaria, Germany

The James L. White Innovation Award of The Polymer Processing Society is awarded for innovative developments in the field of polymer processing technologies with recent commercial impact. It aims to recognize originality, innovation and creativity among researcher(s) or inventor(s) in the science and technology of processing polymers and polymeric products.

Volker Altstädt has been actively developing innovative processing chains for plastics production in close cooperation with industry for more than 25 years. The aim has always been to make the processes more cost-effective and sustainable, and to reduce the weight of materials in the components to enable lightweight designs. The process developments were always accompanied by material analyzes to gain a basic understanding of the material behavior when optimizing the process chain using structure-property relationships.

Three important examples with industrial impact will be presented in the lecture. One example ist related to sport industry, the second example is related to composite industry and the last example on bead forms is related to packaing and construction industry.

Keynote (S04-212, Time: Tuesday 10:15, Room: Monosuisse)

Separation of metal/polymer composites' interface by gas bubble nucleation for multi material products' recycling

Taki Kentaro[1], Mori Yuto[1], Sharma Rajesh Kumar[1], Miyata Ken[1], Ito Hiroshi[2]

[1]Kanazawa University, Ishikawa, Japan
[2]Yamagata University, Yamagata, Japan

Multi material products have rigid interfaces of component materials to improve products’ durability and reliability. However, the rigid interface makes it difficult to material-recycle the products because separation of two materials by breaking the interface is more difficult for more rigid interfaces. In this study, we have developed a new technique to material-recycle of composites with the aid of physical foaming techniques.

Keynote (S04-213, Time: Tuesday 13:45, Room: Monosuisse)

Stretching-induced Foaming of Gas-laden Thermoplastic Elastomer

Ohshima Masahiro[1], Lin Weiyuan[1], Hikima Yuta[1]

[1]Kyoto University, Kyoto, Japan

Stretching-induced foaming of gas-laden thermoplastic elastomer (TPE), styrene-isobutylene-styrene (SIBS), was successfully conducted. CO2-laden TPE plates were prepared by an injection molding process without foaming and later foamed by stretching. The effects of CO2 concentration in the gas-laden elastomer, stretching rate, and strain rate on bubble diameter and bubble density of the resulting foams were investigated, and a new bubble nucleation rate model was proposed to reflect the stretching effect on bubble nucleation in foam. The model has the stretching rate-dependent elastic strain energy in the bubble nucleation rate to explain that the higher stretching rate and the higher strain produce more bubbles of smaller size. The proposed bubble nucleation model clarifies the effect of the elastic modulus of TPE on the bubble nucleation barrier.

Keynote (S04-353, Time: Wednesday 10:15, Room: Monosuisse)

Improvement of Foamability and Properties of PC Foams by Governing the Crystallinity of PC Foaming with the Existence and Advantage of the Rubbery Nanofibrillar Network

Park Chul B.[1], Akrami Hamidreza[1]

[1]University of Toronto, Ontario, Canada

This study is focused on developing advanced manufacturing technologies for increasing the foamability of low-density PC foams and simultaneously enhancing the mechanical properties of PC foams by governing crystallinity. In this research, we take advantage of in-situ nanofibrillation as an efficient, low-cost, and environmentally friendly tailored technique for the boosting melt strength of polycarbonate (PC) and other properties. PC/ Ethylene Propylene Diene Monomer Rubber (EPDM)-fibril composites are prepared by a fiber-spinning system. Benefits from the crosslinked rubber phase as well as nanofibrillation processing play the main roles in property improvement, developing uniform crystallinity, and therefore; enhancement of expansion ratios and cell densities due to increasing the strength of the skin in foaming. The strain hardening response exhibited by the fiber-spun prevents cell-wall rupture during cell growth due to the viscosity increase with strain. Furthermore, Flow-induced crystallization behaviour of second-phase fibrils on the main phase in the form of a trans-crystalline layer during cell growth. By controlling the crystallinity with the presence of a nanofibrillar network and timing, the uniformity of cells highly increases and therefore, makes PC foams stiffen. Consequently, a larger amount of blowing agent is retained within the fiber and up to a much higher expansion ratio is seen compared to the normal PC.

Oral (S04-036, Time: Tuesday 10:40, Room: Monosuisse)

Super-Thermal Insulating Polyimide-Graphene Composite Aerogels with Exceptionally High Surface Area for High-Temperature Applications

Aghababaei Tafreshi Omid[1], Ghaffari-Mosanenzadeh Shahriar[1], Rastegardoost Mohammad Mahdi[1], Saadatnia Zia[1], Park Chul B.[1], Naguib Hani E.[1]

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Aerogels are referred to a unique class of nano-porous materials with outstanding properties, namely ultralow density, excellent optical transparency, and low dielectric properties. Unlike silica-based inorganic aerogels, which suffer from brittleness, polyimide (PI) aerogels possess enhanced mechanical flexibility and robustness. This feature, along with their outstanding thermal stability, makes PI aerogels favorable for high-performance industrial applications. Recent studies showed the addition of nanofillers into PI aerogel matrix would be an effective strategy for improving their properties for targeting various applications, such as pressure sensors, and microwave absorption. However, due to the sensitive relations between contributing parameters in aerogel synthesis, improvement of a specific property result in deterioration of another property, which can limit their functionality. To address such challenge, this study presents fabrication of a novel PI composite aerogel through incorporation of graphene-based nanoparticles into nano-porous structure. It is demonstrated that addition of graphene is a successful technique in controlling the shrinkage of aerogels, and upon addition of only 0.5% GNP, shrinkage is reduced from 16% to only 6%. Nitrogen adsorption/desorption analysis of aerogels further shows that specific surface area (SSA) can be effectively improved, and the highest SSA of ~400 m2/g is obtained. Moreover, the combination of low density and GNP effect in interrupting the heat transfer in composite aerogels results in exceptionally high thermal insulation of 25.47 mw/mK. Eventually, it is shown that due to the shielding effect of layered structure in graphene, thermal stability is improved, and high onset of decomposition of 610 °C is obtained. Considering the rapid development of thermal management industry, proposed composite aerogels can have widespread applications in advanced energy materials, thermal insulation, and aerospace.

Oral (S04-043, Time: Tuesday 11:00, Room: Monosuisse)

Fleece Composites as Membrane in Alkaline Electrolyzers

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As chemical feedstock, energy storage or plain, shipping or truck fuel, hydrogen (H2) potentially offers all the opportunities in contributing to reach the zero carbon dioxide emission targets. Nevertheless, mankind lacks satisfactory technological solutions for emission neutral hydrogen production since polluting the atmosphere comes with no realistic price. Thus, efficiency improvements in hydrogen production from water must be made to shift hydrogen technologies to economic competitiveness. Therefore, the German Institutes of Textile and Fiber Research Denkendorf (DITF) work together with its partners at the Zentrum für Sonnenenergie- und Wasserstoff-Forschung Baden-Württemberg (ZSW), the German Aerospace Center (DLR) and the Hahn Schickard Institutes to develop alkaline water electrolyzers further. In this research association the DITF focuses on the development of membranes, based on polymers and enhanced by polymer-based fleece. State of the art melt blow or ultracentrifuge wet fleece spinning technology is afforded to process polymers with a high-performance nature (like PEEK, PESU) that is underlined by high melting points, high molecular weights and chemical resistance and inertness. The resulting fleece show small fiber diameters (in or even below the micrometer range) and thus a homogeneous and small sized pore-like structure within the fleece material. Doctor blading the fleece with polymer solutions of poly(sulfone)s or polybenzimidazoles form fleece (fiber) composites. These composites show low gas permeabilities, a key property of membrane materials to reach high gas purity and plant safety. In addition, the reachable fleece composites thickness of less than 100 µm allow electrochemically optimal arrangement of the electrolyzer cell and low ohmic resistances. These efficiency improvements can only be made under preservation of the required mechanical properties given by the composite structure.

Oral (S04-087, Time: Tuesday 11:20, Room: Monosuisse)

Evaluation of the potential of polybutylene terephthalate / poly(phenylene oxide) blends for bead foam production

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A lot of development in the field of bead foams can be recognized within the last two decades; this concerns mainly the establishment of novel polymers with higher functionality compared to the classic bead foams (i.e., expandable polystyrene and expanded polypropylene) to enabled a broader field of application. Up to now, bead foams with increased sustainability (e.g., from polylactide), enhanced mechanical properties (e.g., from thermoplastic polyurethanes) or higher thermal resistance (e.g., from engineering and high performance polymers). The latter also sometime come along with an improved flame resistance (e.g., polyetherimide). However, often engineering polymers (polyesters such as polybutylene terephthalate or polyethylene terephthalate) are used, which lead to higher service temperatures but have an unfavorable burning behavior. Besides the addition of flame retardants, a possible strategy to improve the burning behavior is to create blends with intrinsic flame-retardant polymers. PPE - Poly(phenylene oxide) - is one possible material for this. Due to its flow behavior the processing is not trivial. This study aims to understand the rheological and thermal behavior of PBT/PPE blends and to evaluate its potential for bead foam application in a continuous process. Stable processing was possible up to a PPE content of 10 wt. %. The crystallization behavior changes by the addition of PPE and the lowest bulk density for the foamed beads was 130 kg/m3. By using an epoxy-based chain extender the blend morphology can be influenced and a fine and homogeneous distribution of PPE in PBT can be achieved.

Oral (S04-102, Time: Tuesday 11:40, Room: Monosuisse)

Understanding chemical foaming of highly filled ceramic feedstocks for injection molding

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As energy efficiency is becoming an increasingly important factor today, lightweight material concepts experience a significant increase in relevance. This is especially true for thermal processes such as the sintering of powder metallurgically processed metal or ceramic parts that often require temperatures of up to 1600 °C or even higher. Since these processes involve a high amount of energy input, there is also a high potential for saving energy by employing lightweight structures. Here, a significant amount of energy can be saved by reducing the weight of supporting plates that need to be heated up in addition to the parts being sintered. This can be achieved by deploying porous supporting plates through a foaming process that is induced within injection molding. The process behind this is the PIM process, which combines the advantages of the geometric complexity and the efficiency of the injection molding process with the desired material properties of a specific ceramic material. The present work demonstrates the foaming behavior of a feedstock material with varying alumina contents. Here, the filler content was systematically varied between 0 vol.-% and 50 vol.-% in a single component PE-LD matrix. In addition to that, the composite was dry blended with 0 wt.-%, 2 wt.-% or 5 wt.-% of a selected chemical foaming agent prior to injection molding. The molded plates were then evaluated regarding to their pore distributions, pore sizes and filler contents in defined distances to the sprue. It was shown that an increase in filler portion significantly decreases the pore content, while the amount of foaming agent added only had minor impact on the degree of porosity. A rising filler content also resulted in an overall decrease in pore sizes.

Oral (S04-105, Time: Tuesday 12:00, Room: Monosuisse)

Development of temperature-resistant, recycled PET/PBT bead foams using reactive extrusion

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Bead foams make it possible to produce components with complex geometry at low specific weight. In recent years, efforts have been made to develop bead foams from engineering polymers such as poly butylene terephthalate (PBT) or polyamide 6 (PA6) to widen the field of application, e.g., higher service temperatures.1,2 Bead foams are usually produced in a two-step process, the production of foamed particles and the subsequent fusion of these by steam in a cavity to a final part. In the case of semi-crystalline polymers, the formation of a double melting peak during the production of the foamed particles allows the fusion while maintaining the cellular structure.3,4 These multiple melting peaks can be achieved either by annealing or by blending with a suitable partner. For a continuous process, the annealing method is not very suitable, therefore we chose polymer blending of PBT and recycled PET. In this study, the influence of blowing agent pressure and chain extender concentration on the density and cell morphology of rPET/PBT bead foams will be investigated. Furthermore, the influence of different chain extenders (Joncryl, PMDA and Joncryl/PMDA) on the foaming process will be investigated. Chain modifiers are necessary for foaming linear polyesters, especially recycled polyesters, to improve flow properties (increased shear viscosity and melt strength).2,5,6 The experiments were carried out on a tandem extrusion line followed by underwater pelletizing. The composition of the polymer blend and the concentration of chain modifiers were systematically varied. CO2 was chosen as blowing agent It was shown that the concentration of the chain extender has a stronger influence on the bead density than the type of chain extender used. This can be attributed to the influence of the chain extender on rheological properties, e.g., a significant increase of the melt strength.

Oral (S04-165, Time: Tuesday 14:10, Room: Monosuisse)

Polylactic acid bead foams produced by extrusion foaming

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Polylactic acid (PLA) is a thermoplastic polyester produced from renewable sources which has received an enormous attention in the last years due to its biodegradability and biocompatibility. On the other hand, polymeric foams are materials that combine the properties coming from the polymeric matrix with the advantages of a cellular structure. Thus, foams are suitable for applications that require lightweight materials with properties like good thermal insulation or high acoustic absorption without a huge loss in the mechanical capabilities of the material. The foaming of expanded PLA beads (ePLA) is challenging due to the low melt strength and slow crystallization kinetics of the raw materials, which results in a narrow processing window. Therefore, modification of the polymer, use of the appropriate additives as well as correct selection of the process conditions must be done to have a proper foam. In the current work the foamability of ePLA has been explored using the Sulzer's foaming extrusion process. This process represents an alternative to the traditional autoclave foaming technology, which for ePLA has turned out to be a costly technique with unsatisfactory properties in the molding of the beads. The molding of the beads is a challenging process due to its dependence on the crystallization rate and the degree of crystallization of the foams produced. For producing the beads, specific grades of PLA with different D contents were selected, combined with the addition of a chain extender that modify the final viscosity, an appropriate selection of blowing agents (isobutane and/or CO2), as well as the election of the correct process conditions. All these previous parameters have an influence in the final crystallinity and crystallization rate of the foams. The ePLA beads obtained using the Sulzer extrusion process, exhibit low densities (c.a. 20 g/l) with relatively low open cell contents (lower than 15%) and good molding properties, even for complex pieces.

Oral (S04-228, Time: Tuesday 14:30, Room: Monosuisse)

Effect of Polyamide and Crosslinking Agent on the Foam ability of PEBAX Foam

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Polyether block amide (PEBAX) is an engineering thermoplastic elastomer (TPE) known for possessing high impact resistance and significant flexibility over a wide range of temperatures. However, due to their low modulus, most TPEs shrink after foaming. This study investigated the foaming ability of PEBAX via solid-state batch foaming using CO2 as the blowing agent. Polyamide 6 (PA 6) and multifunctional epoxide, Joncryl ADR-4385C (ADR), was added as an anti-shrinkage agent and chain extender to increase the expansion and reduce the shrinkage of PEBAX foam. Before foaming, the mechanical properties of the material were tested. The result shows that adding PA 6 increases the modulus and reduces the tensile strength, while the elongation at the break did not significantly decrease. Still, the presence of ADR reduces the elongation at break significantly. Interestingly, the low temperature impact performance of PEBAX was not affected. The foaming result showed that adding PA 6 improved the foam expansion and reduced foam shrinkage. However, a crack was observed in the foam structure and was eliminated by adding a chain extender. The PEBAX/PA 6/ADR blend shows an expansion ratio of 5.8 times and shrinkage of less than 5%.

Oral (S04-249, Time: Tuesday 14:50, Room: Monosuisse)

Service life analysis of district heat PU pipes via calculation of activation energy

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Accelerated aging test method was developed using a lab scale stainless steel reactor. Variable temperature and air pressure with constant time was used to analyze the effect on service life and activation energy of polyurethane (PU) foam samples obtained from industrial district heating (DH) pipe system. Accelerated aged PU samples were then analyzed using thermogravimetric analysis (TGA). The data obtained from TGA machine were used for analysis and calculation of activation energy (Ea) using Arrhenius equation. From that equation activation energy (Ea) and service life were predicted using modified Chang method. Multiple temperature range (150 °C to 190 °C) were used to analyze and to predict the service life of PU of DH pipe system. As the given temperature in reactor is increased the aging of PU samples also increased and service life decreased. Constant air pressure of 3 bar and constant aging time of 70 hours were used for all the experiments of accelerated aging.

Oral (S04-275, Time: Tuesday 15:10, Room: Monosuisse)

A flexible polypyrrole membrane with ultrahigh areal specific capacitance

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Polypyrrole (PPy) is a conductive polymer with good electrical and electrochemical properties. It has attracted much attention to be used as electrode materials in energy storage devices such as supercapacitors. In this work, the roles of different effectors such as dopant, template, and post-synthesis treatment on the areal-specific capacitance of a flexible PPy membrane have been evaluated. The properties of the membranes were evaluated using a scanning electron microscope (SEM) for morphology, Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) for chemistry, thermogravimetric analysis (TGA) for thermal stability, and cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) for electrochemical activity. The presence of methyl orange (MO) and the treatment of the PPy-MO membrane with sulfuric acid dramatically increased the specific capacitance of the membrane from 2226 mF/cm2 to 6417 mF/cm2. It is likely that the superposition effect of MO and acid treatment helped to enhance the charge-specific capacitance. Furthermore, the following four dopants, i.e., polystyrene sulfonate (PSS), chlorine anions, docusate sodium (AOT), and camphor sulfonic acid (CSA) were investigated in the presence of MO and acidic treatment. The areal specific capacitance of the membranes was 7238 mF/cm2 for chlorine anions, 13264 mF/cm2 for CSA, 19179 mF/cm2 for AOT, and 4458 mF/cm2 for PSS, measured between 0 - 0.8 V at 1 mA/cm2. Based on obtained results, the PPy membrane doped by AOT possesses the highest specific capacitance which makes it a good candidate for use in energy storage devices.

Oral (S04-287, Time: Tuesday 16:00, Room: Monosuisse)

Tuning Thermal performance of Low density and high strength nanofibrillated cellulose/polymethylsilsesquioxane aerogel with surfactant

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Nanofibrillated cellulose (NFC) and polymethylsilsesquioxane (PMSQ) based aerogel had been prepared by the sol-gel method. Low density and high strength nanofibrillated cellulose aerogel were prepared by the freeze-drying method. The goal of the present work is to study the impact of surfactant and base catalyst on the thermal and mechanical performance of the corresponding aerogel. The bulk properties of aerogel were predicted by the rheological premonitory. The chemical structure of the aerogel was studied by Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), and solid-state Nuclear Magnetic Resonance (NMR). X-ray microtomographic analysis (XMT) confirmed the homogeneous and monolithic structure of aerogel. The lowest thermal conductivity was achieved as 23.21 mW/m K with V-0 and HBF rating through UL-94 test. Thermal performance of aerogels was cross-verified through modeling and simulation in COMSOL multiphysics platform. The mechanical properties of aerogel were evaluated by monolithic compression test in axial and radial compression test up to 90% strain, cyclic compression loading-unloading, and reloading test, flexural test and dynamic mechanical analysis. The time-temperature analysis has shown around 5°C temperature difference in the middle of the room after using the aerogel panel at the exposed surface, which assists in the practical application of the synthesized aerogel panel. Nanofibrillated cellulose aerogel would be a candidate in practical applications such as heat insulators, kinetic energy absorbers, and energy efficiency buildings.

Oral (S04-349, Time: Tuesday 16:20, Room: Monosuisse)

Improving Foaming Ability of Polyolefins Using Thermoplastic Elastomer Nanofibrils

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High-density polyethylene (HDPE) exhibits poor melt strength which limits its widespread application especially where it is exposed to an elongational deformation flow in processes such as film blowing, melt spinning, and foaming. In this study, by taking advantage of in-situ nanofibrillation of thermoplastic polyester ether elastomer (TPEE) in HDPE matrix, we improved the rheological properties as well as the foamability of HDPE. TPEE consists of a hard crystalline segment of polybutylene terephthalate (PBT) and a soft amorphous segment of polyether. The polarity of these two groups causes TPEE to be thermodynamically incompatible with non-polar HDPE. Therefore, styrene/ethylene-butylene/styrene copolymer grafted maleic anhydride (SEBS-g-MA) as a compatibilizer was used for reducing the interfacial tension between two blend components. In the first step, a 10% masterbatch of HDPE/TPEE with and without compatibilizer was prepared employing a twin screw extruder. Next, to fabricate fiber-in-fiber composites, the 10% masterbatch was diluted and processed by spunbonding. Scanning electron microscopy (SEM) revealed that not only the spherical size of HDPE/TPEE decreased significantly after SEBS-g-Ma inclusion, but also a much smaller TPEE nanofiber size (60-70nm for 5%TPEE) was achieved. Moreover, the extensional rheological results showed strain-hardening behavior for both compatibilized and non-compatibilized stretched samples at earlier times, at a given extensional rate, compared to the unstretched counterparts. It is worth mentioning that the improvement of extensional rheological properties was more pronounced for compatibilized samples compared to the non-compatibilized ones. This can be attributed to smaller nanofiber size and consequently higher aspect ratio as well as a more robust 3D fibrillated network. Finally, batch foaming was conducted to investigate the foamability of fibrillated nanocomposites.

Oral (S04-385, Time: Tuesday 16:40, Room: Monosuisse)

The dissolution mechanism of a blowing agent in the novel foam injection molding process, SOFIT® (RIC-FOAM)

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Foam injection molding (FIM) using an environmentally benign physical foaming agent (PBA), such as carbon dioxide (CO2) and nitrogen (N2), has been attracting significant attention. Recently, we developed a novel FIM process called SOFIT® or RIC-FOAM that does not need high-pressure PBA pumping or compression system. Instead of the high-pressure gas generation system, PBA is delivered to the molten polymer directly from the gas cylinder. We also developed a near-infrared spectroscopy NIR-probe with high heat resistance (210 oC) and pressure (100MPa). The probe allows us to monitor the blowing agent concentration in foam injection molding processes. Installing the NIR monitoring system with the probes to FIM, where polypropylene was foamed using CO2 as a blowing agent, we investigated the gas-dissolution performance of the newly-developed FIM: The effects of barrel temperature, screw rotation speed, polymer feeding rate, and holding time on the CO2 concentration were investigated. The relationship between these operating parameters and CO2 concentration was analyzed using a starvation zone model.

Oral (S04-420, Time: Tuesday 17:00, Room: Monosuisse)

foAIming: artificial intelligence in foaming

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We illustrate challenges and preliminary results of the foAIming project (artificial intelligence and robotics in polymer foaming) that aims at exploiting robotics and artificial intelligence (AI) methods in the fields of materials science/engineering and chemical engineering to improve foaming processes and to achieve new foams with novel structural and functional properties. In this project, we propose the design and development of a robotic system for the management of foaming experiments that allows: i) the autonomous/interactive conduct of the experiments, ii) the measurement of the properties of the foams, and iii) the analysis, modeling, and tuning of the foaming process. In the proposed system, a robot manipulator is used to handle polymeric samples and products. The robotic platform is managed by an autonomous/interactive control system responsible for experiment planning, execution, supervision, and evaluation. The introduction of AI techniques is expected to contribute to the understanding of the complex phenomena associated with the foaming of polymers. In this perspective, machine learning methods are expected to support effective experiment setting and data interpretation. From an infrastructural point of view, the development of a robotic platform that permits remote and autonomous experiment execution can lead to interesting implications in terms of safety, innovative teaching, and sharing of information, materials, and equipment with research centers and companies.

Oral (S04-452, Time: Wednesday 10:40, Room: Monosuisse)

Preparation of PP-TiO2/Graphene hybrid Nanocomposites Foams for EMI Shielding Applications

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Polymer composites and their foams for Electromagnetic Interference Shielding (EMI) shielding applications have been a topic of rigorous study for the past few years. In such composites, a polymer matrix helps in the uniform distribution of added nanofillers along with providing a durable, flexible, chemical-resistant layer. Nanofillers added in such polymers are generally selected on basis of their conductivity and dielectric properties. Conductive nanofillers provide efficient dissipation of electromagnetic energy throughout the matrix whereas dielectric nanofillers help absorb the electromagnetic radiations for the formation of charged dipoles. Foam structure also provides the added advantage of Multiple Internal Reflections which enhances the efficient absorption of EM waves by providing increased interaction between the EM waves and shielding material. As a precursor to this work, a microcellular foam composite of polypropylene with 2 wt. % TiO2 nanoparticles with cell size in the range of 7-10 µm and cell density of 17.66 x 106 were prepared. Such foams provided higher compression strength (about 30%) when compared to neat polypropylene. The establishment of a relation between rheological properties and foam processibility of such composites had also been attempted. This work primarily focuses on the preparation of a microcellular foamed structure of PP-TiO2/Graphene hybrid nanocomposites for EMI shielding applications. TiO2 nanoparticles of particle size in the range of 10-20 nm had been efficiently distributed in the polypropylene matrix. TiO2 nanoparticles provide a relatively high dielectric value of around 100, which is sufficiently high with a lower cost. Graphene nanoflakes with 10-15 layers of graphene provide high conductivity along with a larger surface area. This work also focuses on the efficient distribution of titanium dioxide nanoparticles and graphene nanoflakes throughout a polypropylene foam matrix through optimized processing conditions.

Oral (S04-487, Time: Wednesday 11:00, Room: Monosuisse)

Rheology-driven design of pizza gas foaming

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This study proposes a novel dough aeration method based on a new foaming technology of thermosetting polyurethane (PU), focusing on the case of Neapolitan pizza. Both pizza and PU are prepared through two concurrent processes, curing and foaming, and as long as these two processes occur concomitantly, a good solid foam is obtained. The novel process uses a physical blowing agent and the imposition of a rheology-driven designed pressure history during baking as a substitute for the leavening stage. In this way, leavening and baking occur concomitantly without any kind of chemical blowing agent, and this severely reduces both the timing and number of processing steps, thus also eliminating the health problems that might occur after yeast ingestion. The evolution of the dough structure during baking is studied by a rheological characterization at leavening and baking conditions. These experimental pieces of information are used to evaluate the time available for blowing agent sorption under pressure during early baking stage, and to guide the pressure release during the final baking. Three critical process parameters are identified, namely the maximum pressure, the saturation time and the Pressure Drop Rate (PDR). Their effect on the final properties of the product is thoroughly studied as well, focusing on the volume expansion ratio, with the aim of achieving an optimally foamed pizza.

Oral (S04-494, Time: Wednesday 11:20, Room: Monosuisse)

Hybrid Silica-based Aerogels for Efficient Water Treatment Applications

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The ever-increasing urbanization and industrialization activities have led to an expansion of wastewater discharge in the environment, which negatively impacted the ecosystem and human health. Therefore, several methods have been developed for water remediation. However, adsorption/sorption, using porous materials, have been proven to be one of the most efficient tools to tackle this issue. Herein, we report the development of different 3D porous materials, including silica aerogels and aerogel-in-foam composites, with tailored surface properties for the removal of various pollutants from aqueous media. These materials have shown remarkable performances for oil sorption, as well as heavy metals and dye adsorption applications. Moreover, these materials were found to exhibit excellent thermal and mechanical properties with high stability under harsh operating conditions and outstanding recyclability. The properties and the performance of these different adsorbents for the uptake of a wide range of pollutants was tailored via engineered interface and structure, as well as surface modification strategies. For enhanced oil-water separation applications, we have used numerous strategies to induce high hydrophobicity. We have also investigated different functionalization approaches in order to increase the concentration of active binding sites and improve metals/dyes adsorption efficiency. Moreover, our materials were able to remove these pollutants from polluted water selectively and continuously, which proves their potential scalability and usability on a large scale.

Oral (S04-497, Time: Wednesday 11:40, Room: Monosuisse)

Foaming thermoplastic polyurethane with methylal

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This work investigates the use of methylal (dimethoxymethane) as a blowing agent in thermoplastic polyurethane (TPU) foaming. Under the adopted processing conditions, methylal is in the liquid state, unlike blowing agents typically used in foaming; therefore, we investigated two different paths. First, we experimented with the liquid foaming strategy, foaming TPU with methylal in the liquid state alone. Second, we used methylal as a co-blowing agent together with CO2 or N2. In both cases, we investigated the effect of pressure, pressure drop rate, and temperature on the resulting foam density and morphology. Overall, methylal proved to be an effective blowing agent, especially in cooperation with other gaseous agents, where it severely improves the expansion ratio of the final product. The problem of the shrinkage of TPUs in foaming is also addressed, with a focus on understanding which phenomena are predominant in causing volume shrinkage and how to prevent it. The stress-relaxation behavior and diffusivity of the gases are considered, and from these, different strategies are proposed. We worked on both the process variables during foaming and the storage conditions during aging of the foams, and we collected the results in terms of density reduction over time. Once again, methylal proves fundamental, affecting not only the foamability, but also the foams shrinking behavior.

Poster (S04-174, Time: Thursday 17:00, Room: Foyer)

High Electromagnetic Interference Shielding Performance of Thermoplastic Polyurethane Foams Fabricated by scCO2 Limited Foaming

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Constantly pursuing high electromagnetic interference shielding (EMI SE) performance of flexible polymer materials to achieve high valuable products has become an eternal goal of polymer researchers. In this work, high EMI SE properties of thermoplastic polyurethane (TPU) foams were fabricated via supercritical carbon dioxide limited foaming. The results show that TPU foams via limited foaming possess higher expansion ratio compared to free foaming samples, more than that, higher EMI SE properties of TPU foams were achieved by limited foaming. TPU limited foams with 1 wt% MWCNT loading content has the most excellent electromagnetic interference specific shielding (EMI SSE) performance with average value more than 30 dB·cm3/g. This work guides a facile and green method to fabricate high electromagnetic interference shielding performance of TPU or other flexible polymer materials, which might cater to the deep interest of polymer researcher in this field and give some inspirations to manufacture high valuable products.

Poster (S04-641, Time: Thursday 17:00, Room: Foyer)

Temperature and Strain Rate Effects on the Compressive Properties of 3D-Printed Polylactic Acid Microcellular Foams

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The purpose of this study is to investigate the effects of testing environment temperature and loading strain rate on the mechanical performance of 3D-printed polylactic acid (PLA) microcellular foams. Expandable PLA filaments were first prepared at 0, 1.0, 2.5 and 5.0 wt.% loadings of thermally expandable microspheres (TEM) using single screw extrusion process. Test specimens of PLA foams at various TEM loadings were then 3D-printed via fused filament fabrication process. Densities of the printed foams were found to decrease with an increase in the TEM loading in the range of about 1.2 to 0.316 g/cm3. Quasi-static and Split Hopkinson Pressure Bar (SHPB) dynamic compression testing was conducted at testing temperatures of -15 ,5, 25 and 40°C. For all the temperatures tested, the 3D-printed foams exhibited typical three-phase behavior, i.e., elastic, plateau, and densification regions. Overall higher compressive yield strength values were observed under dynamic loading conditions for various densities and temperatures. With an increase in the temperature, both the elastic modulus and stress (at 50% strain) of unfoamed and foamed samples were found to decrease. The stress however seemed to decay at a faster rate at higher temperature ranges.

Poster (S04-685, Time: Thursday 17:00, Room: Foyer)

Foaming behavior of polymer blends with a well-aligned ribbon structure and anisotropic mechanical properties

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Microcellular foams have unique properties and have found innovative applications such as packaging, insulation, sports equipment and aviation. Super-critical carbon dioxide (CO2) is an appealing foaming agent as it is much less environmentally harmful compared to classical foaming agents. This work aims to use CO2 as a foaming agent to obtain a polystyrene (PS)/polyamide 6 (PA6) blend with well-aligned ribbons of PA6 through tape extrusion. The foaming temperature is lower than the melting temperature of PA6 and is higher than the glass transition of PS. Thus, the PA6 phase remains in the solid state during the foaming process, and thus acts as a heterogeneous foaming nucleation agent. After saturation with CO2 under a certain pressure followed by pressure release to the atmospheric one, a multilayered PS/PA6 foam with alternate porous PS and solid PA6 is obtained. The cell size of the foam is smaller than that of the pure PS foam because the solid PA6 ribbon not only acts as a heterogeneous foaming nucleation but also restricts cell growth. More interestingly, those cells are oriented perpendicularly to the aligned PA6 ribbons. Therefore, the foam exhibits anisotropic mechanical properties. For example, the compressive strength of a foam with cells oriented along the direction perpendicular to the ribbon direction is much higher than that along the direction parallel to the ribbons direction.

Keynote (S05-045, Time: Tuesday 10:15, Room: EMS-Chemie)

A Virtual Process Chain for Glass Mat Thermoplastics

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Chopped fiber materials provide potential to be used for complex geometries for functional lightweighting. The material targeted within this study is Tepex Flowcore, a long glass fiber reinforced polyamide (PA/GF) glass mat manufactured by Lanxess. The glass fibers are nominally 50 mm long, with a random orientation in-plane. Compression molding of Flowcore is divided into two different stages within the mold, thermoforming and squeeze flow. Simulation can provide insights into issues such as local wrinkling and incomplete mold filling. In this study, experimental molding analyses are presented and the outcomes of a virtual process chain for GMT materials is described. A sequential simulation approach is explained including the sub-steps of thermoforming and squeeze flow. Constitutive modeling remains continuous through a unified material modeling approach and important state variables are predicted, such as fiber orientation or temperature. The virtual process chain is applied to different geometries with increasing complexity and the benefits and challenges for digital product and manufacturing process development are described. The development and measurement of the material input parameters (using rheometry) and the validation measurements (including computer tomography) for the simulations is described in detail.

Keynote (S05-049, Time: Tuesday 13:45, Room: EMS-Chemie)

Flexible and Durable Conductive Fiber Polymer Nanocomposites for Stretchable EMI Shielding Materials

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The emergence of wearable and portable electronic devices increases the demand for flexible and stretchable electromagnetic interference (EMI) shielding materials that would prevent the malfunction of electronic systems that measure key human health indicators. In this work, highly stretchable and conductive steel fibers (SSF) /polypropylene (PP) composites are fabricated via a combination of extrusion and injection molding processes. Benefiting from the alignment of SSFs and specific crystalline phases created inside PP, the resultant composite brings excellent stretchability of 600%, tunable EMI shielding effectiveness (SE) as high as 25 dB, along with high electrical conductivity of 8×10^-1 S.cm^-1, and impact strength of 5 KJ.m^-2. The structure, thermal properties and electrical properties of the composites were characterized by scanning electron microscopy (SEM), differential scanning calorimetry (DSC), X-ray diffraction (XRD), and a four-probe volume resistivity tester.

Keynote (S05-391, Time: Wednesday 10:15, Room: EMS-Chemie)

Hierarchical Graphene Nanoplatelet and Glass Fiber Hybrid Composites with Synergy-Induced Gradient Interphase

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Considering the global increase in environmental awareness, manufacturing industries have predominantly adopted the use of lightweight materials, to decrease the energy consumption and the amount of material used in the fabricated products. In this work, hierarchically structured hybrid polypropylene (PP)-based composites reinforced with graphene nanoplatelets (GnP) and glass fiber (GF), were fabricated by injection molding to elucidate the synergistic effect and how it can be tailored to produce mechanical properties not possible in biphasic composites. In this hierarchical system, the GnPs are chemically and electrostatically attached to the GFs producing a gradient interphase that favours load transfer at the interface. This is attributed to the local nanoscale stiffness variations in the interphase regions between the GFs and the matrix, due to the increased degree of trans-crystallization encapsulating hierarchical structure, which thereby affects the overall bulk properties of these composites. Additionally, mean-field homogenization theory was applied to predict the effective modulus of the fabricated composites. The theory showed reliable predictions for composites with low reinforcements contents. However, for higher reinforcement contents, inter-reinforcement interaction and agglomeration were attributed to the discrepancy between the experimental results and theoretical predictions. As a result, the homogenization theory was modified to incorporate a core-shell structure that better resembles the hierarchical reinforcement system, leading to reliable predications at all contents. Ultimately, this work presents an experimental and theoretical approach to understand the synergistic effect in hierarchically structured hybrid composites, to tailor their mechanical properties for variety of advanced high-performance applications, where mechanical performance and lightweight are simultaneously imperative to meet the energy efficiency requirements of the future

Keynote (S05-394, Time: Wednesday 13:45, Room: EMS-Chemie)

Development of self-healing soft sensor using thermoplastic elastomer materials

Clemens Frank[1], Georgopoulou Antonia[1], Kein Jana[1], Susoff Markus[2]

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Self-healing polymers have gained attention in the field of soft robotic devices to improve the shelf-life. However, integrating a self-healing sensor can be challenging. In this work, we developed a moisture sensor using polyesterol and polyetherol based thermoplastic polyurethanes (TPU) and carbon as a conductive filler component. To improve the self-healing properties of TPU, blends based on TPU and ethylene vinyl acetate (EVA) were investigated. The healing temperature and self-healing properties of different blend vinyl acetate contents were investigated. Besides, the influence of the flow properties of EVA on self-healing was investigated. The best self-healing of TPU EVA blends was investigated at 110 °C for a blend vinyl acetate content of 13.1 wt%. By using an EVA with a higher melt index, TPU-EVA polymer blends showed better self-healing. The investigation of the self-healing properties was based on mechanical testing. In TPU-EVA-CB composites, CB decreased the mechanical properties after self-healing significantly. The electrical properties recovered after self-healing completely.

Keynote (S05-575, Time: Thursday 10:15, Room: EMS-Chemie)

Non-covalent modification of graphene nanoplatelets to produce electrically and thermally conductive polyamide composites

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Previous studies on polymer composites containing graphene nanoplatelets (GNP) have shown that achieving a good dispersion in these composites remains a challenge. This is due to the aggregating nature of GNPs arising from van der Waals interactions, specifically π-π interactions. In this work, we implement a simple non-covalent surface modification technique to improve the dispersion and interaction of GNP with a polyamide matrix. We use trimellitic anhydride (TMA) as the functionalising agent, due to its ability to form π-π interactions with GNP, while the anhydride group can form covalent bonds with terminal amine groups of the polyamide chains during the melt compounding process. Non-covalent modification of the GNP is achieved by mixing desired amount of TMA with GNP in a counter rotating batch mixer at a temperature of 250 ^{0}C, during a thermomechanical exfoliation process. Adsorption studies revealed 2-4 wt.% of TMA as the optimal amounts for functionalization. Functionalization with TMA resulted in a decrease in specific surface area of the GNP indicating adsorption on GNP sites, along with increase in surface energy and oxygen content. Evaluation of composite morphology showed good dispersion and distribution of the GNPs. The mechanical properties, including the flexural modulus showed significant improvement with GNP content, while the impact strength of the composites improved in composites containing between 10-20 wt.% GNPs. We also achieved a maximum thermal conductivity of 3.3 W/m.K for 40 wt.% composites, and an electrical conductivity of 0.2 S/m, with an electrical percolation threshold at 15 vol.%. Thus, we demonstrate an effective and straightforward modification of GNP for applications in polymer composites.

Keynote (S05-582, Time: Thursday 13:45, Room: EMS-Chemie)

Processing-Structure-Property Relationships for Composites of Block Copolymers and Graphene Nanoplatelets

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The properties of graphene nanoplatelet (GNP) filled block copolymers are determined by the preferential localisation of the nanoplatelets in the copolymer hard and soft segments and consequently the effect of platelet inclusion on microphase separation phenomena. Composites of block copolymers including, thermoplastic polyurethane (TPU) and poly(ether-b-amide) (Pebax) going from high hard segment (low soft segment) to low hard segment (high soft segment) and graphene nanoplatelets of different lateral dimensions were prepared by melt mixing in a twin screw extruder. Evidence from a combination of differential scanning calorimetry (DSC), dynamic mechanical thermal analysis (DMTA) and small angle X-ray scattering (SAXS) showed that the nanoplatelets, irrespective of platelet size enhanced microphase separation. Where the hard segment is crystallizable, the GNP has a significant nucleating effect. In this presentation, the effect of GNP inclusion and platelet size on the high shear rate rheology of different block copolymers (TPU and Pebax) and the role the localisation of the filler plays in phase separation and subsequently in governing mechanical properties is described.

Keynote (S05-639, Time: Friday 10:15, Room: EMS-Chemie)

Advanced Biocarbons from Sustainable Resources and their Lightweight Polymer Composite Materials: Current Status and Future Opportunities

Misra Manjusri[1]

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Lightweight green composites from renewable resources are attracting increased attention from advanced manufacturing sectors. A strong momentum for weight reduction in auto parts without compromising strength and durability is motivated by a need for a better fuel economy, particularly in electric vehicles. To increase renewable content in materials, we use thermo-chemical conversion (pyrolysis) of biomass/agri-food/forestry resources and waste feedstock to overcome the key challenges associated with the use of traditional natural fibres in melt processed biocomposites. Biocarbons have high thermal stability, which broadens their use as a reinforcing filler in a range of plastics - from traditional thermoplastics like polypropylene, polylactic acid (PLA), to engineering thermoplastics such as polyamide, polyphthalamide (PPA) and polyphenylene sulfide (PPS). In addition, biocarbons have been also used effectively in composites with waste plastics. Overall, biocarbon composites can be very effective as one of the enabling strategies towards sustainable materials in advance manufacturing and a circular bioeconomy.

Keynote (S05-708, Time: Wednesday 17:00, Room: EMS-Chemie)

Processability of Ultrahigh Molecular Weight Polyolefin and Their Foams for High Performance Applications

Ghosh Anup K.[1]

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Ultrahigh molecular weight polyolefin (UHMWPO) with outstanding performance properties stand out as suitable materials for a wide range of applications, such as biomedical, vibrational reduction, and electromagnetic and sound wave shielding. The extreme molecular weight of UHMWPO in the range of (1.1–8.2) millions, develops entanglements inherently, which leads to limitations in processability by conventional polymer processing techniques due to high melt viscosity. UHMWPOs can be used by plasticizing with low-molecular-weight polyolefin wax or blending with other olefins with relatively low melt viscosity without compromising their inherent properties. Further, blends or plasticized UHMWPO are foamed using supercritical CO2 to be used in ballistics as well as for sound absorption due to its close cells, high cell density, high strength-to-weight ratio, excellent energy dissipation in terms of impact toughness, and shielding to wave propagation. Nanocomposite foams of UHMWPO can also be used in the field of electrical or electronics, where the electrical conductivity, dielectric constant, and magnetic permeability play a critical role, by incorporating nanomaterials such as CNTs, rGO, carbon fiber, non-metallic particles, and hybrids of these nanofillers in blends or pristine form. UHMWPO-based nanocomposite foams containing carbonic fillers have greatly expedited scientific research efforts in electromagnetic interference (EMI) shielding as well as piezoresistive sensing devices. Moreover, a wide array of advanced functional applications for foams, such as lightweight applications, heat and sound insulation, tissue engineering, oil spill clean-up, shape memory, and flexible materials, are within the scope of the UHMWPO's nanocomposites and their foams.

Oral (S05-008, Time: Wednesday 16:00, Room: EMS-Chemie)

Cellulose nanofiber reinforced TPE for biomedical applications

Maji Purbasha[1], Naskar Kinsuk[1]

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Styrene-b-poly(ethylene-co-butylene)-b-polystyrene (SEBS) based thermoplastic elastomers are gradually gaining attention in biomedical applications like artificial heart valves, pacemaker connectors, suture materials, implants, vascular grafts, wearable electronics, and flexible sensors etc. They are prospective options for addressing the difficulties of poorer mechanical performances, non-recyclability of silicon-based implants, and poor hydrolytic and oxidative stability of thermoplastic polyurethane-based implants. However, because of their physical crosslinking, the materials tend to lose their mechanical performances and show a change of shape or creep on long-term use. In this study, we sought to develop novel SEBS-based biocomposites by incorporating a non-toxic, high aspect ratio, plant-derived cellulose nanofiber (CNF) as a reinforcing agent with maleic anhydride-grafted SEBS (mSEBS) as a compatibilizer. Esterification reaction between CNF and mSEBS leads to the grafting of high molecular weight mSEBS on CNF surface. The presence of hydrophobic polymer on CNF fibrils significantly reduces aggregation of them while improving their hydrophobicity. The final composite was prepared by melt-mixing of SEBS with CNF-g-mSEBS. This step leads to many discrete CNF-g-mSEBS domains into the SEBS matrix. The presence of mSEBS at CNF/SEBS interface results in a gradual change of modulus across the interface and improved mechanical performances. At 0.2% CNF loading, a maximum gain of 73% in tensile strength and 24% in extensibility was attained, in addition to a 30% improvement in modulus @300%. The composites are expected to show excellent strength, toughness, improved creep, and flex fatigue resistance while maintaining their biocompatibility. The prepared composite could be a desirable material for polymeric heart valves, stents, and wearable devices.

Oral (S05-022, Time: Tuesday 10:40, Room: EMS-Chemie)

Effect of fibre orientation and volume fraction on mechanical properties of all-polyamide composites

Cordin Michael[1]

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Industry is more and more interested in all-polymer composites, because they can be easily recycled due to their chemical similarity of reinforcing fibres and matrix material. Besides these composites are high-performance materials due to their very good adhesion between matrix material and reinforcing fibres. Recycling is possible by simple melting processes if thermoplastic polymers are used for the fabrication of this type of composite material. This prevents environmental pollution. The talk describes the preparation of all-polyamide composites by technical embroidering of polyamide-6-polyamide-66 hybrid yarn in the desired orientation on a polyamide-6-fleece, followed by thermoforming at 230°C to produce the final composite material. The mechanical properties of these composites were then characterized by quasi-static and high-speed tensile testing and penetration tests as a function of fibre orientation and the ratio of used polyamide-6 and polyamide-66. The relationship between E-modulus and fibre orientation was modelled by the modified rule-of-mixture theory as well as the theory of elasticity for orthotropic materials. The results show clearly, that all-polymer composites are an attractive option to replace metals and other classical construction materials, including conventional composite materials.

Oral (S05-056, Time: Tuesday 11:00, Room: EMS-Chemie)

Macro-mechanical analysis of nettle yarn (Girardinia diversifolia) reinforced PLA biocomposites under tensile stress

Nandi Parna[1], Das Dipayan[1]

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Primary ways in which conventional polymeric composites largely contribute to environmental degradation are, through the emission of toxic air pollutants and the generation of hazardous solid waste. As a result, it is suggested that an alternative to conventional polymeric composites be required, preferably one with less of a negative effect on the nature. "Natural Fibre Reinforced Bio-matrix Composites," also known as Biocomposites, is an exciting new type of engineered material with the potential to improve sustainability in a number of ways. In this study, nettle/PLA unidirectional biocomposites were developed by varying the mass proportions of nettle yarn prepreg and PLA biomatrix. In a unidirectional biocomposite, the reinforcements at the weakest cross-section cause the material to fail. When the longitudinal tensile stress is amplified even further, a cumulative number of prepreg yarns become broken, leading to catastrophic failure of the composite. Rupture of the composite occurs at a breaking strain value of the composite, which is interpreted to be very close to half of the prepreg assembly's maximum strength. Accordingly, the ratio of breaking stresses of the composite and maximum strength of prepreg assembly describes the coefficient of utilization of unidirectional prepreg strength (ϕ), where lowest values (composite with 50 wt% reinforcement) indicate greatest effectiveness as reinforcement. A spectacular example of the two distinct modes of failure of a unidirectional composite subjected to longitudinal tensile loading is observed. The brittle failure with reinforcement breaking is most obvious in low reinforcement volume fraction ($V<0.49$) composites. Composites having high reinforcement volume fraction ($V>0.49$) show fracture due to reinforcement slippage. In this scenario, microcracks present at various locations throughout the composite may fuse together due to debonding of reinforcement along the loading direction or sheared failure of matrix.

Oral (S05-098, Time: Tuesday 11:20, Room: EMS-Chemie)

Development of hybrid composites with the use of novel sustainable reinforcing system

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This work focuses on the development of hybrid composites reinforced with the use of a novel filler system to reduce the environmental burden of the composite. The sustainable filler system consisting of biocarbon (BC) particles, short basalt (BF) and carbon (CF) fibers was used as the reinforcement for POM, PC/ABS and PBT/ABS blends. The use of BC particles was aimed at eliminating mineral fillers (e.g. chalk, talc) by fully biobased material, while basalt and carbon fibers could be considered as an alternative to glass fibers (GF). All materials were prepared with the same 20 % filler content differentiating the BC/BF/CF ratio. The prepared materials were manufactured using injection molding and different types of standard test samples were prepared. For some samples, the thermo-oxidative heat aging procedure was applied. Samples were subjected to characterization like mechanical (static Tensile, Charpy impact tests), thermomechanical (DMTA, HDT), thermal and rheological (DSC, rotational rheometery) and structure properties examination. In order to assess fiber distribution within the material structure, the samples were scanned by microtomography method (μCT). The obtained results indicate the positive effects of using BC/BF/CF as filler. The filler system could be successfully be used as a substitute for mineral fillers. In addition, low density of the chosen fillers could lead to a reduction of the part weight. The analysis of the mechanical test results showed a significant impact of BF content on the stiffness. Thermomechanical properties of composites have been visibly improved compared to the unmodified samples. One key aspect of using hybrid reinforcement is the reduction of structure anisotropy, which is usually associated with the presence of fibrous fillers. Studies have shown the positive effect of the addition of BC particles, the unidirectional arrangement of BF/CF was disturbed, which resulted in greater randomization of the composite structure.

Oral (S05-103, Time: Tuesday 11:40, Room: EMS-Chemie)

Improvement of thermal conductivity by adding boron nitrid and expanded graphite to the HDPE

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In various industrial applications standard heat exchangers made of metal are replaced with alternatives such as polymer composites. Resistance to corrosion, chemical stability, hydrophobicity of material and good rheological properties makes high-density polyethylene (HDPE) possible substitute for convectional materials. The lack of HDPE thermal properties (but typical for metals - high thermal conductivity and heat capacity) could be improved by the addition of adequate fillers. In this paper, composites with two kinds of filler, expanded graphite (EG) and boron nitride (BN) with micro-sized particles, were used in high-density polyethylene (HDPE) matrix. Mass ratio of both fillers up to 25 wt.% in HDPE matrices were prepared together with compatibilizer, for which polyethylene-grafted-maleic anhydride (PE-g-MAH) was used. Preparation of samples included melt mixing in a compounder and hot-pressing in the moulds. Compounds were subject for testing on thermal conductivity (TC). With the addition of fillers TC is expected to increase compared to the pure HDPE. Micro-composite with BN as a filler is supposed to have better results in testing for the same mass ratio as in composite with EG.

Oral (S05-111, Time: Tuesday 12:00, Room: EMS-Chemie)

Manufacturing of hybrid thermoplastic fiber reinforced profiles with thermoplastic coating layer – Process development and mechanical recycling

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Pultruded profiles are broadly being used as semi-finished products in the building industry. To access new applications such as large-scale window profiles or local reinforcement of wind turbine blades, the pultruded profiles require not only adequate mechanical properties but also excellent optical as well as haptic properties or must be protected from weather conditions. In the present contribution, a process combination consisting of pultrusion and extrusion process (PulEx) is investigated to functionalize pultruded profiles by inline application of a thermoplastic coating layer. An in-situ polymerizing thermoplastic PMMA resin is used in the pultrusion process and in-line coated with a similar extrusion type material to increase the potential for subsequent mechanical and chemical recycling approaches. After systematic investigations to increase the haul-off speed and identifying a suitable process window for the PulEx process the hybrid profiles quality is accessed by evaluation of surface quality and microscopic examination of the cross-section. Subsequent to the mechanical disintegration of the pultrusion profiles, the incorporation of comminuted pultrusion profiles into virgin polymer in a twin screw extruder is studied. The resulting mechanical properties of the short fiber reinforced compound is evaluated considering weight fraction of the recycled material, the underlying reinforcement fiber length and distribution of the fibers in the compound. The new process hybridization opens the potential for economical one-step production of thermoplastic profiles with improved mechanical properties through integration of continuous fiber reinforcement and improved mechanical recycling potential.

Oral (S05-119, Time: Tuesday 14:50, Room: EMS-Chemie)

Induction heating direct joining of carbon fiber reinforced thermoplastic and galvanized steel with hot water treatment

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Hybrid joining of galvanized steel (GS) and carbon fiber reinforced thermoplastic (CFRTP) can contribute to the lightweight design of vehicles while maintaining good mechanical properties and anti-corrosion ability. Induction heating direct joining (IHDJ) is a promising hybrid joining method, which mainly contains two steps: (1) treating metal surface to form micro-nano structures, (2) heating metal part via induction heating to make polymer part melt and flow into the metal surface structures to form joint. IHDJ has many advantages such as high efficiency, low cost and little damage to joining parts because of non-contact heating. In this study, hot water treatment (HWT) was proposed as a suitable surface treatment method for GS in IHDJ. HWT can produce complex needle-like nanostructures on GS surface by simply dipping a metal specimen into hot water for a few minutes, which was expected to contribute to forming strong anchoring effect at the joining interface. The objective of this study is to confirm the feasibility of IHDJ of CFRTP and GS with HWT. The joining strength was examined by tensile-shear tests and the joining interface temperature was measured by an ultra-thin thermocouple during the induction heating process. The result shows that a high joining strength of 38.0 MPa was successfully obtained with the proposed strategy. The joining mechanism was investigated by studying the relationship between the joining interface temperature and joining strength. Too low or too high interface temperature both caused weak joining strength. The optimal joining interface temperature was around 300 ℃. The cross section of joining interface and fracture surfaces were observed to further analyze the fracture mechanism. Low interface temperature will lead to low polymer fluidity and poor infiltration to the metal surface structure, while high interface temperature will lead to degradation of zinc coating because of its low melting point of 419.5 ℃.

Oral (S05-201, Time: Tuesday 14:30, Room: EMS-Chemie)

Evaluating The Optimum Settings for The Inline Defect Detection of Glass-Fiber-Reinforced Unidirectional Thermoplastic Tapes by Optical Coherence Tomography

Wenninger Michael[1], Marschik Christian[1], Schnaitter Mathias[2], Hochleitner Gernot[3], Berger-Weber Gerald R.[2], Steinbichler Georg[2]

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While for several decades composites were mainly manufactured from thermosets such as epoxy or polyester resins, a strong trend has emerged towards the use of thermoplastic matrix systems. Due to their reversible melting behavior, they offer several advantages including a better recyclability, higher degree of automation, and much faster productions speeds. To raise the productivity of thermoplastic composite manufacturing processes, appropriate and reliable inline quality assurance systems are needed. Optical coherence tomography (OCT), originally developed for biomedical applications, has been proven to be a powerful tool for the non-destructive testing and characterization of unidirectional (UD) fiber-reinforced thermoplastic tapes made from glass fibers. Prior offline OCT measurements on a test rig demonstrated the reliability and robustness of the method in detecting visually apparent tape defects such as gaps or fiber breakages and also insufficiently impregnated fiber regions as one of the most critical bulk defects. In this work, inline OCT measurements were performed on stationary PC/GF UD tapes in an industrial-scale production line. Systematic experimental analyses were carried out, in which the OCT sensor was moved transverse to the tape width and the influence of (i) A-scan sampling rate, (ii) A-scan averaging, and (iii) sensor travel speed on measurement accuracy, and data size was evaluated. Measurement accuracy was validated using optical microscopy. Optimum sensor settings were derived to improve the inline capability of the measurement approach under real production conditions. Inline experiments with a moving tape will be conducted with these settings.

Oral (S05-223, Time: Tuesday 16:00, Room: EMS-Chemie)

Synthesis and characterization of poly(lactic acid) biobased composites with Lignin/Nanolignin additives

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Lignocellulosic materials are utilized as biobased fillers to enhance the physicochemical and mechanical properties of PLA-based materials. In this work, biobased PLA/lignin composite films were prepared by solvent casting to synthesize a masterbatch followed by melt mixing with neat PLA. Pale brown coloured films were then produced by compression moulding. Two series of films were prepared using either micro-scale raw lignin or nanolignin at four different lignin contents of 0.5, 1.0, 2.5, and 5.0 wt%. The films were characterised by Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD) and Differential Scanning Calorimetry (DSC). Additionally, the mechanical and antioxidant properties were also investigated, and their antioxidant activity was assessed with the 2,2-Diphenyil-1-picrylhydrazyl (DPPH) method. All the PLA composite films showed a uniform dispersion of lignin/nanolignin. Hydrogen bonding was detected between PLA and fillers, while crystallization of PLA was suppressed by both lignin and nanolignin. All composite films showed enhanced tensile strength and Young's modulus, especially when nanolignin was used as a filler, due to its finer dispersion in the PLA matrix, as shown by SEM and TEM micrographs. Antioxidant properties of the composite films were also improved compared with neat PLA, especially at higher filler contents. Acknowledgement This research was funded by the European Community's Horizon 2020 Framework Program H2020 (grant number 952941 Project: BIOMAC, European Sustainable BIObased nanoMAterials Community; https://www.biomac-oitb.eu).

Oral (S05-260, Time: Tuesday 16:20, Room: EMS-Chemie)

3D-Consolidation Process for Direct Manufacture of Advanced Continuous Fiber-Reinforced Thermoplastic Composite Components

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Thermoplastic composites are attracting increasing momentum in the aerospace industry due to their potential to face current needs in terms of cycle time reduction, automated manufacturing, and recycling. In state-of-the-art press forming processing, a consolidated composite preform, made of stacked continuous fiber-reinforced thermoplastic tapes, is heated above melt temperature of the thermoplastic matrix, transported to a press or injection molding unit and formed into a component geometry. In the time from transport of the hot preform to the finished mold closing process, an inhomogeneous and significant temperature loss occurs in the processed material. Due to the characteristics of thermoplastic melts, a drop in temperature leads to an increase in matrix viscosity and causes higher resistance in the materials deformation modes, which restrain the formability. One can argue that forming takes place in a relatively uncontrolled manner. Especially for advanced geometries or tailored laminate architectures, this can evoke the risk of defect formation, geometric inaccuracies, or insufficient final consolidation. With the objective of realizing advanced net shape tailored thermoplastic composite components at a high-quality level, the approach of 3D-consolidation is presented in this study. The approach is based on merging the forming and consolidation of a spot-welded ply stack within one process step by applying a three-stage procedure consisting of a pressureless heating stage, an isothermal forming/consolidation stage and a pressurized cooling stage. This study presents the technical implementation of the 3D-consolidation approach by means of a specially developed tool concept on a force-controlled consolidation unit. The concept is evaluated and results presented on the basis of an aerodynamic component consisting of an automated manufacturable tailored laminate architecture made of CF/LM-PAEK unidirectional tapes.

Oral (S05-270, Time: Tuesday 16:40, Room: EMS-Chemie)

Multi-layer Co-extrusion of Highly-Filled Polymer Composites via the Layer Multiplication Technique

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Recent work in the area of multi-layer co-extrusion has led to the creation of multi-layer structures of highly-filled polymer composites. Solid glass microspheres of two different sizes (average particle diameters: 7.7 μm and 39 μm) were examined at various loading levels up to 40 volume % (64 weight %) in an low density polyethylene matrix for sheet extrusion in multi-layer co-extrusion via the layer multiplication technique. The work examines the capabilities and limitations of the process to achieve high numbers of thin layers when utilizing high filler loading levels. The resultant properties of the structures that can be created and maintained of multi-layer films are examined.

Oral (S05-272, Time: Wednesday 10:40, Room: EMS-Chemie)

Ultrasound-assisted extrusion of graphitics materials reinforced HDPE

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For decades, creating new materials by mixing nanoparticles with polymeric matrices has attracted the attention of both industrialists and academics. Indeed, adding particles with good electrical or mechanical properties, such as graphitics materials, to polymer matrixes, allows to obtain materials with enhanced properties for varied applications including automotive, energy storage, aerospace, and biomedical among others. However, to harness the properties of the nanofillers, a good distribution and dispersion of the nanofiller within the polymer must be obtained. Extrusion processes coupled with ultrasound sources has been shown in the literature as an effective method to obtain an efficient dispersion of fillers such as clay and carbon nanotubes. However, the propagation mechanism of the ultrasonic waves within molten polymers and nanocomposites is far from being understood. This work investigated the effect of ultrasonic vibration during melt extrusion on the dispersion of different graphitic materials in a high-density polyethylene (HDPE) matrix. Several HDPE nanocomposites graphitic materials of 2 different size distributions (D90* = 33 or 128 µm) were processed using a twin-screw extruder, with and without being subjected to ultrasonic waves. The ultrasonic waves were applied in the mixing zone of the barrel, using an ultrasonic probe with a frequency of 20 kHz and a maximum amplitude of 45 µm. The dispersion of GnP was assessed using small-angle oscillatory shear tests and electrical conductivity tests. The morphology of the composites was also observed using scanning electron microscopy SEM and polarized optical microscope POM. It was shown that the dispersion state of graphitics materials in HDPE was dependent on the amplitude of the ultrasound applied during melt extrusion.
(*D90 means 90 % of particles have a size below)

Oral (S05-283, Time: Wednesday 11:00, Room: EMS-Chemie)

Automated Quality Assessment of UD Tapes

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Unidirectional (UD) fiber-reinforced composite materials have played a central role in lightweight construction for several decades, as they combine excellent mechanical properties with low weight. Currently, we are observing a trend toward thermoplastic matrix systems whose reversible melting behavior confers several advantages over their thermoset counterparts. The mechanical properties of the tapes depend on the fiber content and uniform distribution as well as their efficient impregnation with polymer melt. Inhomogeneities such as dry fiber regions, fiber breakages, and air or polymer-filled gaps (pores) lead to defects in the tapes and final composite parts. Avoiding such defects already during the production process would greatly improve yields and product quality. This requires first a quantifiable understanding of the quality features of the tapes (Critical Quality Attributes, CQA), and subsequently the identification of a non-destructive technology that can identify defects in real-time. This enables the association of quality attributes with process states, the determination of Critical Process Parameters, and ultimately an efficient process control. To this end, we have developed objective criteria that describe UD tape quality and an automated method for systematic offline investigation of UD tape cross sections. In particular, we applied computer vision and statistical methods to microscopy images of tape cross-sections to automatically determine several quality parameters for fiber distribution and porosity. The analysis of several tape specimens delivered the ground truth for the investigation of non-destructive, real-time capable monitoring techniques. The offline methodology was implemented in a GUI and represents the basis for the data-informed optimization of UD tape production.

Oral (S05-323, Time: Wednesday 11:20, Room: EMS-Chemie)

Changes in physical-chemical properties in thermosetting-composite-based printed circuit board materials induced by friction-based joining processes

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The present work deals with the changes in physical-chemical properties induced by friction-based joining techniques on thermosetting composite materials. Initial feasibility studies on the production of joints with FR4 printed circuit board substrates via Friction Riveting presented challenges due to the thermal properties of these laminates, especially above 300 °C, where thermal degradation and decomposition occurred. To investigate the behavior of friction riveted FR4 laminates, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) measurements were applied in the range of 25 °C and 500 °C for FR4 with single (FR4-I Cu) and double (FR4-II Cu) copper layers. The DSC results showed exothermic transitions while TGA curves displayed a rapid reduction in sample weight. Both phenomena were observed around 300 °C, where epoxy decomposition reaction occurs followed by the formation of carbonized products (char). The present study clarifies the degradation aspects of joining FR4 and potentially printed circuit boards as a result of high process temperatures in friction-based joining, thus establishing a basis for the manufacturing of friction-riveted joints with functional PCB material.

Oral (S05-332, Time: Wednesday 11:40, Room: EMS-Chemie)

Synthesis and characterization of biodegradable poly(ethylene succinate)/hemp fiber composites as alternative WPCs

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The switch to a circular economy, recycling, or reusing could be an essential instrument to tackle the current triple planetary crises on climate, biodiversity, and pollution. With this work, our goal is to promote alternative and more sustainable materials for the construction of Wood Plastic Composites (WPCs). The ever-increasing plastic wastes mainly from polypropylene and high-density polyethylene led us to use poly(ethylene succinate), PESu, a biodegradable polymer with good mechanical properties. On the other hand, deforestation due to the increased consumption of wood led the scientific community to use substitute products to replace natural wood. A great idea turned out to be the agricultural crop residues, as the bark of the hemp plant. Hemp is lightweight, has hydrophobic properties, long-term mechanical stability, and presents a low carbon footprint. So, in this work, PESu was synthesized by a two-step melt polycondensation reaction. The composite materials containing hemp fibers in different concentrations were prepared by melt mixing in a twin screw extruder. The structural properties of the materials were studied by Fourier Transform Infrared spectroscopy (FTIR), and X-ray diffraction (XRD), while the dispersion and interfacial adhesion of the fibers was examined by Scanning Electron Microscopy (SEM). In addition, the thermal stability and degradation of the composites were investigated using thermogravimetric analysis (TGA). Moreover, the degradation of the samples was also tested in a soil environment. Finally, the mechanical properties of the prepared materials were investigated through tensile and impact strength tests. Acknowledgments This research has been co-financed by the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH-CREATE-INNOVATE (project code: T2EDK – 00008).

Oral (S05-354, Time: Wednesday 14:10, Room: EMS-Chemie)

Back-injection of unidirectional tapes: an efficient way to lightweight composite parts

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The trend towards lightweight construction continues throughout all sectors. Due to their low density, plastics play an important role and are increasingly replacing metals. Possibilities for implementing a lightweight construction strategy range from the use of continuous fibre-reinforced thermosets to short fibre-reinforced thermoplastic parts. By combining thermoplastic foam injection moulding and back injection of continuous fibre-reinforced tapes, such lightweight structural components can be economically realised in high quantities. Potential applications of the backfoaming of continuous fibre-reinforced tapes are sport goods. For example a high-end surf fin produced using the injection moulding process has already been realised. In surfing, the properties of the fin are of immense importance, as its behaviour in the water influences the overall handling of the surfboard. The most important characteristic value is the so-called flex (bending stiffness) of the surf fin. The total weight of the surfboard and therefore of the individual parts is also very important. To meet these two requirements for a surf fin, conventional surf fins are produced using infusion or RTM processes with thermoset resin systems, which is costly and time-consuming. By integrating unidirectional thermoplastic carbon tapes into the injection moulding process, similar properties can be achieved with a much more efficient manufacturing process. The carbon tapes are inserted into the injection mould and back-injected. The tapes lying on the outer sides form a compression and tension band (sandwich component) under a bending load. By aligning the fibres and the cut-out geometry, the properties can also be fine-tuned; thanks to the lower density of the plastic core, the weight of the fin remains low.

Oral (S05-359, Time: Wednesday 14:30, Room: EMS-Chemie)

Surface, mechanical, and thermal properties of multifunctional polymer nanocomposite PVDF-HFP/PVP/MoO3 with antimicrobial activity

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High-tech materials such as polymer nanocomposites (PN) offer the possibility of application in many fields. Materials of particular interest combine multiple functional properties so that they can be used for a variety of purposes, including nanofibers, medical devices and implants, filaments for 3D printing, surface coatings, antibacterial coatings, etc. In the present work, the polymer composite, i.e., PVDF-HFP/PVP and the polymer nanocomposite PVDF-HFP/PVP/MoO3, were prepared as thin films by a solvent casting method. The prepared polymer composites exhibited nanostructured surface with less conductive islands of PVP in the PVDF-HFP polymer matrix, which altered the mechanical and thermal properties of the composite [1]. On the other hand, the addition of PVP to PVDF-HFP reduced the elastic modulus and hardness compared to PVDF-HFP thin film. However, the PVP polymer decreased the crystallinity of PVDF-HFP and affected the crystal structure as the presence of polar beta- and gama-phases, respectively, was detected. The properties improved even more by adding MoO3 nanowires into the polymer composite. The formed polymer nanocomposite PVDF-HFP/PVP/MoO3 (PN) exhibited inhomogeneous nanostructured surface with hydrophilic properties [2]. The MoO3 nanowires in the nanocomposite reduced the surface roughness but improved the mechanical properties, increased the wetting angle and thermal stability compared to the PVDF-HFP/PVP polymer composite. The results showed, that this PN exhibited excellent antimicrobial activity against bacteria, yeasts and molds [2]. The antimicrobial activity of PN is activated by water, which first slightly dissolves the surface of MoO3 nanowires. As a result, the molybdic acid is formed, which leads to hydrolysis of the PVP polymer and the formation of carboxylic acid and ammonium salts. [1] Gradišar Centa, U. et al. (2022), Coatings, 12(9), 1241. [2] Gradišar Centa, U. et al. (2020), Surface Innovations, 9(5), 256-266.

Oral (S05-360, Time: Tuesday 14:10, Room: EMS-Chemie)

Production of flexible and highly thermally conductive polymer pipes by targeted alignment of filler particles

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Pipes in heat exchanger systems are usually made of metallic materials to achieve a high level of thermal conductivity and therefore efficiency. However, substitution with polymer materials would have great potential. Plastics save weight, can be produced at lower cost and with lower emissions, and are ideally suited for use in corrosive or chemically aggressive environments in which most metallic materials fail. The challenge, however, is their much lower thermal conductivity by comparison, as polymers tend to be classified as thermal insulators. To compensate this, fillers are used which conduct the thermal energy through the polymer matrix. Though, the high filler ratio required to achieve an acceptable level of thermal conductivity has a significant impact on the material properties of the polymer matrix, such as the flexibility, which is drastically reduced. The consequence is a fragile material that is only conditionally suitable for use in heat exchanger systems. This paper therefore describes a novel approach to effectively increase the thermal conductivity of polymer materials while minimizing the use of fillers. In conventional die designs fillers are aligned in the direction of extrusion (axial orientation) due to shear on the die walls. However, this orientation leads to inefficient utilization of the filler potential to increase thermal conductivity through the pipe wall. To improve this, several die concepts for targeted alignment of filler particles in the direction of thermal flow through the pipe wall (radial orientation) are compared in this paper. The most efficient die concept allows an increase in thermal conductivity of up to 48 % compared to a conventional die with the same amount of fillers. Accordingly, the filler ratio can be reduced to maintain the flexibility of the polymer matrix to a greater extent.

Oral (S05-376, Time: Wednesday 14:50, Room: EMS-Chemie)

Experimental investigation toward processing of high performance nanocomposites with the fully sustainable PLA and well dispersed cellulose nanocrystals (CNCs)

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Currently, continuous extrusion of high performance polylactic acid (PLA) nanocomposites with good dispersion of cellulose nanocrystals (CNCs) meets the great difficulties. In this work, the novel strategy was designed to firstly study the effects of PEG-coated CNCs (P-CNCs) on the critical properties of PLA composites; and finally produce the large quantity PLA/P-CNC composites with high performance by the continuous extrusion process. The preparation of composites with PEG-coated CNCs in lab scare was performed using the freeze drying (FD) method: aqueous suspension including different contents PEG and CNC was mixed by solvent mixing; and then PEG-coated CNCs (P-CNCs) dried by freeze-drying were mixed into PLA to prepare composites using melt-mixing. Based on this, liquid feeding (LD) method evaporating water during continuous extrusion process was then comparatively explored. In addition to enhancing the dispersion of CNCs by coating, we found that the PEG, as a plasticizer and a carrier, could highly improve the toughness of composites and decrease the viscosity of system. Besides, CNCs and its coating PEO at the certain ratios had the synergistic effect of crystallization on PLA matrix. 9% P-CNCs with the appropriate PEG:PLA ratio 2:1 as the candidate was finally chosen to produce composites using LF method and compare with the same formulation prepared by FD. We found that sub-micros scare agglomerates of CNCs were still observed with negligible difference between FD and LF method, which was further supported shear rheological measurements. They also shown the considerable elongation at break and oxygen barrier properties compared with pure PLA. Finally, the composites using LF method are expected as a component to produce flexible multilayered films with barrier properties using co-extrusion process.

Oral (S05-402, Time: Thursday 16:20, Room: EMS-Chemie)

Optimized processing of Polyurethan/PET Composites manufactured by RTM

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Glass or carbon fiber reinforced polyurethane (PU) and low-density PET foams as core material are an ideal choice for structural components in all kind of transportation applications. An efficient processing route for the manufacture of such structures is the Resin Transfer Molding (RTM) process. Elevated temperature and pressure enable a fast curing, thus demolding of the finished part is possible within minutes. Process optimization for monolithic and sandwich parts though is crucial. To be able to manufacture lightweight RTM parts, new PU-systems have been developed and implemented in high-pressure RTM (HP-RTM, for monolithic parts) and low-pressure RTM (for sandwich parts). Extensive process optimization in parallel with mechanical testing was carried out to characterize the process in comparison with established material combinations. The obtained results show that PU-RTM is a promising combination, in particular concerning cycle times, costs and impact properties. Fatigue life has to be optimized by improving the fiber-matrix interface.

Oral (S05-406, Time: Wednesday 16:20, Room: EMS-Chemie)

Development of novel thermoplastic resin with low processing temperature and excellent adhesion to carbon fibers for aircraft structural composites

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Carbon fiber-reinforced composites with thermoplastics such as PPS, PEEK, PEKK, LM PAEK etc. have been utilized for aircraft structural parts due to their high toughness, formability, heat resistance and recyclability. However, the thermoplastics also have some drawbacks that they must be processed under very high processing temperature at least more than 330℃ and have relatively low interfacial adhesion strength between fibers and the matrices resulting in poor mechanical properties. In order to overcome these, we have developed a new thermoplastic matrix resin based on polyethersulfone (PES). The resin was blended with a Tg enhancer and a functional plasticizer to lower the processing temperature. The target Tg over 180°C and processing temperature less than 250°C of the blend could be adjusted by the appropriate mixing ratio of the additives to base PES resin. In particular, the composites with the resin showed better compressive strength and interlaminar shear strength as well as tensile strength than those of reference composites. The reason was confirmed by the fact that the resin has many functional groups originated from the plasticizer which can improve the adhesion with the carbon fibers. The functional groups were characterized by FT-IR analysis. In addition, we also confirmed through DSC measurement that the Tg and processing temperature were affected by the softening temperature of the plasticizer and its composition. Finally, we fabricated the sound carbon composites at 250°C displaying the suitable Tg (~177°C) measured by DMA and good mechanical properties. The CAI (Compression strength after impact) of the composites will be tested to prove the toughness which is one of the critical properties determining the preference of thermoplastic composites compared to thermoset composites.

Oral (S05-415, Time: Wednesday 16:40, Room: EMS-Chemie)

Micro and nano-structured multicomponent composites for ultra-high absorbance of Electro-Magnetic Radiation

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The present work deals with the development of nanostructured polymeric composites with high absorption of electromagnetic radiation. The strategy relies on tailoring their internal architecture leading to a morphological structuration with local electrical/magnetic properties through a nanolayer coextrusion and In-Mold Electronics process. In this regard, films containing conductive and/or magnetic fillers are investigated as support for Plastronic devices against electromagnetic pollution. The challenge is to obtain a 3D electrical/magnetic filler network with high orientation/ordered distribution. The chosen system for this work focuses on a PE matrix with different molecular architectures (linear and branched) containing fillers of various dimensions and shapes to be coupled with amorphous high-Tg polymers, used as confining materials. Therefore, the filler compounds were obtained through the dilution of masterbatches using a new Extensional Mixing Element for improved dispersive mixing in twin-screw extrusion. In this talk, SAOS experiments were firstly performed in the molten state to correlate the viscoelastic properties to the percolation depending on the characteristics of the fillers. Specific attention is devoted to investigate the rheological behaviors in correlation with morphological properties (SEM, TEM) to be compared to suspension models. Secondly, effects of flow were investigated in the nonlinear regime and in the transient regime that display less maturity in the rheology community, despite their importance for processing. Naturally, effects of strain rates in shear or elongation on the alignment of particles and viscosity/elasticity ratios, were explored in order to predict the coextrusion and IME processing windows. Finally, the multicomponent extruded systems were investigated through dielectric spectroscopy and EMI shielding measurements to determine their final electrical and magnetic properties and their shielding effectiveness of EMI.

Oral (S05-417, Time: Thursday 12:00, Room: EMS-Chemie)

Novel approaches for compression molding of LT-PEM fuel cell bipolar plates

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Functionalized polymers in general are claiming more and more novel applications. For example they can function as a metal replacement. Especially the E&E-market or green technologies require highly engineered polymers for many new needs e.g. a high level of thermal or electrical conductivity in polymers. To functionalize an intrinsic isolating polymer high loadings of conductive fillers, like graphite, carbon blacks, CNTs, metals and/or others, are necessary. The mixtures, so called compounds, exhibit a significantly different behavior in processing and in their properties compared to neat polymer. Thus far compounds were processed using either compression molding or injection molding. Both of these processes come with distinct limitations considering their application. The degree of filler in injection molding is limited due to increasing viscosity with increasing filler content. Compression molding has long cycle times and high energy consumption due to heating and cooling of the massive steel pressing mold. In an ongoing research project "Ultrapress2", a novel approach for compression molding of LT-PEM fuel cell bipolar plates will be discussed which can eliminate the disadvantages of previously mentioned common molding processes by using inductive heating paired with ultra-high-performance concrete as a molding tool. Another approach is studied in a second project "Esspresso". Here, the material is first compression molded only using temperatures up to 90 °C and are subjected to curing afterwards. This process decouples the time and energy consuming steps and therefore improves the efficiency of bipolar plate production.

Oral (S05-428, Time: Thursday 10:40, Room: EMS-Chemie)

Investigation of multifunctional architectures by tailoring flexible domains in inherently brittle epoxy-based composites through gradient interphase formation with bio-based thermoplastic elastomer grades

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Multifunctional materials are essential for innovative structural designs that benefit with no added material cost and weight. Combining distinct properties into a single material is welcomed in all engineering realms, from robotics to life sciences. Here, fibre-reinforced composites are important structural materials for future sustainable transportation, energy storage, and biomedical technologies due to their excellent combination of reducing weight and improving mechanical performance. This study focuses on multifunctional architectures in composite materials, tailoring elastic behaviour in an inherently brittle epoxy-based fibre-reinforced composite material through gradient interphase formation with a bio-based thermoplastic elastomer. Here, a fast-curing epoxy system for automotive applications was tested with two grades of a bio-based block copolymer. First, the interphase formation was characterised via optical hot-stage microscopy and Raman spectroscopy. The analysis revealed high diffusion rates followed by a reaction-induced phase separation resulting in a large interphase thickness of >120 µm at various curing temperatures showing the high affinity between the constituents. Second, a demonstrator was designed and manufactured that combines fused filament fabrication with a vacuum infusion process. Here, a flexible domain was integrated and tailored with variable stiffness properties, presenting a brittle–to–ductile material architecture at the micrometre scale. The demonstrator shows a flexible response in the bending direction without crack initiation – and stiff behaviour in the tensile direction. Consequently, the study anticipates using gradient material structures for more efficient and generative design concepts.

Oral (S05-429, Time: Thursday 11:00, Room: EMS-Chemie)

Flax/polypropylene composites for lightened structures

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The optimization of the processing method for poly-(propylene) (PP)/plant fibre composites compounding is a key point in the development of semi-structural parts, especially for automotive applications. The aim of this original and innovating work is to study, at different scales, the impact of extrusion equipment and fibre length on the composite's mechanical performances. Firstly, we studied the flax fibre morphology after compounding and injection as well as their individualization in injected specimens. Secondly, we focused on the effects they had on tensile properties. The impact of the processing tool was observed on fibre morphology and division; as well as the use of a BUSS system instead of a twin-screw extruder, which enhances the fibre individualization, however, the number of kneading areas should be reduced in order to preserve the reinforcement aspect ratio. We highlight the fact that the composite's mechanical performances are highly impacted by the fibre division and cell wall stiffness, as demonstrated by in-situ nanoindentation tests. Finally, the use of longer fibres is not a priority for the improvement of mechanical performances; it induces a more difficult fibre division and an increase in the compound viscosity, which could endanger the moulding process of the final part

Oral (S05-473, Time: Thursday 11:20, Room: EMS-Chemie)

Investigation of Electrical Conductivity Properties Wood-Based Panels

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Wood-based materials are one of the most used materials in engineering applications. Along with global warming and the increase of non-recyclable waste materials, thanks to their biodegradable, recyclable nature and being renewable source, wood products have recently attracted attention. Plywood, fiberboard, and particleboard are the three predominant types of wood-based panels and have been widely used as alternative material for furniture manufacturing, decorative panel manufacturing and construction due to their stable physical properties. The aim of this research is to developed smart fiberboards (MDF) for house flooring. Smart MDFs can be placed around kitchen and bathroom areas where flooding is most likely to be used as a flood warning sensor. In order for MDFs to be used as sensors, they must have an electrically conductive structure. In this study, conductive carbon black (CB) was added to the wood fibers at different ratios to make the MDF electrically conductive. The electrical conductivity of MDF under different moisture contents was investigated. In addition, internal bond tests were carried out to examine the mechanical properties of MDFs. According to the test results, the electrical conductivity of the MDFs increased with the addition of CB. However, a slight decrease in mechanical properties was observed. It was determined that the electrical conductivity was adversely affected for the samples left in an environment with high moisture content.

Oral (S05-527, Time: Thursday 11:40, Room: EMS-Chemie)

Reducing the supercritical CO2 permeation of pressure vessels at elevated temperatures using nanocomposites of high-density polyethylene and graphene nanoplatelets

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High-density Polyethylene (HDPE) has shown great potential in the design and development of liners for pressure vessels at elevated temperatures due to superior moisture and chemical resistance together with a good balance of strength and ductility. Aiming to mitigate carbon footprint, minimizing carbon dioxide permeation through this thermoplastic polymer, particularly at elevated temperatures and pressures, is of primary importance. To this end, two scalable and cost-effective material improvement strategies have been implemented in this study, i.e., a 2D particulate system and laminar structures based on Graphene Nano-Platelets (GNP) reinforced HDPE. Low Graphene content HDPE/GNP nanocomposites were manufactured by melt-mixing with nanoparticle weight fractions ranging between 0.1% to 2 %. Effects of processing conditions namely, temperature, rotational speed of a twin-screw compounder and cooling rate were examined to achieve optimum particle distribution as well as the degree of crystallinity. Compression molded nanocomposite coupons were tested using an in-house permeation cell with supercritical carbon dioxide at 60 °C. The mechanical properties of the nanocomposites were also investigated in terms of creep and dynamic moduli. Improved mechanical properties and a reduction in CO2 permeation of up to 40% resulted when using GNP contents lower than 0.5% weight fraction. The results suggest that the developed HDPE/GNP nanocomposites are useful as eco-materials for pressure vessels using HDPE liners.

Oral (S05-532, Time: Wednesday 15:10, Room: EMS-Chemie)

Fabrication of PBAT/HDPE blend-based nanocomposites with improved anti-dripping and physical properties

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Carbon nanotubes (CNT) and graphene nanoplatelets (GNP) were added individually into poly(butylene adipate-co-terephthalate) (PBAT)/high density polyethylene (HDPE) blend to fabricate blend-based nanocomposites. For the immiscible PBAT/HDPE blend, maleated HDPE acted as an efficient compatibilizer. SEM results revealed that CNT and GNP were finely dispersed in the PBAT matrix and HDPE domains. Sea-island blend morphology was transformed into a co-continuous morphology after loading CNT, but not GNP. Thermal property studies showed that CNT and GNP both assisted the nucleation and crystallization of HDPE during crystallization. Heat distortion temperature and thermal stability were improved by adding CNT or GNP to the blend, with CNT displaying a better efficiency than GNP. Burning tests also showed improved anti-dripping performance when CNT or GNP were added. An evident enhancement in rigidity of 3 phr CNT-loaded composite (34 and 88% increase in Young’s/flexural moduli, respectively, compared to the parent blend,) was observed. Impact strength of the blend increased by up to 87% at 3 phr CNT loading, whereas adding GNP had a negative effect. Rheological properties revealed the pseudo-network structure development in the composites. The 3 phr CNT and GNP loading reduced the electrical resistivity of the blend by nine and five orders of magnitude, respectively. References 1. R. E. Drumright; P. R. Gruber; D. E. Henton, Adv. Mater. 12, 1841, (2000). 2. F. C. Chiu; C. Y. Kan; J. C. Yang, J. Polym. Sci. PartB: Polym. Phys. 47, 1497, (2009).

Oral (S05-544, Time: Thursday 14:30, Room: EMS-Chemie)

Role of gelation and filler amount on mechanical properties of composite PVC highly filled CaCO3

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Polyvinyl chloride (PVC) composites are getting a lot of industrial interest for floor applications. However, the complex morphology, the gelation mechanism and its implication on mechanical properties remain not fully understood limiting the optimization of materials performances. PVC gelation process is supposed to play an important role on materials properties. Gelation involves grains break down, crystallite fusion, chains interdiffusions and recrystallization upon cooling. The aim of this research was to study the influence of gelation and the filler amount for highly filled PVC materials on the mechanical properties of the parts. The impact of the filler amount was investigated in the case of PVC filled with 30wt% and 70wt% of CaCO3. Both formulas were dry blended before extrusion at different temperatures (160, 175 and 190°C) and screw speeds (80, 100 and 120 rpm) to investigate the effect on gelation degree. The extrusion was carried out using a contra-rotative twin screw extruder with gravimetric feeder. For both filler contents, the gelation was characterized by swelling in acetone and DSC was used to calculate a gelation degree. This latter was obtained by the measurement of the areas of the endothermic peaks related to the fusion of the different crystalline parts. Mechanical properties were evaluated by both 3 points bending tests and Charpy impact tests. Structural evolution and the interface between PVC and filler were analyzed thanks to SEM observations. The increase of the amount of filler modifies greatly the mechanical properties leading to a more rigid material. In addition, a poor interaction PVC-filler is believed to cause a drop in stress and strain at break as well as fracture toughness at high filler rate. Results show significant differences in the gelation degree depending on extrusion temperature and screw speed. This improvement in gelation degree causes an increase in fracture toughness indicating a structural evolution.

Oral (S05-548, Time: Thursday 14:50, Room: EMS-Chemie)

Investigation of the Hybridisation Effect of Cost-effectively Compounded Pre-impregnated Glass Fabric on Sheet Moulding Compound

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Hybridised sheet moulding compound (hybrid SMC) with pre-impregnated glass fibre fabric (F-SMC) was identified as an outstanding solution for applying on the subcomponents of the battery casing based on the numerical analysis. A lower part weight and cost with fewer assembly processes than the state of art technology made of aluminium were shown. Aim to further evaluate the manufacturing feasibility of co-moulding F-SMC with basic SMC and the resulting hybridisation effect on the mechanical properties, the study below has been conducted. The manufacturing feasibility was investigated at an 1800 T hydraulic press at AZL. Two different F-SMCs with varied weft and warp ratios and different laminate architectures (sandwich structure and one-sided) were taken into account on a 3D tray-based part, similar to a battery tray. For the variant, F-SMC on one side, it is easy to be over-moulded by the basic SMC using the standard compression process parameters. For the sandwich structure, the cavity between the F-SMCs can also be filled. Both layouts present an excellent interface without any significant air voids investigated by optical microscopy. In addition, the mechanical properties were studied under the uniaxial transverse loading on one homemade test cell. Compared with the F-SMC on one side under compression stress, the sandwich structures provide a much more significant reinforcing effect with an increase in the flexural modulus, strength and absorbed energy at failure by 61.3 %, 78.2 % and 88.4 %, respectively. The reinforcing effect was also proved by a reduced sample strain at the maximal load without any dominant concentration, indicating a mild and homogenous requirement on the fibres. After the lifetime, the components can be easily recycled with the hybrid SMC’s consistent resin and fibre type.

Oral (S05-553, Time: Thursday 16:00, Room: EMS-Chemie)

Highly thermally conductive PEEK/hexagonal boron nitride composites realized by scalable twin-screw extrusion: thermal transport properties and rheological behavior

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To meet the growing thermal management demands in the electronic and aerospace industry, the development of thermally conductive lightweight polymeric materials has received a lot of attention. Although polyetheretherketone (PEEK) offers excellent performance in terms of dimensional stability, chemical resistance, and tolerance in extreme environments, its low intrinsic thermal conductivity (~0.2 W/mK) greatly restricts its use in areas requiring high heat removal rates. In the present work, thermally conductive yet electrically insulating hexagonal boron nitride (hBN) filler has been incorporated in the PEEK matrix by an optimized twin-screw extrusion technique. The use of a bespoke screw profile and optimization of feeding zones and process cycles resulted in PEEK/hBN composites demonstrating exceptionally high in-plane and through-plane thermal conductivity of 12.451 and 2.337 W/mK, respectively. The superior heat dissipation characteristics of prepared composites were validated by a non-destructive infrared thermography (IRT) technique. Compared to neat PEEK, the PEEK/hBN composite, at 60 wt% loading, has a 34 times higher thermal diffusivity owing to the presence of a well-connected hBN network. In addition, we performed a comprehensive rheological characterization of the prepared composites. A progressive increase in complex viscosity and the shear thinning behavior of composites was noted as a function of filler amount. Moreover, to outline the allowable melt viscosity ranges for each composite composition, viscosity buildup profiles were constructed using the temperature and time-dependent rheological results. These profiles can be utilized to tailor the residence time of a composite melt by varying the filler concentration and process temperature during advanced manufacturing processes especially extrusion-based additive manufacturing (AM). In conclusion, these PEEK/hBN composites can be used for commercialization and large-scale production of high-performance polymer-based heat diffusers in next-generation thermal management systems.

Oral (S05-557, Time: Tuesday 15:10, Room: EMS-Chemie)

Study of carbon nanotube and organoclay-filled PLA/PVDF/PMMA ternary blend-based nanocomposites

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Carbon nanotubes (CNTs) and organoclay (30B) were included into poly(lactic acid) (PLA)/poly(vinylidene fluoride) (PVDF)/poly(methyl methacrylate) (PMMA) (70/30/30) ternary blend, with PMMA serving as a compatibilizer for the PLA and PVDF phases. SEM results showed that PMMA tended to entangle more with PVDF than with PLA, and thus formed a pseudo co-continuous PLA(rich)-PVDF(rich) phase morphology in the blend. The added CNTs and 30B clay were mainly distributed in the PVDF(rich) phase and PLA(rich) phase, respectively, to achieve nanocomposites. The co-continuous bi-phasic morphology was more evident in the composites than in the parent blend. Thermal property results showed the nucleation effect of CNTs and 30B on the crystallization of PLA and PVDF; the presence of PMMA obviously retarded the crystal growth of PVDF due to their miscibility. Adding CNTs or hybrid CNT/30B evidently improved the anti-dripping performance of the ternary blend in burning tests. The elongation at break (EB) of PLA/PVDF blend drastically improved with PMMA inclusion, and the composites also showed excellent ductility. The EB of PLA and PLA/PVDF blend increased by up to 18.9 and 14.4 times, respectively, after forming the 3-phr hybrid CNT/30B-loaded composite. Rigidity of the PLA/PVDF/PMMA blend increased after forming the composites; flexural modulus increased by 16% with 3 phr CNT loading. Rheological property analysis suggested that (pseudo)network structure formation in the composites. The electrical resistivity of the ternary blend decreased by 11 orders at 2 phr CNT loading (double percolation achieved), and the percolation threshold was at 0.5 phr CNT loading.

Oral (S05-570, Time: Thursday 16:40, Room: EMS-Chemie)

Toward the minimization of residual stress in thermoplastic composite wound parts

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The use of thermoplastic composite materials in the different industrial sectors has expanded considerably over the past few decades, notably wound structures such as pressure pipes, tanks and reservoirs. Filament winding is a widely used manufacturing technique to produce these thermoplastic composite structures which present multiple advantages such as an increasing productivity. Rapid manufacturing usually involves thermal gradients through the thickness of the structure. The addition with other phenomena such as in-situ chemical reactions leads to residual stresses in the final structure. This is due to the difference in the thermal expansion coefficients of the different constituents and the evolution of the matrix volume due to polymerization or crystallization. The residual stresses then affect the structural integrity of the part and can lead to premature cracking when external service loads are applied. In this study, an experimental characterization of the residual stresses is carried out along with a numerical model in order to assess the residual stresses level in a two steps process wound structure, then process parameters effect on these stresses are discussed. Finally , different solutions are proposed in order to reduce the residual stress linked to the manufacturing parameters.

Oral (S05-579, Time: Friday 10:40, Room: EMS-Chemie)

Developing Nano-fibrillated Flame Retardant Polymer Composites: Addressing Deteriorated Mechanical Properties

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High flammability of thermoplastics has imposed catastrophic fire incidents in buildings, automobiles, and electronics devices. This has led to the enforcement of strict fire safety standards and regulations to mitigate the extensive fire risks and damages. To meet these standards, additive flame retardant (FR) compounds have been widely applied to reduce polymers' flammability. Incorporating halogen-free FRs is an effective strategy to fabricate fire-proof polymers without detrimental impacts on the environment or human health. Nevertheless, developing polymeric materials with fire retarding features by inclusion of FRs, whether in high or low contents, compromise other properties, particularly mechanical properties. This work demonstrates the promise of nanofiber rubbers in compensating the deteriorated mechanical properties of the flame-retardant composites. The main matrix is polystyrene, which is extremely brittle and incorporating flame-retardant such as nanomaterials within the matrix intensifies its brittleness. We demonstrate that presence of nano-fibrillar toughening fillers effectively improves the toughness of the composites without sacrificing the stiffness. Nanofiber rubbers show considerable effectiveness in enhancing the matrix's toughness. To maintain the rubber's fibrillar morphology, high temperature rubbers such as chemically modified Thermoplastic Polyester Elastomer (TPEE) was used as the rubber phase. The tailored composite containing modified TPEE nanofibers and hexagonal boron nitride (h-BN) as the flame-retardant nanomaterial shows high fire retarding efficiency and mechanical performance including toughness. Thus, the proposed strategy can be implemented to fabricate flame-retardant composites with enhanced mechanical properties.

Oral (S05-580, Time: Friday 11:00, Room: EMS-Chemie)

Tailoring asymmetric filler arrangement towards enhanced through-plane thermal conductivity of polymer composites

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Thermally conductive polymer composites with asymmetric fillers exhibited highly anisotropic thermal conductivity (i.e., a limited through-plane thermal conductivity), since asymmetric filler were volunrable to orientate along flow direction during melt proccessing. By introducing nonconductive hollow glass microspheres (HGμS), we observed that the through-plane thermal conductivity of polymer composites containing flake graphites (FG) was increased instead of decreasing when replacing FG with 3 wt% HGμS. The increase of through-plane thermal conductivity resulted from the dilution of FG by HGμS was ascribed to the reduced structural anisotropy of FG network in the presence of HGμS. In other words, the in-plane orientation of FG along the flow direction was inhibited during melting proccessing. By futher replacing FG with the combination of HGμS and alumina (Al2O3) microspheres, a synergy of enhancement of through-plane thermal conductivity was observed. Such a synergy was attributable to the combination of "bridging" of FG platelets (by Al2O3) with the "tailoring" of FG arrangement (by HGμS).

Oral (S05-610, Time: Friday 11:20, Room: EMS-Chemie)

New reinforcing approach for bio-based UV-curing resins: hybrid lignocellulose fillers with improved synergy and wood structure mimics

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Lignocellulose is the most abudant natural polymer and lignocellulosic fillers have been widely investigated for various polymer blend applications. Great interest in lignocellulose is related to the fact that they have great potential for using wood biomass and recycled plastics. However, besides cellulose nanocrystals (CNC) and nanofibrils (CNF), not much of the lignocellulose has been used for UV-curing resins, and none in hybrid compositions. In the present work, the effects of hybrid lignocellulose fillers – lignin (LN) / CNC / CNF / hemicellulose (HC) ratio-varying compositions on UV-curing bio-based resins have been investigated for wood mimic material coatings. The effects on the macromolecular chain network, surface morphology, thermomechanical properties, and thermostability were carefully studied. A combination of scanning electron microscopy, DMA Cole-Cole plot, contact angle, etc., allowed us to get a sense of hybrid filler distribution and interaction with the polymer matrix. It was identified that an increase in the HC content of up to a certained point contributes to more even particle distribution. HC also contributes to porous surfaces, while CNC smooths surface morphology and CNF increases defects. Thermomechanical testing revealed the unique benefit of hybrid reinforcement, showing higher results than single filler compositions. 17 different compositions clearly show the benefit of hybrid lignocellulosic reinforcement and the ability to tune performance and achieve wood structure mimicking coatings. Acknowledgement: This research was supported by Riga Technical University’s Doctoral Grant program.

Oral (S05-626, Time: Friday 11:40, Room: EMS-Chemie)

Polymer Blend/Composites for Single-Use Applications

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The polymers used for packaging applications are more than one third of globally produced plastics each year. The big chunk of this are single-use plastics which mostly ends up in landfills and oceans creating serious environmental pollution. To overcome this issue polyesters are the promising materials with ester linkages that have potential to hydrolysis in certain ambient conditions. However, choice of raw materials and polymer microstructure is the determining factor to make a polyester eventually biodegradable. Furthermore, the right combination of these polymers with a variety of organic and inorganic fillers can fulfill the requirement of desired material for different applications. For example, the neat polybutylene adipate terephthalate (PBAT) has certain limitation in processing to be used in product development. These limitation can be overcome by its compounding with other polymers such PLA, PBS, PHA etc. along with inorganic (Calcium carbonate, talc, silica) and organic fillers (starch, lignin, natural fibers). The present work shows a facile method for PBAT compounding with PLA and inorganic fillers (Calcium carbonate, talc) to develop blend/composite suitable for application development. Further, the effect of blend formulation and process conditions (temperature profile, pressure etc.) on the characteristics of blend/composite (thermal, mechanical, and microstructure) are described.

Oral (S05-643, Time: Thursday 14:10, Room: EMS-Chemie)

Design and production of polymer nanocomposite multilayers for efficient EMI shielding

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Electronic devices emit local electromagnetic (EM) waves. These secondary EM waves may interfere with the operation of neighbouring electronic components or the wireless EM networks that connect them; this phenomenon is known as EM interference (EMI). Devices can be protected from electromagnetic radiation by a layer of shielding material where the incident power is reflected by and/or absorbed throughout the shield. Metals are conventionally used as EMI shielding materials. These exhibit high shielding effectiveness values, yet most of the incident power is reflected to the environment due to the metal's high electrical conductivity. To reduce this secondary EM pollution, one can make use of polymer-based shielding materials, which can partially absorb the incident EM power. The challenge is to produce polymer composite-based shields exhibiting sufficiently high shielding effectiveness where shielding is dominated by absorption instead of reflection. An attractive design strategy for EMI shielding is the stacking of layers with alternating electromagnetic properties. In this way, wave absorption can be enhanced via multiple wave reflections between the layers. This principle has been explored experimentally in the recent literature, yet some outstanding questions remain on how the interplay between the electromagnetic properties, geometry and stacking order of the composite layers dictate the multilayer's shielding properties. The aim of this work is twofold. We make use of an analytical transfer matrix model to elucidate the effect of stacking on EMI shielding performance and identify optimal design cases for PMMA-CNT shields. Selected cases are manufactured via a compression molding route and tested to validate the use of the model. Emphasis is place on (1) challenges encountered during processing and electromagnetic testing of the shields and (2) future avenues for 3D-printing of nanocomposite shields to facilitate more complicated gradient structures.

Oral (S05-645, Time: Friday 12:00, Room: EMS-Chemie)

High-perfomance EMI shielding polyester / carbon nanoparticles composites with tailored structure

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The current growing worldwide challenges to defend society from military, cyber and hybrid threats and attacks demand to development of new strategies to resist. One such challenge is an electromagnetic defence of critical infrastructure, as well as mission-critical equipment. Energy distribution, information and communication systems, transportation, safety, and economic security infrastructures are all vulnerable to electromagnetic interference (EMI). The high-frequency EMI elimination remains a significant challenge to be solved even nowadays. For example, the growing threat from very small and inexpensive but high-power, high-frequency devices in the hands of terrorist organizations are posing new challenges. These current needs provide a significant opportunity to cultivate the development of novel advanced polymer materials for cybersecurity technologies. Polymer composites are investigated to assess the rising threat of intentional high-power signal interference by improving resilience to electromagnetic effects (e.g., EM attacks). Nanostructured biobased polyester blends combined with carbon nanoparticles with tailored metal oxide are investigated for the present needs.

Oral (S05-679, Time: Thursday 15:10, Room: EMS-Chemie)

An efficient interface model to develop scalable methodology of melt processing of polypropylene with graphene oxide produced by an improved and eco-friendly electrochemical exfoliation

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Substantial effort has been made to prepare graphene-based polymer composites with all kinds of morphologies, dimensions, structures, and properties. Among those polymers, polypropylene (PP) is commonly used in thermoplastic composite applications as a matrix since it possesses high heat distortion temperature, transparency, flame resistance, dimensional stability, and high impact strength. Moreover, significant weight reduction can be achieved when PP is integrated into commodity products due to its low density compared to other polyolefins. Despite some improvements in the PP/graphene nanocomposites, poor dispersion and agglomeration of graphene still remain a challenge. Therefore, this study aims to develop a scalable and straightforward adaptation methodology for melt processing of polypropylene (PP) to provide a high degree of exfoliation of multilayer graphene oxide (GO) by using a high-shear mixer. Electrochemically produced GOs were synthesized and directly used in the production of melt-blended PP composites. GO was first produced by an improved and eco-friendly electrochemical exfoliation by using an environmentally friendly aqueous methanesulfonic acid (MSA) and a sodium sulfate salt system to minimize the environmental impact. The produced GOs then were melt blended with PP and their mechanical, thermal, morphological, and rheological properties were investigated under different GO loadings. The characterization results indicated that GO obtained in MSA solution caused a 71% increase in flexural modulus and 46% in flexural strength with the addition of 1 wt% GO. The rheological characterization also showed that dispersion and viscosity improved with lower GO loadings compared to the neat polymer by providing cost-effective and scalable graphene manufacturing. GOs,which are produced much faster by electrochemistry, can be used directly in composite productions, and have the potential to significantly increase the performance of the polymer composites.

Poster (S05-058, Time: Thursday 17:00, Room: Foyer)

Developing Highly Conductive Polystyrene/PEDOT:PSS/Carbon Nanotube Nanocomposite with Segregated Structure for Electromagnetic Interference Shielding

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Electromagnetic pollution is a major concern for public health and the proper functioning of electronic devices. The commonly used electromagnetic shields (i.e., metals) are susceptible to corrosion, hard to process, and expensive. Conductive polymer nanocomposites (CPN) are a promising alternative. However, constructing a well-established conductive network within a polymer matrix, vital for electromagnetic interference (EMI) shielding, is still challenging using conventional processes. This research aimed to enhance the EMI shielding performance of CPN by developing highly conductive segregated structures through a simple process. Polystyrene (PS) beads (0.2–25 µm) were synthesized using an emulsion-evaporation technique. The conductive components were carbon nanotube (CNT) and poly(3,4-ethylenedioxythiophene):polystyrene sulfonate (PEDOT:PSS). The nanocomposites were fabricated by dispersing PS beads, CNT, and PEDOT:PSS in deionized water, followed by vacuum filtration, solvent treatment, and hot press molding. CNT particles showed a higher tendency to assemble with PS beads of smaller size, which led to the formation of a segregated structure in the molded sample. Moreover, PEDOT:PSS promoted the conductive network by constructing a conductive bridge between CNT particles. The solvent-treated PS/PEDOT:PSS/CNT nanocomposite exhibited high electrical conductivity of 2.35 S/cm and specific EMI shielding effectiveness of 55.7 dB/mm (with dominant absorption mechanism) at 2.0 wt.% CNT and 4.0 wt.% PEDOT:PSS loading. This research proposed a simple approach to develop an effective EMI absorber shield.

Poster (S05-063, Time: Thursday 17:00, Room: Foyer)

Production and characterization of glass fiber/LDPE composite material for w-beam guardrail

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Guardrails are common and important elements of the road safety system. Among the semi-rigid roadside barriers, steel w-beam guardrails are dominating, although they are prone to corrosion and incur relatively high costs of maintenance. As an alternative, fiber-reinforced polymer materials, possessing such advantages as resistance to corrosion and fatigue, elastic deformation capacity, and low density, are being considered. Both thermoset and thermoplastic matrix composites have been studied for roadside restraint applications, the latter having the advantage of more ductile deformation response. Replacing of the steel by a thermoplastic polymer composite as the guardrail material is likely to bring economic, environmental (e.g. utilization of recycled plastic), and functional benefits. The aim of the present study was developing of an efficient production technology of E-glass fiber/low density polyethylene (LDPE) guardrail. A laboratory-scale rolling and thermoforming line has been developed allowing continuous feeding of LDPE sheets with glass-fiber yarn reinforcement located between them. In the rolling machine, LDPE sheets are subjected to a short-term heating to ca. 400 ˚C causing partial melting of the sheets and penetration of the polymer into the yarns. The in-plane mechanical properties of the composite produced were characterized by tensile and compressive tests along the reinforcement direction, transverse tensile tests, rail shear, and compact tension tests. The data obtained were used for modelling of w-beam guardrail response, suggesting that the thickness of composite sheet needs to be markedly greater than that of the steel one to ensure comparable structural rigidity and load-bearing capacity. A quasi-static validation tests of prototype guardrail elements have been performed revealing their load-bearing capacity.

Poster (S05-150, Time: Thursday 17:00, Room: Foyer)

Fabrication and photodegradation behaviors of biodegradable copolymer/organically-modified layered double hydroxide nanocomposites

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A series of biodegradable copolymer/organically-modified layered double hydroxide (m-LDH) nanocomposites were successfully synthesized by polycondensation reaction and solution-mixed processing. The results from the Fourier transform infrared spectroscopy (FTIR) and wide-angle X-ray diffraction analysis of the m-LDH indicated the occurrence of organic modifier intercalation. FTIR and gel permeation chromatography were used to characterize the evolution of the biodegradable copolymer/m-LDH composites after being artificially irradiated via a light source. The change of carboxyl group produced via photodegradation were analyzed to illustrate the enhanced UV protection ability of m-LDH in the composite materials. From the experimental observation, the carbonyl index of the biodegradable copolymer/m-LDH composite materials after photodegradation for various times was significantly lower than as compared to that of the pure biodegradable copolymer matrix. These results were also confirmed by the molecular weight reduction in biodegradable copolymer with increasing m-LDH content, possibly due to the UV reflection ability by the m-LDH. This study presents a novel approach of improving the UV photodegradation of a biodegradable polymer using an organically modified layered double hydroxides.

Poster (S05-240, Time: Thursday 17:00, Room: Foyer)

Composite materials from recycled HDPE and hemp fibers - Compatibilization

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The environmental problems faced by our modern societies are one of the major concerns of the 21st century. The general public is looking for more sustainable products, pushing industries and scientific communities to seek for greener processes and products. Waste valorization has attracted a great deal of interest since it would allow for a more efficient use of natural resources. On the other hand, recycling is necessary especially for polymeric materials that are not biodegradable, and EU has set high goals in terms of waste recycling. A drawback of recycling is the progressive deterioration of the mechanical properties of the recycled materials. It is well known that natural fibers can reinforce polymeric materials, improving their properties. In this context, we designed sustainable composite materials from recycled high-density poly(ethylene) (HDPE) and hemp fibers, from agricultural wastes. HDPE being a rather hydrophobic material and hemp fibers being rather hydrophilic, the use of compatibilizers to improve the adhesion of the fibers to the polymeric matrix is necessary. In the present work, two different compatibilizers were studied. The dispersion of the fibers in the polymeric matrix and the interfacial adhesion were examined by scanning electron microscopy (SEM). The impact of the compatibilizers on the mechanical properties of the composites was further investigated. Acknowledgements This research has been co-financed by the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH-CREATE-INNOVATE (project code: T2EDK – 00008).

Poster (S05-278, Time: Thursday 17:00, Room: Foyer)

Compounding of Very Long Nanotubes into Polycarbonate

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Our group has previously looked at the effect of aspect ratio on the properties of polycarbonate-multiwalled carbon nanotube composites Journal of Polymer Science Part B: Polymer Physics, 52, 73 (2014). However, we were limited to aspect ratios of ~500 (~5 micron tubes, ~10 nm diameter). Our group has recently developed a method to produce tubes of aspect ratio ~5000 with the same diameter with yields (nanotube:catalyst+support) of 30:1. By breaking the tubes using a ball mill, we have investigated the relationship between nanotube length and thermal, electrical and rheological properties in the region between 5000:1 to 500:1. Of significant interest is how nanotube length decreases with mixing in a conical twin-screw extruder and how there is a balance between nanotube dispersion and nanotube length with respect to electrical conductivity and rheological properties.

Poster (S05-289, Time: Thursday 17:00, Room: Foyer)

Talc characteristics from composite rheology

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Conventionally the Wagener and Reisinger power law model quantifies the shear thinning behaviour of a polymer-clay composite in an effort to describe filler/matrix interaction. Here the use of the Wagener and Reisinger power law model to infer characteristics of large particle in polymer composites is examined. Specifically, can the shear thinning exponent of systems with comparable composition (i.e. the volume rate is equivalent, the matrix is identical) be used to classify fillers in terms of their size. Different talc types were used to prepare composites with a polypropylene copolymer formulation. Rheological investigations were carried out using a parallel plate rheometer. Both granular and injection moulded samples were tested in order to account for degradation effects that occurred during processing. Since the volume occupied by the filler particles were the same for all the composites, any difference in flow behaviour would result from the specific filler talc used. The methodology would allow for comparison of especially aspect ratios of different talc grades while considering, that during processing, changes in basic filler properties might arise due to delamination, cleavage of agglomerates or even breakage of lamella. Within these trials an order of averaged particle sizes from smallest to largest could be identified with evaluation of the shear thinning coefficient. The results further indicate a sensitivity to processing.

Poster (S05-336, Time: Thursday 17:00, Room: Foyer)

Preparation of Polypropylene random reinforced with SiO2 and Montmorillonite nanofillers with enhanced dimensional stability for piping systems

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Due to climatic changes occurring during the last decades, extreme temperature deviations are observed throughout the year. These changes may cause considerable damage to the hot/cold water piping assembly by the continuous thermal expansion-contraction of the piping material leading to crack initiation and finally the rupture of the pipes over time. This study focuses on the preparation and characterization of materials, which present low thermal expansion and enhanced mechanical properties. Polypropylene Random copolymer (PP-R) is used widely as a piping material. To address the issue of dimensional stability, PP-R multilayer pipes are reinforced with a filler (most commonly Glas Fibers at large amounts) reducing their thermal expansion. The main goal of this study is the development of PP-R composites using fillers at low contents, enhancing the dimensional stability together with PP-R's mechanical properties. Two sets of PP-R nanocomposites were prepared, containing SiO2 nanoparticles and Montmorillonite nanoclays (MMT), respectively at various loadings (1-15 wt.%). The prepared materials' morphological characterization was conducted using Scanning and Transmission Electron Microscopy (SEM, TEM). Thermal stability was estimated by Thermogravimetric analysis (TGA), while the melting and crystallization behavior was evaluated by Differential Scanning Calorimetry (DSC). A dilatometer was employed for the thermal expansion measurements. Tensile tests were carried out for the mechanical properties characterization of neat PP-R and its prepared nanocomposites. The incorporation of the nanofillers in the PP-R matrix induced enhanced dimensional stability while the elastic modulus of the PP-R/SiO2 nanocomposites increased considerably for low filler contents (1-2.5 wt.%). Acknowledgments Financial support provided by the Operational Program Competitiveness, Entrepreneurship and Innovation (EPANEK 2014-2020/Action RESEARCH–CREATE–INNOVAT, project code: T1EDK-02575).

Poster (S05-369, Time: Thursday 17:00, Room: Foyer)

Polyvinyl alcohol/custom-synthesized graphene nanocomposites: effect of graphene functionality on rheological properties and EMI shielding performance

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Graphene is a wonder material with unique two-dimensional (2D) structures. Its excellent electrical conductivity, large specific surface area, and intrinsic mechanical flexibility make it an ideal filler to enhance the mechanical, electrical, and thermal properties of nanocomposite materials at much lower loadings than traditional fillers. In this study, three different graphenes with diverse surface functional groups and polarity were prepared by electrochemical exfoliation and pre-treatment process using different acids, namely phosphoric acid, sulfuric acid, and nitric acid. The highest polarity was associated with the sample pre-treated in nitric acid. In the next step, conductive polymer nanocomposites were prepared by mixing the graphenes with poly (vinyl alcohol) (PVA) matrix using solution mixing method. The effect of surface functional groups and polarity of nanofillers on the formed nanostructure and thus the final properties of each nanocomposite were analyzed by optical microscopy, linear and nonlinear rheology and electrical measurements (e.g., conductivity and EMI SE). Our results showed that the EMI shielding/electrical conductivities are highest for samples containing graphenes pre-treated with nitric acid. Samples containing graphenes pre-treated with sulfuric acid at the same concentrations have the second highest values and are similar to samples containing graphenes pre-treated with phosphoric acid at the same concentrations. The enhanced EMI shielding and electrical properties of the nanocomposites containing graphenes pre-treated with nitric acid were attributed to the enhanced dispersion of this graphene due to its higher polarity compared to the other graphenes.

Poster (S05-514, Time: Thursday 17:00, Room: Foyer)

Development of Advanced Composites through Surface Characterization of Fibers and Fillers by Inverse Gas Chromatography: A review

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The ultimate quality and performance of polymer based fiber/nano-fiber composites depends strongly on the interfacial interaction of the components. In particular, the surface energy and surface area of fibers and fillers are directly related to the compatibility, dispersibility and reinforcement quality. To enhance the adhesion properties at the interface, fibers/fillers are often exposed to various surface treatments. In this studies, surface areas and energies of fillers and natural fibers from different origins and surface treatments have been investigated by Inverse Gas Chromatography (IGC). IGC is a well-known tool for the characterization of particulates, fibers and films. IGC experiments were carried out using iGC Surface Energy Analyzer with a flame ionization detector. Whole fiber samples (without cutting) between 1.0-1.5 grams were packed into silanized glass columns. Surface area and energies of natural fibers and cellulose nano-fibers from different sources and surface treatments were investigated in this work, using IGC. The excellent reproducibility of the analysis establishes that the IGC technique can be applied to a wide array of natural based fibers and nano-fibers. In addition, surface energy measurements elucidated further surface properties that could be used to completely characterize the surface properties of nanomaterials. Nevertheless, IGC helps to design the interface compatibility of filler and matrix.

Poster (S05-572, Time: Thursday 17:00, Room: Foyer)

Preparation and characterization of composite materials from wastes of olive oil production

Zamboulis Alexandra[1], Xanthopoulou Eleftheria[1], Malletzidou Lamprini[1], Papadopoulou Electra[2], Chrissafis Konstantinos[1], Bikiaris Dimitrios N.[1]

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According to the International Olive Council, the European olive oil production is around 2.000.000 tons/year, approximately 2/3 of the world production. The cultivation of olive trees and the production of olive oil generate important volumes of wastes which are generally dumped without further processing. In this context, the aim of the present research work is to propose solutions to valorize these wastes. High-density poly(ethylene) (HDPE), a versatile but petroleum-based thermoplastic, is one of the most widely used polymers nowadays. Despites its numerous properties, it is not biodegradable, contributing to the environmental pollution and the generation of microplastics in the oceans. The obvious solution is to recycle HDPE, nevertheless a drawback of recycling is the progressive deterioration of the properties of the polymer, especially the mechanical properties, resulting in many cases in the downcycling of the polymer as a filler in other plastics. It is well-know, that the addition of fillers, besides endowing the polymer matrix with new properties, can contribute to improve the mechanical properties. To reinforce recycled HDPE, we designed composite materials with olive core wood, a waste generated from olive oil production. The preparation and characterization (infra-red spectroscopy, X-ray diffraction, scanning electron microscopy) of these composite materials will be presented in the present communication. Acknowledgements This research has been co-funded by the European Regional Development Fund (ERDF) and Greek national funds through the Operational Program "Competitiveness, Entrepreneurship and Innovation", EPAnEK 2014-2020, under the call "Aquaculture"-"Industrial materials"-"Open innovation in culture" (project T6YBP-00161).

Poster (S05-585, Time: Thursday 17:00, Room: Foyer)

Study of ecofriendly composite materials from recycled HDPE and hemp fibers

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As a concept, circular economy aims to transition from a linear economic system, to a more sustainable one, by re-using resources in alternate circles, in order to balance our use of (finite) natural resources. Recycling and re-using are principal pillars of this idea. Additionally, the valorization of wastes is another strategy to decrease the use of new resources and increase the sustainability of our modern societies. Despite its numerous advantages, high-density poly(ethylene) (HDPE) is a non-biodegradable polymer; a lot of efforts have been put into recycling HDPE without deteriorating its mechanical properties. In this context we have prepared a series of composite materials from recycled high-density poly(ethylene) and hemp fibers, isolated from agricultural wastes, in the presence of Joncryl, used as a compatibilizer. These eco-friendly composites were designed to replace conventional wood/plastic composites. Furthermore, the utilization of natural fibers in order to reinforce polymeric materials is a well-known strategy and in low contents, hemp fibers are expected to improve the mechanical properties of recycled HDPE. The materials were characterized by infra-red spectroscopy and X-ray diffraction. The molecular weight of the materials was estimated by measuring the melt-flow index. Finally, the dispersion of the fibers in the polymeric matrix was examined by scanning electron microscopy (SEM). Acknowledgements This research has been co-financed by the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH-CREATE-INNOVATE (project code: T2EDK – 00008).

Poster (S05-589, Time: Thursday 17:00, Room: Foyer)

Study of novel bio-based epoxy resins from adipic acid

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Polymers are an integral part of our societies and the development of synthetic polymers has significantly contributed to the improvements of our quality of life. Nevertheless, these victories came with a price, namely environmental pollution. To promote a more circular economy and to reduce dependency on crude oil, which is a finite resource, substantial resources have been thrown in the fields of bio-based polymers. As their name suggests, epoxy resins are a class of polymers that contain epoxide groups. They have a wide range of applications, from coatings to electronics, composites and adhesives. In the present work, we will present novel bio-based epoxy resins from adipic acid. Adipic acid, one of the most industrially important carboxylic diacids, has been identified as a suitable platform chemical for bio-based production. Epoxy resins from adipic acid were synthesized in a two-steps synthetic process, and further characterized. Curing was investigated with two cyclic amines. Encouraging results were obtained and the epoxy resins will be further used as bio-based adhesives for three-layered ecological panels, with panels from dead Posidonia oceanica leaves.

Poster (S05-598, Time: Thursday 17:00, Room: Foyer)

The influence of hydrochar as a partial filler on the rubber products mechanical properties

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The rubber products have a wide range of applications, and its typical mixture recipe contains Sulphur, essential additives and reinforcement filler. Carbon black (CB) is a traditionally used filler, due to its low cost and graphite crystal structure which improves the final product's thermal and mechanical properties. The CB production is highly dependent on fossil energy sources (crude oil, coal and natural gas), and often generates various pollutants during use, and represents a cost of resources. It is of great importance to find an appropriate replacement of conventional raw materials with renewable ones, in order to obtain environmentally friendly rubber products. The dominant non-renewable filler in the rubber industry can be partially replaced, with a sustainable, alternative biofiller, obtained by hydrothermal carbonization of waste and renewable agricultural materials. The hydrothermal carbonization process parameters were optimized ($T = 300$ °C, $p = 86.6$ bar, $t = 30$ min) in order to obtain hydrochar with maximum carbon content and to reduce energy consumption and increase the complex process efficiency. The attention was devoted to determine the innovative rubber mixture recipe containing optimal hydrochar proportion, beneficial to obtain the products for special applications. The samples with different ratio of hydrochar and CB were prepared, and the mechanical properties (hardness, tensile strength, elongation at break) were tested. The obtained studies revealed that the mixture containing low ratio of hydrochar can partially replace CB, without causing the significant difference in rubber products mechanical properties. The production of "green" materials could provide the appropriate conditions for the circular economy application, since agricultural secondary products and waste biomaterials were reused through the process of hydrothermal carbonization, and became an important raw material for the production of sustainable rubber materials.

Poster (S05-600, Time: Thursday 17:00, Room: Foyer)

Influence of curing on fracture behaviour of electrically conductive adhesives

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Motivation For assembling electrical contacts to screen printed silver tracks a reliable and easy to apply connection method is required. The reliability and processing of different electrically conductive adhesives (ECA) were investigated in literature before [1]. In this study the adhesion of ECAs to screen printed silver tracks on coil coated steel sheets under different curing conditions were investigated. This contribution shows there are notable differences in the fracture pattern depending on the used curing method. Materials and Methods For this study adhesion strength was measured and the fracture pattern of tested adhesive joints was evaluated. In this survey adhesive joints were prepared with several commercially available ECAs. Silver pads printed on coil coated steel sheets served as a substrate. The silver pads were screen printed with different commercial silver inks. The adhesives were cured at varying temperatures and times in a convection oven or by inductive heating of the steel substrate. The adhesive strength was examined by measuring the tensile strength of adhesive joints with a pull-of-test adapted from industry standard DIN EN ISO 4624. The fracture pattern of the tested probes was investigated by light microscopy. Cross sections of samples were inspected with scanning electron microscopy. Results Fracture patterns could be classified to adhesive and cohesive failures as well as mixtures of them. The curing method had an influence on fracture behaviour. Variation of curing time, curing temperature and heating source resulted in either adhesive or cohesive fracture for the same adhesive joint. Pull-of-forces of up to 1.2 kN were measured for some adhesives. Conclusion With ECAs strong connections can be achieved on printed silver tracks, if a correct curing procedure is applied. [1] U. Eitner et al., Characterization of Electrically Conductive Adhesives, Energy Procedia, Volume 27, 2012, Pages 676-679, ISSN 1876-6102

Poster (S05-637, Time: Thursday 17:00, Room: Foyer)

Effects of Bondline and Substrate Thickness on the Mode II Fracture Toughness of Composite/Adhesive Joints

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Glass fiber reinforced plastic (GFRP) is commonly used in wind turbine blades (WTB). The joining of several GRFP components in a WTB is usually obtained using structural adhesives. The GFRP/adhesive joints usually experience mixed-mode loading (opening and shear), with mode II being a major phase. The experimental evaluation of the shear behavior is usually conducted using the end-notched flexure (ENF) test, which provides the value of the fracture energy required for the onset of interlaminar crack propagation by shear forces. There are standard methods such as ASTM D7905 that have provided the procedure for the test. This method has particularly been developed for crack growth and fracture energy evaluation in unidirectional fiber composites not composite/adhesive joints. However, this method has also been used for the Mode II fracture energy evaluations in composite/adhesive joints. To better understand the applicability of standard ENF method for composite/adhesive joints of WTBs, this work focuses on the dependency of the measured critical strain energy release rate (GIIc) on the specimen geometry, in particular, substrate thickness, adhesive layer thickness, and pre-crack location. The substrate effect on the fracture behavior was assessed using three different substrate thicknesses (3, 5, 6.3 mm) and the bondline thickness was examined from 0.25 to 4.00 mm. Overall, with an increase in the substrate thickness, GIIc was increased, and the fracture surface examination suggested a more cohesive failure behavior. The bondline thickness also significantly affected the GIIc value. GIIc first was increased with an increase in the bondline thickness up to a maximum value. At very large thicknesses, e.g., 4 mm, the GIIc value dropped. The location of the pre-crack, either on one side of the adhesive layer or in the adhesive layer mid-plane did not seem to have a significant effect on the measured GIIc.

Keynote (S06-065, Time: Thursday 10:15, Room: Sulzer Chemtech)

Investigation of the influence of screw geometry on the resulting melt quality for cast film extrusion

Trienens Dorte[1], Schöppner Volker[1]

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In plastics processing, polymers are plasticized so they can be further processed into products or semi-finished products in the extrusion process. During the lifetime cycle, the plastic is processed at least once on screw machines on its way from the raw material or granulate to the product. In order to achieve an increase in throughput with identical product quality, the extrusion process must be designed optimally. Since the construction and testing of prototypes is very costly and time-consuming, only empirical values and simulations remain as reference points for screw design. However, simulations based on the current state of the art do not allow the expected product quality to be predicted with sufficient accuracy. Up to now, the product as well as the melt quality can only be determined in experimental investigations and shown as a function of various process properties, such as pressure changes and degree of melting. In this paper, the cause-effect relationships are first revealed experimentally in an extensive extrusion test program. Since good melt quality has a significant influence on product properties, suitable criteria for assessing and evaluating melt quality are first selected and verified by means of experimental investigations. In order to determine the influence of changes in the geometry of a screw concept on melt quality, two different screw geometries each of a barrier and standard three-zone screw were investigated, as well as the influence of two shear and mixing part combinations. Thermal and material homogeneity are used as criteria for melt quality. Based on this, the correlations between input and output parameters will be described in an analytical model. The integration of the model into extrusion simulations will then allow the prediction of the expected melt and product quality.

Keynote (S06-622, Time: Wednesday 17:00, Room: Sulzer Chemtech)

Numerical Simulation and Experimental Validation of Twin-Screw Compounding of Polylactide and Polybutylene Succinate Blends

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The morphology of thermoplastic blends is a major factor in governing their mechanical properties. Thus, the morphology development of thermoplastic blends during twin-screw compounding is an area of prime interest in polymer industry. Therefore, the aim of the study was to correlate simulated process data of twin-screw compounding with experimentally found morphologies of blends from biobased Polylactide (PLA) and Polybutylene Succinate (PBS). Numerical simulation of the mixing process of a lab-scale twin-screw extruder was done with ANSYS Polyflow software for various blend compositions and screw speeds. As a result, a maximum local shear rate of 2041 1/s was found for the highest simulated screw speed of 400 1/min for the PLA/PBS-20/80 blend in the dispersive mixing section of the screw. Interestingly, no significant influence of the blend composition on maximum local shear rate but on maximum local shear stress was found. An increasing content of PBS led to an increase in maximum local shear stress with its peak at 212 MPa for the PLA/PBS-50/50 blend. Further increase in PBS content led to a reduced maximum local shear stress, indicating a co-continuous morphology at equal content of PLA and PBS in the blend. Subsequently, the resulting values of the maximum local shear stress for each blend were used to calculate the capillary number and to predict the droplet breakup according to the Grace model. The experimental validation of the numerical simulations was done by dead-stop compounding experiments and morphological analysis of blend samples along the screw by Scanning Electron Microscopy. In result, a good correlation of the predicted particle breakup by numerical simulations and the capillary number approach and the experimentally found particle diameters was found for all studied blend ratios and screw speeds. Thus, the studied workflow could help to reduce the needed numbers of experiments to adjust the morphology and properties of PLA/PBS blends.

Oral (S06-003, Time: Wednesday 16:00, Room: Sulzer Chemtech)

Development of an asymmetric torque model for the two screws of a twin screw extruder

Austermeier Laura[1], Schöppner Volker[1]

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In order to modify a polymer with additives or fibers in a way that is suitable for processing, co-rotating twin-screw extruders are generally used. An important factor in the melting of the plastic, which is necessary to homogeneously incorporate other components, is the energy input. Thermoplastics soften before they liquefy and finally, when subjected to excessive stress, are damaged and eventually decompose. A targeted energy input in the melting zone, which is sufficient to plasticize the material without damaging it, is therefore desirable. Bute apart from that, other zones like mixing zones should not be neglected. The underfed condition in which twin-screw extruders are usually operated results from the metering of the components, as well as the different flight pitches of the screw elements, which are used to build up and down for different process steps. In the extruder, a partial filling of the chambers with plastic melt occurs, which simplifies, for example, the degassing of the melt. Due to the same direction of rotation of the two screws, there might be a different distribution of the total melt or granules to the two screws and a different energy input. The present approach to determining the local torque input is based on the assumption that a resistance is created by the plastic, which torsions the screws. This torsion can be calculated to the required torque and thus the energy input can be determined. The higher the filling level of the screws, the higher the resistance and thus the higher the energy input. In order to show the influence of the degree of filling of the screws on the energy input in the extruder, investigations were carried out on a twin-screw extruder in which different partial fillings were provoked and the applied screw torque was measured.

Oral (S06-033, Time: Wednesday 16:20, Room: Sulzer Chemtech)

Extension of the Melting Model for Co-Kneaders regarding the Plastic Energy Dissipation

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The co-kneader is a single-screw extruder, which performs an oscillating motion in extrusion direction additionally to rotating. The four rows of kneading pins along the cylinder walls combined with the interrupted screw flights make a high mixing possible, while enabling gentle processing at manageable shear forces and exact temperature control. Although the co-kneader is used for a wide variety of material systems as polyvinylchloride or polyamide and especially for special applications in the industry, its process design is mainly based on experimental trials. Therefore, an analytical model which allows a description of polymer melting and flow in the kneader was developed by Rudloff et al. In this previous work satisfactory simulation results could be achieved when using a retaining ring at the end of the melting zone which causes material backlog. This enables to define the melting start at the begin of the first mixing element and to assume a fully filled melting zone as well as apply dispersed melting models. However, if the co-kneader is operated without a retaining ring, the pellets can pass through the melting zone and only plastic deformation occurs, until the melt film is formed further towards the die by heat conduction. In this paper, a first model approach of plastic energy dissipation (PED) for Co-Kneaders is developed to specify the start of melting more precisely for all configurations. In the model the energy dissipation was formulated as a function of granulate geometry and weight, mechanical material parameters and degree of granulate deformation. This allows to extend the existing dispersive melting model to include the melting contribution from PED.

Oral (S06-044, Time: Wednesday 16:40, Room: Sulzer Chemtech)

PCU Twin Screw, a new machine concept focusing on homogenization & degassing of recycling material streams (regrind, films)

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In recycling, material streams are collected, shredded, sorted, and washed before regranulation and further usage. We have developed a new innovative machine based on a pre-conditioning unit (PCU) directly feeding a twin-screw extruder. Combining the advantages of a pre-conditioning unit: (i) moisture reduction, (ii) handling of fluffy materials with low bulk density, (iii) bulk density increase, (iv) homogenization, and (v) preheating of the solid input material with the superior melt homogenization and degassing capacity of a twin-screw extruder provides the possibility of novel applications. The superior mixing and degassing capability of a fully intermeshing co-rotating twin-screw extruder in direct comparison to a single-screw extruder can considerably improve the quality of the repellets. However, the advantages come with disadvantages, e.g. hard contaminants can lead to wear on the twin-screw extruder due to the narrow clearances and the intersection area. Additionally, the melt cannot be filtered before degassing, which would considerably impair the quality of material streams with undesired gaseous impurities (e.g. paper, wood).

Focusing on the special tasks for handling recycling streams, the machine concept will be critically compared with existing recycling technologies available on the market based on the end product quality, energy consumption, degassing efficiency and throughput.

Two single-screw concepts (INTAREMA TVEplus®, INTAREMA TE) will be compared with the twin-screw concept. The comparison of specific energy consumption for a defined material stream is particularly relevant because the system boundaries of twin- and single-screw extruders are commonly defined at different process steps for recycling materials with high humidity, low bulk density and high impurity, which makes a fair comparison impossible.

The combination of pre-conditioning unit and twin-screw extruder (PCU-DS) enables us to set similar system boundaries for direct comparison and also include energy consumption in decision process.

This work shows the development of the new PCU-DS system, a comparison of different recycling systems in terms of quality, degassing efficiency, energy consumption and mass throughput as well as theoretical design guidelines.

Oral (S06-187, Time: Thursday 12:00, Room: Sulzer Chemtech)

Thermodynamics of water-assisted compounding

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In recent years, water-assisted extrusion has become increasingly popular in the production of mineral or bio-nanocomposites to achieve good dispersion between matrix and filler and to avoid agglomeration in the composite. In the process, water is injected into the extruder at high pressure and temperature to remain in the liquid state and is removed at the end of the process by degassing with a vacuum pump. Water-assisted compounding can thus combine the advantages of good dispersion and safe operation with low investment and operating costs. At the IWK (Institute for Materials Technology and Plastics Processing), projects have already been carried out in the field of water-assisted compounding of coffee grounds for thermoformed cups. It has been noticed that the process stability must always be monitored when adding fluids, as the addition of water has a great influence on the thermodynamic conditions of the system and the thermal behaviour of the material, such as the freezing point, in the running process. Especially when up-scaling the entire water-assisted compounding process to larger production plants, the existing thermodynamic processes in the extruder play an important role and it would be advantageous to be able to analyse them more precisely. Therefore, the IWK has set itself the goal of evaluating methods to better understand the thermodynamic processes using the example of a co-rotating twin-screw extruder and thus gain a view into the processes inside the extruder. In addition to monitoring a wide range of process parameters in operation during trials, calculations and simulations were also carried out on the thermodynamic processes for the compounding process. The presentation will thus show possibilities and results from the existing work at the IWK for a better understanding of the thermodynamic processes in extrusion.

Oral (S06-315, Time: Thursday 10:40, Room: Sulzer Chemtech)

Production of eco-friendly PLA composite by using waste cellulose and vermiculite

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Concerns about environmental pollution and human health drive attention on usage of environmentally friendly resources instead of petroleum-based polymers in order to decrease carbon footprint. Especially automotive, house appliances and electronic industries which require improved mechanical properties for their products benefit from petroleum-based polymers therefore they highly contribute the CO2 emission. In this scope, waste cellulose and vermiculite were used as reinforcement for polylactic acid to be able to obtain fully biobased and durable polymeric compounds. Fiber structure of cellulose makes polymer stiffer by increasing modulus whereas vermiculite improves flexural strength thanks to its layered structure. These eco-friendly additives at different loading levels combined with a bio-based polymer. Thermokinetic mixer was used to prepare waste cellulose and vermiculite added polymers, so that a difficult-to-process material such as cellulose can be easily processed. To examine mechanical properties tensile tests and there-point bending tests were conducted. Composite materials prepared with waste cellulose and vermiculite showed excellent performance characteristics in terms of mechanical properties compared to neat polymer. A synergistic ratio between vermiculite and waste cellulose was found. 22.5% waste cellulose and 7.5% vermiculite loaded formulation has superior mechanical performance. Young's modulus and flexural modulus were improved by 137% and 127%, respectively. Also contrary to expectation this synergistic formulation shows enhanced bending properties compared to the other compounds.

Oral (S06-396, Time: Thursday 11:00, Room: Sulzer Chemtech)

Improvement of flowability of PP/GF by high-shear rate processing in the specially designed single screw extruder

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We applied high shear rate to PP/GF using a high-shear extruder and examined the flowability and mechanical properties. We found that the spiral flow length was much more increased due to thermal decomposition of PP, but the tensile strength and the tensile modulus were not varied so much despite of large screw rotational speeds more than 1000 rpm. This was because the shear stress acting on the glass fibers was small due to the viscosity reduction and the fiber length was not shortened. Using the processed PP/GF, we produced vibrating plates for speakers whose thickness were less than 0.5 mm.

Oral (S06-407, Time: Thursday 11:20, Room: Sulzer Chemtech)

Study on the Behavior of Carbon Fibers in the Kneading Process Using Twin-Screw Extruders

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Fiber-reinforced thermoplastics (FRTP) which have excellent specific strength, specific impact, durability, and heat resistance, are widely used as materials for parts that require light weight and high strength．It is known that the mechanical properties of FRTP molded products improve until the specific fiber length．However, fiber breakage occurs during the-compounding, or injection molding process, and results in defects of reduced strength．Therefore, the development of the process and simulation technologies that can control the carbon fiber length distribution is highly desired in the kneading process of twin-screw extruders．In this study, using polypropylene (PP) with different length of carbon fibers (CF) as the base material, the effect of kneading block configurations on fiber breakage was investigated by experimentally and numerically．The combinations of three types of kneading blocks were used: R (Right-handed kneading block），N (Neutral kneading block）and L (Left-handed kneading block） as the screw configurations. The mean shear stress carried out by time dependent flow and particle tracking analysis to clarify the fiber breakage. As a result, it was found that no breakage occurred when the initial fiber lengths was about a quarter of the root depth, and the breakage rate was also small regardless of the value of the mean shear stress. It was also found that fiber breakage was accelerated when the initial fiber length was longer relative to the screw root depth and the mean shear stress played important role. The above results indicate the possibility of a establishing design technology that can control the fiber length distribution.

Oral (S06-587, Time: Thursday 11:40, Room: Sulzer Chemtech)

The effect of mixing factors on dispersion, degradation, and tensile properties of HDPE/UHMWPE blends

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High density polyethylene (HDPE) / ultra high molecular weight polyethylene (UHMWPE) blend is expected as an all-polyolefin recycle material that can achieve both excellent mechanical properties and a continuous melt-mixing process. In order to obtain the good mechanical properties of polymer blends, optimal mixing achieving good dispersibility and low degradation is required. However, few studies have focused on the relationship among the melt mixing, the homogenization, the degradation, and the quality of the materials. In this study, the effect of mixing factors on the dispersion behavior and degradation behavior and tensile properties of HDPE/UHMWPE blends were investigated. Blends of HDPE with 5% UHMWPE were obtained by melt mixing in internal batch mixer. Specific Energy Input (SEI), rotation speed of the screws, and atmosphere during mixing were changed. Dispersion behavior of the blends was evaluated using a microscopy and a differential scanning calorimetry (DSC). Degradation behavior of the blends was evaluated using Gel Permeation Chromatography (GPC), oscillatory shear test, infrared spectroscopy and chemiluminescence measurements. The mechanical properties of the samples were evaluated by using a tensile testing machine. From the results of microscopic observation and DSC, the dispersibility progressed with the increase in SEI regardless of the screw rotation speed. The result of GPC shows the molecular chain scission progressed with the increase in SEI regardless of the screw rotation speed. The result of oscillatory shear test shows the formation of branch structure by degradation were affected by both SEI and rotational speed. Both molecular chain scission and formation of branch structure by degradation was suppressed by mixing under nitrogen atmosphere. The relationship between dispersion and degradation and mechanical properties will be discussed on the day.

Poster (S06-445, Time: Thursday 17:00, Room: Foyer)

In-line Monitoring of Pharmaceutical Small Scale Extrusion based on UV-Vis Spectroscopy

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In pharmaceutical technology, the embedding of active ingredients in polymeric carrier materials offers the potential to optimize their pharmaceutical effect by tailoring the dissolution profile in the application. One promising example is the extrusion of amorphous solid dispersions (ASDs) where a poorly soluble drug is molecularly dispersed in a highly soluble amorphous carrier polymer in order to enhance the bioavailability in oral administration. However, this approach only achieves its full effect if the drug is completely dissolved in the polymer. Within this study, a novel method is proposed to monitor the dissolution of drug particles in polymeric carriers in-line during pharmaceutical small scale extrusion. Therefore, a twin screw mini compounder was coupled with special UV-Vis probes that are characterized by their small dimensions. The amount of undissolved drug particles was evaluated based on the transmission, since the light is increasingly scattered if it hits remaining drug crystals. This setup enables a systematic formulation design and optimization through the evaluation of dissolution kinetics using small material quantities. In fact, sample quantities of about 5 mg were evaluated for each measurement which represents 50 wt% of all material inside the die. It is shown that the temperature has a significant effect on the drug dissolution in the polymer. Furthermore, complete drug dissolution is shifted to lower temperatures if higher residence times are applied. Based on the courses of light scattering, regime maps were modeled that specify the process conditions where ASDs are successfully manufactured.

Poster (S06-608, Time: Thursday 17:00, Room: Foyer)

Numerical investigation of continuous mixing of highly viscous non-Newtonian multicomponent fluids by a single-screw extruder

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Proper mixing of highly viscous non-Newtonian multicomponent fluids can be complicated, but it is relevant for the food, catalyst, medical device, and concrete industry to name a few. Continuous mixing is an attractive option, as it can lead to more homogenous products as well as reduced processing time and energy consumption. A well-known method to perform continuous mixing is through an extruder. In this study, a computational fluid dynamics model is developed to simulate mixing of highly viscous non-Newtonian multicomponent fluids in a starved-fed single-screw extruder. The material behavior is described with a Herschel-Bulkley model and the free surface of the material is captured by the volume of fluid method. The model is developed in order to evaluate dispersive and distributive mixing. The first is quantified by Manas-Zloczower mixing index, while the latter is assessed by the Kramer mixing index. The results of the numerical model illustrates that varying the process parameters of the single-screw extruder have an effect on the two mixing indexes.

Keynote (S07-001, Time: Thursday 16:40, Room: Sulzer Chemtech)

Melt-spun biopolyester fibers for sustainable and biomedical applications

Perret Edith[1], Röthlisberger Moritz[1], Dul Sithiprumnea[1], Giovannini Giorgia[1], Hufenus Rudolf[1]

[1]Empa, St. Gallen, Switzerland

Melt-spun biopolyester fibers made of e.g. poly-3-hydroxybutyrate (P3HB) or polycaprolactone (PCL) have attracted great interest for sustainable and biocompatible applications. Such fibers have to meet high demands for biomedical, filtration or biotextile applications. Some applications demand e.g. biodegradable shock-absorbing ductile textiles, while others demand high-tenacity fibers. Stress, temperature and drawing strongly affect the structure and thus also the mechanical performance of the fibers. It is possible to obtain e.g. ductile fibers that have viscoelastic properties, or fibers that are rather brittle but have high tensile strength. In the past, it has been shown that mesophases can strongly influence the physical properties of melt-spun fibers. The cross-sectional microstructure (crystallinity, molecular orientation, phase distribution) is of particular interest for technical textiles, since it affects surface characteristics and mechanical properties of the fibers. It is, however, very challenging to obtain such 2D structural information across polymer filament cross-sections with diameters below e.g. 100 µm. The first part of this presentation will focus on the microstructure analysis (molecular orientation, amorphous, crystalline and mesophase domains) of P3HB fiber cross-sections using polarized high-resolution Raman spectroscopy. PCL fibers have attracted interest for biomedical applications, e.g. for wovens to form scaffold material for tissue engineering or as suture materials. The second part of this presentation will focus on preliminary results of melt-spun liquid filled PCL fibers for drug-delivery purposes. A variety of spinning trials, mechanical testing and drug diffusion results will be presented.

Keynote (S07-037, Time: Thursday 13:45, Room: Sulzer Chemtech)

Variety of structure in the extruded strands of PC containing PMMA

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Structure development during capillary extrusion was studied using binary blends composed of bisphenol-A polycarbonate (PC) and low-molecular-weight poly(methyl methacrylate) (PMMA). Even below lower critical solution temperature, miscibility was found to be affected by extrusion conditions. Flow-induced demixing was detected for the blend containing 30 wt% of PMMA. The blend also showed flow-induced mixing at higher shear rates. Because the shear rate decreased with the distance from the surface, the extruded strand showed a core-shell type structure. Moreover, pressure-induced demixing was detected for the blends with 20-30 wt% of PMMA, at which all area in the strands became opaque. Finally, the concentration gradient occurred without phase separation in the blend with 10 wt% of PMMA. The surface of the extruded strand was found to be rich in PMMA, leading to a good scratch resistance, which was pronounced with increasing the applied shear rate.

Keynote (S07-177, Time: Friday 13:45, Room: Sulzer Chemtech)

New insights into crystallization of heterophasic isotactic polypropylene by fast scanning calorimetry

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An important structural parameter of heterophasic isotactic polypropylene copolymers are the crystalline phases of the iPP matrix as well as of the dispersed ethylene propylene copolymer (EPC). Heterophasic iPP copolymers have an extremely wide application area in thin wall packaging, automotive and pipe products, where the speed of solidification is playing a significant role on the final structure and properties. The solidification process is strongly dependent on temperature and therefore largely affected by the conditions of processing, often involving fast cooling of the melt. For neat iPP, much progress in characterization of the kinetics of crystallization was achieved by employing fast scanning chip calorimetry (FSC), allowing application of process-relevant crystallization conditions. At cooling rates below 150 K/s the neat iPP crystallizes at high temperatures in the stable monoclinic phase building lamellar crystals arranged in spherulitic superstuructre. Cooling at rates above 150 K/s triggers at relatively low temperatures formation of mesomorphic phase of nodular crystal habit without presence of spherulites. The different crystal habits were related to different nucleation mechanisms explained by the bimodal temperature-dependence of the crystallization rate. The two maxima, around 30 and 80 °C, were associated with homogeneous and heterogeneous crystal nucleation, respectively. Considering that EPC particles due to blocky ethylene or propylene sequences exhibit different properties in comparison to fully amorphous particles, or an iPP matrix with a semicrystalline morphology altered by EPC nucleation effects, both affecting ultimate properties, analysis of the correlation between the synthesis-controlled molecular architecture and the rather complex crystallization of such heterophasic copolymers is well justified. For this reason, in the present work the crystallization of both iPP-matrix and ethylene-rich EPC particles prepared by

Keynote (S07-458, Time: Friday 10:15, Room: Sulzer Chemtech)

Solid phase biaxial orientation of polymers

Coates Phil D.[1], Caton-Rose Fin[1]

[1]University of Bradford, Bradford, United Kingdom

Solid phase orientation processing of polymers (at temperatures between the glass transition temperature and melting temperature) leads to enhanced property products, exploiting process-induced molecular orientation, in crystalline and amorphous regions. A range of highly successful axially oriented products have been obtained from our research. More complex biaxial orientation is reported here, demonstrating fundamental research reaching through to enhanced property oriented tube products. These include: • die drawn PLLA tubes (95 micron wall thickness) for bioresorbable arterial stents, with Arterius Ltd), • die drawn polyolefin water pipes (32-160mm outside diameter, with SABIC - ‘BiAx’ pipes recently announced, • shape memory die drawn PETG soft tissue to bone fixations (with Innovate Orthopaedics and the Fortius Clinic for sports injuries); • along with some fundamental in-process structure development studies using real time in-process WAXS/SAXS and off line characterisation with Changchun CIACCAS. The die drawing process in crystalline polymers is shown to ‘lock’ the oriented structure better than free drawing does, leading to high tensile properties which are retained. The balance of axial and hoop orientation in biaxial tube die drawing is a complex function of material and process geometry, and needs to be controlled for example to achieve critical radial strength in stents, and a balance involving low axial orientation yet adequate hoop orientation, both of which vary through the thickness, in pressure pipes. Another paper will cover the finite element modelling of these processes. Also, by contrast, amorphous orientation is relaxed at body temperature in the PETG fixations, causing shape change to make an interference fit with bone. Fuller details of this are in a separate paper on shape memory fixations.

Oral (S07-093, Time: Thursday 14:10, Room: Sulzer Chemtech)

Correlation between morphology and processing parameters in polymer blends: preliminary results towards hierarchical structures

Cravero Fulvia[1], Frache Alberto[2], Arrigo Rossella[2]

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[2]Politecnico di Torino, Alessandria, Italy

Hierarchical polymer-based materials can be defined as blend characterized by a structural order at different scale levels. The system may be made of two or more polymers and a filler can be present. The increasing interest on these composites is due to the possibility to tune the morphology and, consequently, the properties of the overall system. In fact, several studies focus on the correlation of the morphological structure with the mechanical properties, the electrical conductivity, the permeability and the optical properties. One of the main issues related to hierarchical polymer refers to the production techniques. Traditionally, these systems are obtained via a bottom-up chemical approach. This brings along limitations such as the production of small batches and a non-negligible environmental impact due to the use of solvents. Another approach is the top-down production from the melt, as in the case of multilayer extrusion. In this case, the main issue is the restriction on the morphological freedom correlated to the structural constraints of the production tools. In the last years, another approach consisting in the bottom-up production from the melt has been increasingly studied. With this method, the inner morphology of the system develops during the blending or the forming step. The reciprocal arrangement is allowed by a combination of factor. Specifically, both processing parameters and materials features play an important role in the final morphology. It is important to note that the last approach associate the freedom in tuning the morphology proper of a bottom-up method and the benefits of a solvent-free in-line process. For the above reasons, the correlation between the inner morphology and the processing parameters (e.g. temperature, strain) for different polymeric systems is investigated. In particular, blends of bimodal polyethylene also with the addition of UHMWPE, homo and copo polyamides characterized by different viscosities have been studied.

Oral (S07-216, Time: Thursday 14:30, Room: Sulzer Chemtech)

Impact of Orientation Temperature and Resin Density in structural evolution and morphology of MDO-PE films

Guleria Dixit[1], Doelder Jaap den[1], Vervoort Sylvie[2], Ragaert Kim[3], Ge Shouren[4]

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PE based mono material flexible packaging design presents a new alternative to conventional design as it can be efficiently mechanically recycled. The work focusing on outer structural layer of novel design addressed the impact of principle process parameter (Orientation Temperature) and resin parameter (Density) on the structural evolution and final morphology attained by MDO (Machine Direction Oriented)-PE films. 5 different PE resins and their blends chosen for study were processed in a lab scale setup to produce MDO-PE films. One selected resin was further processed at different orientation temperatures during stretching. Tensile tests were performed prior and after MD stretching. X-Ray scattering (SAXS and WAXS) were performed for the analysis of shape, size and orientation of the crystals. AFM (Atomic Force Microscopy) presented the surface morphology. Results showed that deformation and fibrillation of initial crystalline structure was relatively higher in high density resins and their blends. Crystallite dimension and long period increased with increase in orientation temperature indicating higher partial melting and strain induced re-crystallization. Increase in apparent crystallinity was relatively higher for higher density resins and blends. Lower density resin and blends presented comparatively low roughness at the surface in the AFM analysis. Orientations quantified by Herman orientation factors combined with X-ray diffractograms presented highly oriented structure towards stretch direction. Our study revealed the structural evolution of PE films going through MDO process and concluded that enhancement in crystallinity due to fibrillation and re-crystallization of initial crystal structure at relatively higher orientation temperatures in combination with highly oriented structure in stretch direction leads to enhancement of stiffness of MDO-PE films which can be further used as outer layer in PE based design for flexible packaging applications.

Oral (S07-230, Time: Thursday 14:50, Room: Sulzer Chemtech)

ZIF-8 decorated DNA-shaped polymer strips for efficient removal of methylene blue from aqueous solution

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An immobilization of metal-organic frameworks (MOFs) on electrospun products for wastewater treatment has gained considerable attention in recent years. However, the comprehensive relationship between MOF-decorated electrospun architectures and their performance has been investigated rarely. In this study, we prepared polycaprolactone (PCL)/polyvinylpyrrolidone (PVP) strips with DNA-like geometries via immersion electrospinning. By regulating the weight ratio of PCL to PVP, the morphology and the surface-area-to-volume ratio of the PCL/PVP strips could be precisely controlled. Then, zeolitic imidazolate framework-8 (ZIF-8) for removing methylene blue (MB) from aqueous solutions was immobilized on the electrospun strips, resulting in ZIF-8 decorated PCL/PVP strips. The key characteristics of the obtained composite products, such as adsorption and photocatalytic degradation behaviors for MB in an aqueous solution, were carefully investigated. Benefiting from the geometrical feature and high surface-area-to-volume ratio of the as-prepared strips, the ZIF-8 decorated PCL/PVP strips exhibited a high MB adsorption capacity of 151.6 g/mg, which is significantly higher than those in the conventional electrospun straight fibers. In addition, high MB uptake rates, high recycling and kinetic adsorption efficiencies, and high MB photocatalytic degradation rates could be confirmed. This work provides new insight into the rational design of electrospun products as MOF substrates for wastewater treatment applications.

Oral (S07-299, Time: Thursday 15:10, Room: Sulzer Chemtech)

Adhesion modified polypropylene: a sticky situation

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To improve the compatibility of material constituents in light-weight polymer composites or post-consumer recyclates, a maleic anhydride grafted polypropylene (MAH-g-PP) is commonly added to the blend. While the addition of a highly polar compound significantly increases the adhesive properties, the exact role of this modified polypropylene on the improvement of mechanical performance is still unclear. In this work, the crystallization behavior of such polypropylene blends is experimentally investigated under quiescent, shear flow and elongational flow conditions. The addition of a small amount of compatibilizer leads to only a slight acceleration of the crystallization kinetics in quiescent conditions, yet it markedly enhances the crystallization rate when a mild (and strong) flow is applied. Besides a significantly faster kinetics when the system contains a maleic anhydride compatibilization, in the case of shear flow the formation of oriented structures is hindered and the appearance of the beta-modification is suppressed, which in turn affects the mechanical properties of the material. These findings could not have been anticipated based on the quiescent crystallization kinetics and rheological behavior of the blends. In addition, in an elongational flow we measure the same acceleration of the kinetics, yet no difference in crystal type nor crystallite orientation is observed. These combined results highlight the importance of understanding the crystallization kinetics under processing relevant conditions in order to get a step closer towards full control over the crystallizing microstructure, and the subsequent mechanical performance of polyolefin based recyclates and/or composites.

Oral (S07-352, Time: Thursday 16:00, Room: Sulzer Chemtech)

Producing tough, stiff PC nanocomposites with enhanced properties by improvement of compatibility of PC matrix and EPDM rubbery nanofibril network with coupling agent and in-situ nanofibrillation

Akrami Hamidreza[1], Zaoui Aniss[1], Kheradmandkeysomi Mohamad[1], Salehi Amirmehdi[1], Park Chul B.[1]

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In this research, we introduce the concept of in-situ nano-fibrillation as an efficient, low-cost, and environmentally friendly tailored method for the enhancement of polycarbonate (PC) properties. PC/ Ethylene Propylene Diene Monomer Rubber (EPDM)-fibril composites are prepared by a twin-screw extruder. With the presence of a coupling agent and improvement in the compatibility of the PC as the matrix and EPDM as the nano-fibrillar phase, making polar/polar nanocomposites, promising the enhancement more in the rheological and mechanical properties of PC properties. Taking advantage of the better-crosslinked rubber phase as well as nano-fibrillation processing play the main role in properties improvement. Modifications of the mechanical and rheological properties of PC via fiber-spinning of PC/EPDM are distinguished by elongation and crosslinked network of the second phase (EPDM) properly in the main matrix (PC). Morphological observations showed the well-dispersed and distributed fibrillar phase of EPDM with a high aspect ratio in the PC matrix. PC with Nanofibrillated EPDM and coupling agent also improved the mechanical properties, especially the ductility and the toughness, while increasing the stiffness, in comparison with neat PC and PC/EPDM composite. The change in the tensile, Izod Impact and flexural properties was governed by the draw ratio. Hence, having more stretched fibrils due to increasing the compatibility of the matrix and nanofibrillar network is an effective way to maximize mechanical and rheological properties. Rheological investigations proved that PC with nanofibrillated EPDM has dramatically improved melt elasticity compared with neat PC. Linear viscoelastic behaviour of small amplitude oscillatory shear measurements showed a strain-hardening, solid-like, behaviour in the fiber-spun PC/EPDM, which was not observed in the neat PC or the melt-blended PC/EPDM.

Oral (S07-434, Time: Thursday 16:20, Room: Sulzer Chemtech)

Orientation control of carbon films by addition of graphene oxide in the precursor film

Takarada Wataru[1], Yokoyama Takehito[1], Shioya Masatoshi[1]

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Highly oriented graphite materials have excellent thermal conductivity and are used as thermal diffusion films. The authors aim to establish a novel method for the preparation of highly oriented graphite materials from polymeric films. Some polymer films can obtain carbon films by firing at high temperatures, but in many cases the carbonization yield is low and the orientation of the resulting carbon films is also low. In this study, we tried to improve the orientation of carbon films by addition of graphene oxide (GO) in the precursor film. A solution of phenol resin and alkali-treated lignin with 10 wt% GO was used as the precursor solution. The precursor film was formed by the solution-coating method, and carbon films were prepared by carbonizing at 1000 degree C. No orientation was observed in the carbon film prepared without GO, but in the carbon film prepared with GO, the graphite layer tended to be oriented parallel to the film surface. It was also confirmed that the addition of GO increased the electrical conductivity of the carbonized film. These results indicate that carbon crystals had grown from GOs that were oriented during the preparation process of the precursor film.

Oral (S07-516, Time: Friday 10:40, Room: Sulzer Chemtech)

High strength of PP by a high-pressure press

Nishitsuji Shotaro[1], Ito Yukino[1], Sano Hironari[1], Ishikawa Masaru[1], Inoue Takashi[1], Ito Hiroshi[1]

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Polypropylene (PP) is a thermoplastic polymer with low density, excellent ductility and rigidity, and so on. Since PP is a crystalline polymer, these mechanical properties are greatly affected by the morphology. The morphology of PP has a hierarchical structure. The spherulites, which have a size of several microns, are composed of a branched structure of crystal lamellae. The molecular chains are regularly folded in the crystal lamellae. In other words, controlling this morphology is the key to achieving high performance. The purpose of this study is to develop PP isotropically strengthened by press. In the case of press, since the material spread isotropically, the morphology can be isotropically oriented, and it can be expected to achieve isotropically high stress. However, in the case of press in the solid phase, a normal press machine does not provide sufficient pressure, and sufficient press cannot be achieved. Therefore, in this study, a large press machine instead of a normal press machine is used to apply a high- pressure press. This high-pressure press aims to improve the performance of PP. The purpose is also to clarify the relationship between crystal high-order structure and mechanical properties by investigating the crystal high-order structure in detail. The S-S curve obtained from the tensile test of pressed PP has increased the stress at break compared with the S-S curve of as-mold PP. That is, we succeeded in achieving high strength. In order to investigate the reason for this high strength, the morphology was clarified by SAXS and WAXS scattering methods and TEM and OM microscopy methods. As a result, the shear band where deformation is concentrated appeared by press. In other words, the deformation occurs inhomogeneously. This structure causes tension in the molecular chains, leading to increased strength.

Oral (S07-540, Time: Friday 11:00, Room: Sulzer Chemtech)

Effect of biaxial orientation on polyethylene microstructure

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Microcrystalline structure developed during transformation processes influences final properties of polymeric parts. Indeed, many polymer processes involve shear and elongational forces which impart orientation in polymer chains, the degree of orientation enhances final proprieties such as mechanical strength and barrier properties. Moreover, many industrial technologies, such as film blowing, double bubble processing or tentering, involve biaxial orientation. The relationship between orientation and mechanical performances is rather complex, it is even more complex in case of biaxial orientation.

In this work the correlation between biaxial orientation and mechanical performance will be analyzed for commercial grades of polyethylene. A new device was designed and assembled on monoaxial testing machines. Deformation during biaxial test is recorded by using Digital Image Correlation (DIC), which consists in a non-contact method based on images captured during the stretching. Validation tests of device and the DIC method were performed through comparison with both monoaxial and biaxial tests. Machine testing reveals that the results of DIC measurements are consistent with the deformation measured during monoaxial stretching tests.

Biaxial stretching tests were performed under several conditions, namely varying the stretching temperature (always below the melting point), biaxial draw ratio (ratio between the elongation in the two directions), and the strain rate.

Mechanical properties of biaxial stretched polyethylene were evaluated and correlated to morphology and orientation degree.

Acknowledgement:
The work of Francesca Gentile forms part of the research programme of DPI, project #847

Oral (S07-551, Time: Friday 11:20, Room: Sulzer Chemtech)

Development of toughening elastomeric nanofibril by in-situ fibrillation and post-crosslinking in SAN

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The polymer responses to imposed stress categorize from brittle to ductile behaviors, and rubber inclusion has been introduced as one of the most prominent techniques to enhance the toughening of brittle polymers. Although the inclusion of rubber enhances the polymer toughness, it is consequently along with a sharp reduction of polymer stiffness. Therefore, the balance between stiffness and toughness is a major issue that will be studied in this paper. This balance is not only determined by the rubber concentration but also can be examined by the rubber morphological structures. Literature has revealed the effectiveness of spherically distributed rubber on the matrix toughness mainly when the rubber concentration exceeds 20 wt. % where it is high enough to degrade the matrix stiffness. There is a very promising idea to replace the spherical domain with a nanofibril structure to create an isotropic structure by which a higher interfacial area is introduced between the rubber and the matrix due to the presence of a high aspect ratio nanofibril. Therefore, it is aimed to minimize the rubber content and the adverse effect of rubber on the matrix stiffness. Other than the challenges in the development of nanofibril rubber, there are some troubles in perseverant of developed nanofibril rubber. Therefore, the final balance between toughness and stiffness is determined by two vital keys: the development of nanofibril rubber with a high aspect ratio and maintaining of rubber nanofibril. In this paper, the development of various nanofibril rubbers such as ethylene-propylene-diene copolymer (EPDM), Maleic anhydride modified EPDM (MA-EPDM), and thermoplastic polyurethane (TPU), in the styrene-acrylonitrile copolymer (SAN) matrix by in situ fibrillation is discussed. And, then various crosslinking methods such as gamma radiation and moisture crosslinking as two triumphant candidates for post-crosslinking of nanofibril rubber will be discussed.

Oral (S07-588, Time: Friday 11:40, Room: Sulzer Chemtech)

Characterization of process-induced morphology of injection molded PLA specimens and its influence on mechanical properties at multiple scales

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Polylactic acid (PLA) is one of the most promising biopolymers to replace conventional polymers. It is already used for various applications, e.g. in the form of mulch films in agriculture or in the medi-cal industry for implants. The properties of PLA are well known and well studied. PLA crystallizes very slowly, resulting in an unusual inhomogeneous morphology compared to polyolefins. There-fore, the relationship between the properties and processing parameters of PLA has not yet been sufficiently clarified. To expand the scope, especially in terms of long-term applications and recycla-ble products, a deep understanding of how the structure and resulting properties can be controlled by processing parameters is required. Therefore, we investigated the morphological and mechanical properties of injection molded PLA tensile bars at different length scales as a function of processing parameters e.g. melt temperature, mold temperature and cooling time. In this work, we present the process-controlled formation of crystalline structures, their inhomogeneous distribution within the tensile bars and their influence on the global and local mechanical properties. By means of high resolution X-ray investigations and AFM-measurements, the degree of crystallization and the different crystal modifications as well as the size and distribution of crystalline structures were investigated. Micromechanical tests were performed on thin slices prepared from different microstructural domains. The results show that the degree of crystallinity does not correlate with the integral mechanical properties. Only detailed investigations of areas with different morphological properties could explain the correlation to mechanical properties.

Oral (S07-621, Time: Friday 12:00, Room: Sulzer Chemtech)

Effect of biaxial stretching on the structure-properties relationship of multi-layered PE/EVOH films

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In the last few years, nanoconfinement has been found an extremely effective way to tailor and tune the bulk properties of a polymer film. Among the different ways to achieve nanoconfinement, the nanolayer co-extrusion process is one of the most effective, due to its versatility and efficiency. This process, based on forced assembly technology, enables the production of polymer films composed of up to thousands of alternating layers of two polymers with individual layer thicknesses ranging from micro- to nanometers [1]. As biaxial stretching is known to improve toughness, transparency, and gas barrier properties, it has been shown to be of great interest to combine the effect of nano-layering and biaxial stretching [2]. In the current study, polyethylene (PE)/ethylene-vinyl alcohol copolymer (EVOH) (80/20 v/v) multilayer films were successfully coextruded, with various numbers of layers and different individual layer thicknesses. These films were further simultaneously biaxially stretched with draw ratios of 3X3, 4X4, and 5X5 leading to individual thicknesses down to 30 nm. SEM and AFM analysis has been performed to assess the integrity of the layers before and after biaxial stretching. Furthermore, a complete characterization in terms of thermal, mechanical, viscoelastic, and permeability properties was done to observe and understand the changes induced by the two processes. References [1] Ponting, M.; Hiltner, A.; Baer, E., Polymer Nanostructured by Forced Assembly: Process, Structure, and Properties. Macromol. Symp. 2010, 294, 19-32. [2] Lin, Y., Hiltner, A. & Baer, E. Nanolayer enhancement of biaxially oriented polypropylene film for increased gas barrier. Polymer, 2010, 51, 5807–5814.

Oral (S07-635, Time: Friday 14:10, Room: Sulzer Chemtech)

Shear Anisotropy-Driven Sectorization Imaging of Macromolecular Single Crystals

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Polymer single crystals have shown striking promise in modern applications such as flexible organic field-effect transistors (OFETs) drug delivery biosensors and nanoparticle assembly/patterning in the optical and electronic microdevices. However, there are some aspects of polymer single crystals which are not well studied and understood. One surprising challenge towards the better understanding of the microstructure of polymer single crystals is the difficulty of producing clear images of the sectorization in the single crystals which can provide a better understanding of macromolecular chain architecture and translational symmetry of single crystals. Friction force microscopy (FFM) is an atomic force microscope (AFM) based surface analysis technique that allows for precise friction force measurements of multiscale sliding contacts (i.e., atomic-, nano- and micro-scale). Herein, we demonstrate that an unconventional FFM mode (scan angle ≠ 90°) known as transverse shear microscopy (TSM) allows for unveiling the sectorization in polymer single crystals. The striking ability of TSM in high contrast imaging of the sectorization in polymer single crystals can greatly enhance the non-destructive analysis of single crystal microstructure. For polymer single crystals with a thickness of around 100 Å, we image the sectorization associated with crystal growth along lateral faces by TSM. Meanwhile, by using a linear elasticity model and analyzing the elastic deformation, we demonstrate that TSM imaging contrast originates from the elastic anisotropy of chain folding in various sectors of polymer single crystals. The findings of this study show the intriguing potential of TSM as a precise imaging technique for microstructure analysis of macromolecular materials such as polymers.

Oral (S07-653, Time: Friday 14:30, Room: Sulzer Chemtech)

Transparent biaxially stretched films from polyolefin nanocomposites

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This paper reports on the combined effects of adding nanoparticles and processing, on light transmittance and haze of biaxially oriented polyolefin films used as barrier films in food packaging. Recent results from our lab show that addition of 5 wt% nanoclay can improve the barrier to water vapor by a factor of 2.5. This paper is a study of how processing conditions used in preparing the nanocomposite films affect the morphology and degree of transparency in such films. The degree of transparency or light transmittance in semicrystalline polyolefins depends on crystallinity and the dimensions of crystalline lamellae, which is behind the practice of adding nucleating agents as clarifiers. Biaxially stretched polypropylene films were prepared from polypropylene nanocomposites with 2 to 10 wt. percent of organically modified layered aluminosilicates, also treated with an aminosilane as a coupling agent, and an equal proportion of maleated ethylene-propylene copolymer. The biaxial stretching was carried out by (a) simultaneous stretching to stretch ratios of 7.5 x 7.5 and (b) by sequential stretching to 5 x 9 at 155C and a Hencky stretch rate of 2 s-1. Both wide angle X-Ray scattering (WAXS) and small angle X-Ray scattering (SAXS) were carried out on the film specimens to infer the lamellar dimensions and the anisotropy in the films. The transmittance and haze were recorded with an illumination diffusion instrument (ID-E). The aspect ratio of lamellar dimension along D (040) to the lamellar thickness in extruded sheet before biaxial stretching was lowered from 4 to 2.8 progressively with addition of nanolayers; while after biaxial stretching, this aspect ratio was much lower and ranged from 0.8 to 0.7 progressively in the presence of added nanolayers. Thus, the aspect ratio of crystalline lamellae was altered drastically by biaxial stretching and to a greater extent in the nanocomposites. The transparency was higher after simultaneous equi-biaxial stretching than after sequential stretching.

Oral (S07-703, Time: Friday 14:50, Room: Sulzer Chemtech)

Chemical Treatment and Relatively Low-Pressure Effects on the Nucleation of Poly (L-lactide)/Glass Fiber Composites

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Both fiber-induced crystallization and pressure-induced crystallization affect the surface adhesion between fibers and the polymer matrix [1]. These phenomena involve couplings between physics, chemistry and mechanics that require understandings. Our approach uses the glass as 'models' fibers and the Poly (L-lactide) (PLA) as bio-sourced matrix. Various chemically treated glass fibers (GFs) are investigated, i.e. highly hydroxylated glass fibers (HCl GF), hydrophilic glass fibers (APTES GF), and hydrophobic glass fibers (TMPS GF). A classification of the nucleation efficiency of the various chemically treated GFs toward the PLA matrix is proposed. Based on the highest hydrophilic force, the chemically treated APTES GF was selected and examined under various relatively low-pressure levels (up to 12 bar). Considering individual chemical effect and then combining it with the pressure effect has made it possible to gradually design the role of chemistry and physico-mechanics on the nucleation in bio-based systems. [1] Wang B., Wen T., Zhang X., Tercjak A., Dong X., Müller A.J., Wang D., Cavallo D. Doi: 10.1021/acs.macromol.9b01078 ANR Carnot MINES Paris PSL, project entitled "RecCombios: Recyclabilité de Composites Biosourcés - Recyclability of Biobased Composites") 2022.

Poster (S07-115, Time: Thursday 17:00, Room: Foyer)

Melt Memory Effect at Extrusion and Drawing Processes for Thermoplastic Polyester Elastomer

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A small amount of unmolten crystals and/or highly ordered areas in the molten state may affect the processability as well as the structure in the solid state of a product, which is called "melt memory effect". In this study, the impact of melt memory effect on the structure development at extrusion by a capillary rheometer was investigated using a commercially available thermoplastic polyester elastomer (TPEE). Prior to the extrusion, we successfully found the appropriate temperature to have residual crystals without losing flowability by precise measurements of linear viscoelastic properties. When the sample was extruded at this temperature, the extrudate's swell ratio increased. Furthermore, the drawdown force, i.e., force required to draw an extruded strand, was enhanced. This was attributed to the network structure comprising residual crystals. Moreover, it should be noted that the drawn strands had anomalous oriented structure; i.e., the amorphous chains were oriented parallel to the drawing direction, whereas the crystalline chains were oriented perpendicular. The perpendicular orientation of crystalline chains is originated from the parallel orientation of lamellae grown from the residual crystals. The cross orientation affected the mechanical properties in the solid state.

Poster (S07-182, Time: Thursday 17:00, Room: Foyer)

Thermal characterization of modified linear and long-chain branched polypropylene blends

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Crystalline structure of linear isotactic polypropylene (L-PP) has been a subject of research for many years and is quite well described at this point. Introduction of long-chain branches (LCB) into polypropylene has proven to influence the final structure in very significant way. The high self-nucleating capabilities of branches in LCB-PP introduce enormous number of nuclei during growth and due to this effect, blends of LCB-PP and linear iPP were produced to study nucleating capabilities of LCB-PP. This study extends the scope of work done by Tian et al. (Tian et al., 2007) and Zhao et al. (Zhao et al., 2012, 2013) with analysis of the nucleation effect of LCB-PP in polypropylene blends and LCB-PP's interaction with commercially used nucleating agents for iPP in non-isothermal conditions and creates basis for evolution of crystalline structure modeling during conventional processing methods, such as injection molding and extrusion. Taking in account different crystallization kinetics for α and β phase as well as the presence of LCB, a modified Nakamura equation was implemented to describe the crystallization and obtain the modelling parameters for further industrial use.

Poster (S07-237, Time: Thursday 17:00, Room: Foyer)

Process related properties of injection molded isotactic polypropylene (iPP)

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During processing, in particular in injection molding, semicrystalline thermoplastics are exposed to high thermo-mechanical stresses. As a result of these stresses, the distribution of crystalline structures is usually inhomogeneous, both across the parts cross-section and along the flow length of the part, thus influencing the application-specific properties (optics, mechanics). In order to identify, evaluate and optimize the interrelationships of process-structure-property relationships across different scales, it is of great importance to determine the interactions between the process conditions prevailing in the part and the material-specific crystallization kinetics by using and further developing suitable thermal and rheological measurement methods. This research is part of the DPI program, project #816. In this project, the influence of process-relevant parameters (injection speed, melt temperature, mold temperature) on the morphological part characteristics and resulting mechanical properties of iPP was investigated. The thermal and rheological boundary conditions prevailing in the part during processing were varied and determined by simulation. For a better understanding of the interaction between the process conditions and structure formation, the crystallization kinetics of the material under process-relevant thermal [1] and rheological parameters (cooling rate, shear rate) were determined and compared with the morphology within the part. Furthermore, the influence of the process-induced morphology on the mechanical properties was investigated in the macro and micro scale [2]. The results are suitable for evaluating and complementing models in process simulation and for developing new and further correlative methods for simultaneous data acquisition of material properties and structural changes in mechanical testing. [1] Spoerer Y., Boldt R., Androsch R., Kuehnert I. Crystals, 2021. 11(9): p. 1138. [2] Spoerer Y, Boldt R., Fischer M., Kuehnert I. Conference proceeding DGM - Werkstoffprüfung 2022, Dresden.

Poster (S07-337, Time: Thursday 17:00, Room: Foyer)

Characterization of Hemp-Based Panels with UF and PF resins

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The continuous consumption of natural resources, which reduces the natural environment's sustainability due to deforestation, has led the scientific community to develop substitute products to replace natural wood. The use of agricultural crop residues as alternative materials is gaining. In order to promote the circular economy and reduce raw material procurement costs, industries have turned to those materials. Hemp is a great wood substitute due to its lightweight, hydrophobic properties, exceptional long-term mechanical stability, and its low carbon footprint. Phenol-Formaldehyde (PF) and Urea-Formaldehyde (UF) are the most commonly used wood adhesives in the construction industry. However, their petrochemical origin raises concerns about people's and the environment's safety. In this work, the structural and thermal properties of UF and PF resins, hemp, and hemp-based panels will be examined. The main goal of this study is not only to replace natural wood but also to use eco-friendlier resins by replacing part of PF with soy protein, a work in progress. So, the structural and morphological properties of the materials were studied by Fourier Transform Infrared spectroscopy (FTIR), X-ray diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS) and Scanning Electron Microscopy (SEM). Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC) were used to study the thermal properties, including the thermal stability and thermal degradation of the resins, the neat hemp, and the hemp-based panels. Acknowledgments This research has been co-financed by the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH-CREATE-INNOVATE (project code: T2EDK – 00008).

Poster (S07-382, Time: Thursday 17:00, Room: Foyer)

Hierarchal ultra-lightweight cryogels from nanochitin-jammed bicontinuous emulsions

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Bicontinuous emulsions are non-equilibrium colloidal systems consisting of two immiscible liquids that form co-continuous, tortuous channels. In this study, chitin nanocrystals (ChNC) were used to kinetically arrest a bicontinuous structure during thermal-induced spinodal decomposition of the water/2,6-lutidine system via intra-phase particle jamming. We showcase a remarkably low particle concentration for robust intra-phase jamming using needle-like ChNC particles, which can be explained by their high aspect ratio, intrinsic stiffness, and inter-particle interaction. Moreover, following lyophilization of the devised ChNC-jammed bicontinuous emulsions, hierarchal ultra-lightweight chitin cryogels with open channel and dual-sized porous structures are formed. No shrinkage was observed upon drying the bicontinuous emulsions, verifying the impact of microstructural features on the robustness of ultra-low solid content aerogels. Our results open new avenues toward ultra-low solid content gel emulsions with high non-equilibrium morphologies stabilized by bio-based nanoparticles, broadening the horizon of application of multiphase yield stress fluids.

Poster (S07-659, Time: Thursday 17:00, Room: Foyer)

Detection and analysis of microvoids in optical injection molding parts

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Optical parts in injection molding must comply with ever-increasing demands for optical performance, particularly in applications for electronics and medicine. Even small inclusions of air or chemical residue can sharply reduce the overall quality of these optical parts. Recent innovations in Optical Coherence Tomography (OCT), a technology frequently used in optometry and ophthalmology, can give deep insights into the creation and avoidance of these errors, often just called microvoids. These discoveries could ultimately lead to process optimization strategies for better performing optical parts. In this work, OCT images of different optical parts were compared in a 3D-structural analysis to reveal differences in occurrence, size and distribution of microvoids. In a first trial, typical optical parts like lenses, light guides and larger optics were measured in the OCT. A consistency of microvoid occurrence could be found regardless of part shape or history. Next, further trials sought strategies in process control as well as material treatment to reduce microvoid occurrence for a sample lens part. A DOI with relevant and distinct parameters was made to quantify microvoid occurrence and understand the underlying mechanism of air encapsulation and resulting microvoid occurrence. This work enables the identification of three distinct parameters to reduce microvoid formation in optical parts and reduce the size of the occurring microvoids.

Keynote (S08-488, Time: Wednesday 10:15, Room: St. Gallen)

Effects of adding PMMA to PA6/carbon nanotube composites on the morphology, rheology, and thermoelectric properties of the blends

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The study investigates how the blend ratio of immiscible blends based on PA6/PMMA affects the morphology and properties of unfilled blends and blends filled with 3 wt% singlewalled carbon nanotubes (SWCNTs). Compositions between 50 wt% and 100 wt% PA6 were prepared by small-scale melt mixing and investigated by scanning electron microscopy (SEM) and optical microscopy (OM). The morphology types include matrix-disperse particle morphologies at low contents and co-continuous morphologies at higher PMMA contents, with SWCNTs preferentially localized in PA6. For the blends containing 50 wt% PMMA with different SWCNT contents, melt rheological studies were performed. In line with the greatly increased viscosity after filling PA6 with SWCNTs, the blends with SWCNTs also exhibit significantly higher viscosity values than the unfilled blends. The viscosity curves of PA6/PMMA =50/50 wt% blends with SWCNTs match or exceed the curve of PA6 filled with 3 wt% SWCNTs from 1.5 wt% SWCNT addition, which corresponds to 3 wt% SWCNTs in the PA6 component. This indicates the formation of a co-continuous morphology type, which can be clearly seen in SEM and even OM images. This type of morphology is retained even after the extruded material is compression moulded into sheets. In terms of thermoelectric properties, all composites and blends exhibit n-type behaviour with negative Seebeck coefficients. By varying the blend composition, an increase in the absolute Seebeck coefficient, power factor (PF), and figure of merit (ZT) was achieved compared to the PA6 composite, mainly due to the selective localization and accumulation of SWCNTs in PA6 at constant SWCNT loading. The maximum PFs achieved were 0.13 microW/m·K2 for PA6/PMMA/SWCNT 60/40/3 wt% compared to 0.09 microW/m·K2 for PA6/3 wt% SWCNT, an increase to 144%.

Keynote (S08-496, Time: Wednesday 13:45, Room: St. Gallen)

Modified halloysite nanotubes (HNT) used as part of a flame retardant system for a LLDPE/EVA blend

Taguet Aurélie[1], Viretto Amandine[1], Otazaghine Belkacem[1], Sonnier Rodolphe[1], Jasinski Euphrasie[2], Bounor-Legare Véronique[2], Beyou Emmanuel[2], Auvray Thierry[3], Gyppaz Franck[3]

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Cable industry is continuously improving the mechanical, electrical and fire properties of their sheaths in order to keep competitive. For building applications, flexibility is a big issue while keeping good fire properties. The objective of the present project, funded by the French National Research Agency (ANR), aims at dispersing modified HNT into LLDPE/EVA blend to improve mechanical and ageing properties with appropriate fire behavior. HNT, a tubular aluminosilicate clay, has gain much attention thanks to its numerous interesting properties. Especially, its tubular shape allows to encapsulate different molecules into the lumen (internal diameter of 10-20 nm) for several applications: anticorrosion, antimicrobial, drugs, flame-retardant, microcrack self-healing. Especially, lumen can contain encapsulated molecules to be used as a flame retardant system (in combination with other additives) in polymer blends. Hence, the objectives of the project are: (1) to encapsulate molecules into the lumen that can play the role of a flame retardant system for LLDPE/EVA, (2) to functionalize the inner and/or outer surface of the HNT to selectively disperse it either in LLDPE or in EVA, in order to see the influence of the selective dispersion in mechanical and fire properties. This presentation will firstly explains how native HNT thermally degrade EVA copolymers during the melt mixing process. The second objective of the presentation is to explain how 3-aminopropyltriethoxysilane modified halloysite nanotubes are selectively dispersed into the LLDPE/EVA blend. Finally, the mechanical and fire properties of different LLDPE/EVA/HNT blend nanocomposites are compared.

Oral (S08-139, Time: Wednesday 10:40, Room: St. Gallen)

Investigation of reactive and non-reactive compatibilizer effects on mechanical, thermal and rheological properties of thermoplastic starch and PLA blend

Farshbaf Taghinezhad Soheil[1], Mansourieh Mohammadreza[1], Fitzpatrick Daniel P.[1], Hesabi Mohammadnabi[1], Major Ian[1], Pezzoli Romina[1]

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Thermoplastic starch (TPS) is one of the main biobased and biodegradable candidates to substitute general-purpose oil-based polymers used in the packaging industry. TPS shows poor mechanical properties, mainly low elongation at break and toughness, one of the main barriers to its implementation in the area. Blending TPS with other polymers, such as polylactic acid (PLA), is a common strategy to improve their performance however, due to immiscibility and low physical interaction between the phases, this route is not sufficient. In this research, a commercial grade of TPS was melt mixed with polylactic acid (PLA) using different non-reactive and reactive compatibilization systems to increase the interfacial interaction of the blend and therefore, improve its mechanical performance. Poly(ethylene-co-methyl acrylate-co-glycidyl methacrylate) (EMA-GMA) was chosen as a reactive compatibilizer. The glycidyl group in EMA-GMA can react with ester and hydroxyl groups in PLA and TPS, respectively. Polylactic acid (PLA) and polybutylene adipate terephthalate (PBAT) were melt mixed with maleic anhydride (MA) and dicumyl peroxide (DCP) to prepare PLA-g-MA and PBAT-g-MA. Then, PLA-g-MA, PBAT and PBAT-g-MA were used as compatibilizers in TPS/PLA blend. Although all the compatibilizers affected the mechanical properties of the TPS/PLA blend, the TPS/PLA/PBAT-g-MA (50/50/4) system showed the greatest improvement, increasing the tensile elongation at break from 6% to 30% and enhancing the ductile behaviour of the blend. The compatibilized TPS/PLA blends also showed enhancement in thermogravimetric analysis (TGA), more pronounced for samples containing EMA-GMA and PBAT-g-MA. Besides, the effects of compatibilizers on TPS/PLA blends were investigated by differential scanning calorimetry (DSC) and rheological tests, providing valuable insights into their thermal and melt behaviours.

Oral (S08-202, Time: Wednesday 11:00, Room: St. Gallen)

The effect of adding high density polyethylene to a polypropylene matrix to thermo-shear degradation in the melt phase.

Haenen Sam[1], Desplentere Frederik[1], Buffel Bart[1], Ginzburg Anton[1], Cardinaels Ruth[2]

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Degradation of isotactic polypropylene(PP) and High-density polyethylene(HDPE) can occur in an accelerated way during melt processing. Hence a study of the thermo-oxidative and thermo-mechanical stability is performed on a corotating twin-screw extruder Leistritz ZSE 18. First, the origin and effects of degradation are studied. The effect of shear stresses and residence time during compounding on the chemical composition, rheological behavior and mechanical performance is examined. In the next phase of the research, the thermo-mechanical stability of PP is enhanced by adding HDPE to the PP matrix. Small amplitude oscillatory shear measurements showed a significant decrease of PP's viscosity (-40%) after 1 compounding cycle. This is in contrast to HDPE where no differences were observed. The viscosity loss is more visible in the terminal regime indicating a higher impact on the long chain molecules due to β-scission reactions. Fourier transform infrared measurements did not show any presence of secondary oxidation products. Thus, thermo-shear is expect to play a dominant role in PP's degradation mechanisms compared with peroxy radicals. The addition of a relatively small amount HDPE (<20 wt%) to the PP matrix could enhance thermo-shear stability without decreasing mechanical performance. Adding 10 wt% HDPE to PP recovered almost completely the viscosity loss originating from processing. However, HDPE and PP remain two separated phases. Due to low interfacial tension and well-dispersed uniform particle size, no negative influence is observed in the mechanical properties. These results provide important insights into the thermo-shear stability of PP and HDPE during melt processing. Furthermore, the addition of a relatively small amount HDPE to the PP matrix can counteract the thermo-mechanical degradation mechanisms that affect the properties of PP. In the end, these findings can create more opportunities to use PP recyclates in different types of applications .

Oral (S08-319, Time: Wednesday 11:20, Room: St. Gallen)

Effects of SEBS-g-MAH addition on the vibration damping and mechanical properties of MABS/VDT blend

Sujon Mohammad Abu Shaid[1], Merinoa Iñigo Trueba[1], Quaade Thomas Sørensen[2], Andriollo Tito[3], Islam Aminul[1]

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This study explored the influence of Maleic Anhydride-grafted Styrene Ethylene Butylene Styrene (SEBS-g-MAH) compatibilizer on the development of a novel kind of polymer blend to increase the vibration damping property of Methyl Methacrylate Acrylonitrile Butadiene Styrene (MABS) by compounding with a Styrene-based engineered elastomer (tradename VDT). Most of the research related to polymer blends has been focused on enhancing the material's stiffness, thermal or electrical conductivity by incorporating stiffer materials like glass fiber, graphene, CNT and so on. However, a limited amount of study has been done to investigate the possibility of increasing the damping property of the polymer by the use of melt compounding. Thus, a multiphase polymer blend was formulated by melt mixing in a twin screw extruder with three different weight ratios (10, 20, and 30 wt%) of VDT to enhance vibration damping with a minimum tradeoff in stiffness property. To improve the compatibility between MABS/VDT, SEBS-g-MAH was used with three different weight percentages (2, 4, and 6 wt%) and the effect of the compatibilizer was compared without it as well. The compatibility and effectiveness of the compatibilizer were investigated by studying their microstructure, tensile, dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC), nuclear magnetic resonance (NMR), light optical microscopy (LOM), and scanning electron microscopy (SEM) analysis and the samples were prepared by injection molding. The damping performance has been shown to improve as the weight percent of VDT in the blends increases. It was also found that the addition of 4 wt % of SEBS-g-MAH had the highest effect on the improvement of the damping performance and tensile strength compared to the additions of 2 wt % and 6 wt % of the compatibilizer.

Oral (S08-451, Time: Wednesday 11:40, Room: St. Gallen)

Rheological and Electrical Properties of amorphous PLA through generation of fibre-like oriented crystal network and incorporation of multiwalled carbon nanotubes

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Blend nanocomposites of amorphous polylactide (aPLA; high and low molecular weight)/ semicrystalline polylactide (scPLA; high molecular weight with high crystallinity)-multiwalled carbon nanotubes (MWCNTs) were prepared by a twin-screw extruder below the melting point of scPLA. The maximum weight percent of CNTs in the blend was 0.9 wt%. The extrudates were either pelletized immediately or after drawing them at a drawing ratio of around 10. In the neat blend (aPLA/scPLA), While the rheological properties of the undrawn samples were improved, those of the drawn samples were much more significantly increased, as seen by the small amplitude oscillatory shear rheological behaviour of the samples. scPLA/MWCNTs masterbatch generated a stiffer reinforcing phase and expanded the processing window. This is mainly because more crystallites could be presented as MWCNTs induced the crystallinity of scPLA. What is more, the electrical conductivity of these blend nanocomposites was investigated, and the results showed that blending scPLA/MWCNTs below the melting point to keep most of the crystallites, makes an obstacle for the electrical path by encapsulating of MWCNTs in the crystallites of scPLA (electrical conductivity of ~10^-13 Ω/cm for aPLA/ scPLA-3 wt% MWCNTs). Increasing the temperature during compression molding (higher the melting point of scPLA; to prepare the sheets for electrical conductivity measurements) seemed to remove that obstacle in the low molecular weight aPLA (electrical conductivity of ~10^-7 Ω/cm for aPLA/ scPLA-3 wt% MWCNTs). In this case, the electrical conductivity is comparable with neat nanocomposites of aPLA/1 wt% CNT with the electrical conductivity of ~10^-6 Ω/cm.

Oral (S08-574, Time: Wednesday 12:00, Room: St. Gallen)

Formation of interfacial stereocomplex crystal by reactive blending: New strategy to high performance immscible polymer blends

Li Yongjin[1], Wang Hengti[1]

[1]Hangzhou Normal University, Zhejiang, China

Poly(L-lacid) (PLA), the most appealing representative of bio-based and biodegradable polymer, has received increasing attentions owing to its advantages such as high modulus, superior transparency, and excellent biocompatibility. However, the widespread industrial applications of PLA have been severely restricted due to its inherent brittleness, inferior heat resistance, as well as low crystallization rate. The combination of polymer blending and stereocomplexation (SC) in PLA has been regarded as the most efficient and practical strategy to acquire PLA-based materials with high performance. Unfortunately, the present stereocomplexation researches are still confronted with the following issues: Firstly, the large content of PDLA required in SC strategy make the modification extremely expensive. Besides, the formation of SCs is generally carried out in the PLLA phase. Moreover, the broad feasibility of the approach mentioned above is neglected since previous researches mainly focus on the PLA-toughened system. In order to overcome the above-mentioned problems, a new concept termed as interfacial SC compatibilization (i-SC) is proposed though reactive processing of PLA-based blend in the presence of small amount of reactive PDLA-grafted compatibilizer in this work.

Oral (S08-578, Time: Wednesday 14:10, Room: St. Gallen)

Construction of quasi block copolymer (qBCP) induced by stereocomplexation of enantiomeric PLA-grafts and its application as compatibilizer for immiscible polymer blends

Wang Hengti[1], Li Yongjin[1]

[1]Hangzhou Normal University, Zhejiang, China

Self-assembly of block copolymers (BCPs) yields unparalleled opportunities to integrate exquisite characteristics for preparation of advanced nanotechnology materials. The high-yield manufacturing of BCP is extremely desired, yet rarely exploited. Herein, we displayed that stereocomplex crystals (sc) of poly lactide (PLA) enantiomers, acting as pseudo linkage between adjacent polymer chains, enable formation of quasi-block copolymer (qBCPs) by straightforward melt compounding. The refined morphology, which was resembled with micro-phase separation of classical BCP, can be facilely tuned by varying the length and ratio of enantiomeric PDLA/PLLA grafts. More interestingly, qBCPs demonstrated superiority in compatibilizing immiscible polymer blends, as compared with the classical compatibilizer via reactive blending. Our strategy represents a general method to construct non-covalently linked di-block copolymer via industrially relevant melt processing, which is quite challenging to achieve by existing synthetic technologies.

Oral (S08-581, Time: Wednesday 14:30, Room: St. Gallen)

Extensional Flow Effect on Dispersion in twin-screw extruder : DURABIO/PMMA Polymer Alloys

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Twin Screw Extruders (TSE) are widely used in the plastics industry to achieve continuous compounding and high productivity. During compounding, the screw elements of a TSE primarily generate shear flow. At viscosity ratios above 3.8, shear flow cannot break up droplets, but Grace's experiments showed that extensional flow is possible. It is also known that shear flow causes shear heating during kneading, resulting in pyrolysis of the polymer, while extensional flow does not contribute to heat generation. Therefore, a screw element with small holes was uniquely designed to generate an elongational flow. As materials, we selected bio-polycarbonate (DURABIO) and polymethylmethacrylate (PMMA). As a result of the study using an evaluation device of our own design, we were able to obtain an alloy with high transparency due to high pressure extensional flow. Mixing experiments with TSE were then conducted. The dispersion state was compared by changing the screw arrangement of the kneading type screw element and the extensional type screw element. As a result, it was found that the elongation-type screw element could lower the discharge resin temperature and reduce the dispersed phase size by 56% compared to the kneading-type screw element. Therefore, it was found that the elongation-type screw element is a method that enables dispersion while minimizing thermal degradation of the polymer.

Oral (S08-609, Time: Wednesday 14:50, Room: St. Gallen)

Tuning dielectric material properties of miscible thermoplastic blends for high frequency applications

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Electronic devices and its components have to deal with ever increasing radio frequencies, which in turn rises demands for certain properties of materials in use. Dielectric properties in particular have to be within certain thresholds to allow for circuit board layout and high frequency antenna designs suitable for short-range, low-latency and high-capacity wireless networks such as intended within the currently employed 5G cellular network standard. Here, thermoplastics render a viable alternative to resin-based circuit board substrate materials. However, we see a challenge caused by contrasting trends in thermo-mechanical stability vs. appropriate dielectric values of neat thermoplastic materials. Blending different thermoplastics can help to improve material service temperature while maintaining high frequency applicability. Using a well-established miscible blend system, PEEK/PEI, the author presents findings regarding the impact of blend composition on the aforementioned dielectric properties, i.e. relative permittivity and loss factor. To do so, blends covering the whole PEEK-PEI range are analyzed at 5 and 10 GHz utilizing split post dielectric resonators. Crystallization kinetics are drastically affected by the presence of amorphous and semi-crystalline blend partners, in turn leading to changes in the blend's processability. To deduce correlations between crystallinity and dielectric properties, blends are prepared via melt processing, with specimen subjected to varying temperature profiles. Additionally, high mineral particle filler contents are used match the coefficient of thermal expansion to that of metallic materials applied on the dielectric substrate material to form electrically conductive tracks. Investigations of those particles influencing crystallization behavior and subsequently the resulting compound dielectric properties are carried out by incorporating talc filler into the PEEK/PEI system.

Oral (S08-658, Time: Wednesday 15:10, Room: St. Gallen)

Oxygen Barrier Properties of PHA-Based Bio-blend Flexible Packaging Films Prepared by Blown Extrusion

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Today, the use of biopolymers in the flexible packaging industry has become very important in terms of sustainability and climate crisis. Biopolymer market is expected to grow from 2.08 million tons in 2020 to 7.59 million tons in 2026. Polyhydroxyalkanoate (PHA) is a family of bio-polyesters commonly accumulated by many bacteria, consisting of the following polyesters: poly-3-hydroxybutylate (PHB), poly-3-hydroxyvalerate (PHV), as well as their isomeric polymers and copolymer.

In this study, amorphous grade of PHA was blended for three ratios with both poly(lactic acid) (PLA) and polybutylene succinate (PBS) by using twin screw extrusion. Then, blown film extrusion studies were carried out to determine the optimum ratio for the neat PHA/PLA and PHA/PBS compounds. After the optimum ratios have been determined, the changes in the mechanical, barrier especially oxygen (OTR) and optical properties of bio-blends with the addition of 1 phr PLA chain extender for PHA/PLA, 1 phr Joncryl ADR 4468 plasticizer for PHA/PBS and both reinforcing sepiolite at 3 phr were investigated in particular for flexible packaging. The optimum ratios for both studies were determined according to the reference thickness polyethylene film specifications by characterization tests. The antimicrobial effect on the films were examined by adding functional graphene oxide to the option, which provides the most flexible packaging quality for both PHA/PLA and PHA/PBS blends.

Keynote (S09-030, Time: Tuesday 10:15, Room: Olma)

The effect of Photo-active Nanoparticles on Radiation Curing and Reinforcement of Acrylates

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Curing of acrylates by radiation is commonly used process resulting in brittle thermoset. As shown previously by the Authors, this shortcoming could be remedied by incorporation of nanoparticles (NPs). Surprisingly, it was found that fullerene-like NPs of WS2, which are semiconductors and dark in color, can be used as a photo initiators (PIs), enhancing the degree of conversion (DC) of acrylates when incorporated with conventional PIs. y. Exposure of the NPs to light of appropriate wavelength forms at the photo-active moist surface hydroxyl radicals (reduction) and H+ ions (oxidation) . These free radicals are highly reactive and can accelerate radical curing. Experimental results using acrylate resins photocured via 365 nm wavelength exhibited shortening in the curing time and increase of DC for all resins after adding the WS2 NPs, from 77% for the neat polymer up to 84% for the composite resins. The tensile toughness increased by 15%-30%, the impact strength by 70%-90% and 80% increase in the shear adhesion strength was obtained compared to neat systems. To understand the acceleration effect of the radical photo-curing reaction of acrylate, a series of EPR (electron paramagnetic resonance) measurements were performed to evaluate the free radical's formation of the photo-active WS2 without the addition of conventional PIs. First, inorganic nanotubes of WS2 formed radicals in ethanol upon white light illumination creating a signal of superoxide radical (O2.), which increased upon radiation time. Second, EPR measurments for neat conventional PI in acrylate and EPR measurements for combination of WS2 fullerenes and PI in acrylate showed a different mechanism via OH radicals of the PI and a superoxide mechanism of the NPs. Third, measurements were carried out for 4 compositions of acrylate with 0.3, 0.5, 0.75 and, 1 wt. % WS2 without PI. Samples were irradiated for 5 min under 365 nm wavelength. The DC obtained for nanocomposite acrylate without PI is high (up to 86%)

Keynote (S09-138, Time: Tuesday 13:45, Room: Olma)

A Novel Polymer Nanotechnology Approach to Food Packaging

Ray Suprakas Sinha[1], Bandyopadhyay Jayita[1], Scriba Manfred[1], Lekalakala Rakgoshi[1], Botlhoko Orebotse J[1], Mekoa Caroline[1], Motloung Mpho[1], Ojijo Vincent[1]

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Increased food insecurity has been a national point of concern due to the Covid-19 global pandemic, economic decline and climate change. Adding onto the list of concerns, is ineffective food packaging which compromises the quality of food. It is said, in South Africa, most losses and waste is estimated at 30% and occurs at the processing and packaging stage in the value chain. Using our advanced polymer formulation capability and our well-equipped Nanomaterials Industrial Development Facility, we were able to develop a nanotechnology-based solution which features a nanocomposite technology which will greatly benefit the industry. The technology is based on the incorporation of nano-minerals in polymers to deliver significantly improved barrier properties in a polymer film, which further improves the shelve life of food.

Keynote (S09-529, Time: Wednesday 10:15, Room: Olma)

Multifunctional Biomimetic Nanocoatings

Sun Luyi[1]

[1]University of Connecticut, Connecticut, USA

Large-scale biomimetic organic/inorganic hybrid nanocoatings with a nacre-like microstructure were prepared via a facile co-assembly process. Different from conventional polymer nanocomposites, such nanocoatings contain a high concentration of nanosheets, which can be well aligned along the substrate surface. Moreover, the nanosheets and polymer matrix can be chemically co-crosslinked. As a result, the nanocoatings exhibit exceptional mechanical properties (high stiffness and strength), barrier properties (to both oxygen and water vapor), and flame retardancy, but meanwhile they are highly transparent (maintaining more than 85% of their original transmittance to visible light). The nanocoatings can be applied to various substrates and regular or irregular surfaces (e.g., films as well as foams). Because of their excellent performance and high versatility, such nanocoatings are expected to find widespread application.

Keynote (S09-558, Time: Tuesday 17:00, Room: Olma)

Development of Nanocomposites with Boron Nitride Nanosheets for Thermal Transport and Related Applications

Sun Ya-Ping[1]

[1]Clemson University, South Carolina, USA

Hexagonal boron nitride (h-BN) and exfoliated nanosheets (BNNs) not only resemble their carbon counterparts graphite and graphene nanosheets in structural configurations and many excellent materials characteristics, especially the ultra-high thermal conductivity, but also offer other unique properties such as being electrically insulating, and the extreme chemical stability and oxidation resistance even at elevated temperatures. In fact, BNNs as a special class of 2-D nanomaterials have been widely pursued for technological applications that are beyond the reach of their carbon counterparts. Highlighted in this presentation are significant recent effects in our program, in reference to those in the relevant research field [1], on the development of more effective and efficient exfoliation techniques for high-quality BNNs, the understanding of their characteristic properties, and the use of BNNs in polymeric nanocomposites for thermally conductive yet electrically insulating materials and systems. Major challenges and opportunities for further advances are also discussed. [1] Meziani, M. J.; Sheriff, K.; Parajuli, P.; Priego, P.; Bhattacharya, S.; Rao, A. M.; Quimby, J. L.; Qiao, R.; Wang, P.; Hwu, S.-J.; Wang, Z.; Sun, Y.-P. "Advances in Studies of Boron Nitride Nanosheets and Nanocomposites for Thermal Transport and Related Applications" ChemPhysChem 2022, 23(1), e202100645. Featured on the journal cover (e202100869, January, 2022).

Oral (S09-016, Time: Tuesday 10:40, Room: Olma)

Simplistic hydrothermal synthesis approach for fabricating photoluminescent carbon dots and its potential application as an efficient sensor probe for toxic lead (II) ion detection

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The past decade has witnessed a variety of members of the carbon family along with exposure of carbon dots due to their magnificent properties in sensing, bioimaging, catalytic applications, biomedical fields, and so on. Herein, we report the simple hydrothermal method to fabricate photoluminescent doped carbon quantum dots for the detection of noxious lead (II) ions. Lead (II) ion is very venomous for both the environment and human health for which its detection is demanded area in the research field. The as-prepared carbon dots show excellent photostability, low toxicity and significant photoluminescence properties along with good water solubility. Along with these properties, carbon dots have a quantum yield of approximately 15%. In the practical field of application, these carbon dots have been used as sensing probes for the detection of lead (II) ions with a detection limit of 60 nM. The fluorescence intensity of carbon dots was remarkably quenched in the presence of the lead (II) ion selectively among all the tested metal ions. Furthermore, we have studied the Stern-Volmer relationship for lead (II) quenching along with the explanation of the probable quenching mechanism. Overall, this study mainly demonstrates the sensing application of doped carbon dots for the identification of heavy metal ions in an environmental sample.

Oral (S09-040, Time: Tuesday 11:00, Room: Olma)

In-situ assembled 3-dimensional (3-D) nanostructures of polyphenol tannic acid surface treated Ti3C2Tx and graphene nanoribbons (GnRs) for physiological sensing applications

Mohseni Taromsari Sara[1], Shi HaoTian Harvey[2], Salari Meysam[1], Eskandarian Ladan[1], Park Chul B.[1], Naguib Hani E.[1]

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[2]University of Western Ontario, London, Canada

Polymer-based electrospun strain sensors are increasingly receiving attention due to their network construction, tailorable design, and facile integration into wearable devices. However, post-electrospinning incorporation techniques lead to 2-dimensionally (2-D), non-homogeneous responsive networks accumulated at the two most exposed surface of the mats, which limit the working range and stability. In addition, conductive formulations of unitary material composition hinder the desired sensitivity. In this study, these drawbacks were addressed via a novel strategy consisting of nano-scale materials formulations and a micro-scale in-situ coating technique. Firstly, a novel in-situ, electrostatic attraction-assisted fabrication method consisting of coaxial electrospinning and simultaneous electrospraying was developed to in-situ decorate the cetryl ammonium bromide (CTAB)@styrene-butadiene-styrene (SBS) shell@core fibers with electronegative nanoparticles. Then, a nanohybrid composition of polyphenol tannic acid functionalized 2-D Ti3C2Tx MXene and 1-dimensional (1-D) graphene nanoribbons (GnRs) were formulated. This was to utilize Ti3C2Tx's metallic conductivity as well as GnR's facile orientation and bridging effect to contribute to the formation of a 3-D conductive network. Functionalization with polyphenol tannic acid increased the electronegativity of Ti3C2Tx and its interactions with GnR, which were verified by Fourier-transform infrared spectroscopy (FTIR). The formation of the 3-D in-situ assembled network firstly increased the specific surface area (SSA) of the fibers by 178% and 125% compared to neat SBS and conventionally coated SBS membranes, and secondly enhanced the conductive network formed at all XYZ directions (25.12 S/m, 25.61 S/m, and 10 S/m, respectively). The structure provided exceptionally high sensitivity (GF of 2090 at 550%), excellent dynamic stability (enduring 5000 cyclic test under 100% strain) an optimal applicable physiological sensing.

Oral (S09-067, Time: Tuesday 11:20, Room: Olma)

CuInS2-quantum dot filled films for the use in luminescent solar concentrators

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The current energy crisis and the climate change demands a big change in energy production. Polymer devices could help changing big surfaces of buildings into great energy distributors. Luminescent solar concentrators (LSC) absorb certain wavelength of the sunlight through luminescent particles which emit waves with higher wavelength. Those waves are partly trapped into an optical waveguide and transported to the boundary surfaces. At those boundary surfaces there are photovoltaic cells which convert the concentrated light into useable electric energy. The shift from low wavelength (UV) to higher wavelength (could be in the visible light or in infrared territory) the so called stokes shift normally causes colored devices. To convert this technology into windows as very big parts of houses especially skyscrapers there is a need of thin and transparent devices. In this work a thin polymer film with transparency around 80 % is developed. CuInS2 (CIS) quantum dots were used as luminescent nanoparticles inside polyvinyl butyral (PVB) with concentrations of 0.5 wt.-%, 1 wt.-% and 2 wt.-%. To get a well distribution those nanoparticles were solved in ethylene(vinyl-acetate)-wax (EVA) in a first step (concentration of 20 wt.-% QDs). Afterwards this mixture was compounded into the PVB, and a film was produced. After applying these quantum dot filled film on glass sheets results show that the principle works, and a concentrated radiation can be measured at the boundary surfaces.

Oral (S09-100, Time: Tuesday 11:40, Room: Olma)

An Experimental Study on the Geometrical Effects of Nanomaterials on the Conductive Network Formation and Dielectric Properties in Microcellular Polymer Nanocomposites

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The current study provides insights into how modifying the geometry of fillers in nanoscale would result in a significant effect on electrical properties of the polymer nanocomposites. In this regard, graphene nanoribbon (GNR) was synthesized by unzipping multi-walled carbon nanotubes (MWCNTs) and characterized by means of transmission electron microscopy (TEM), X-ray diffraction (XRD) and Raman spectroscopies. The electrical conduction network formation of GNR/CNT within a polyvinylidene fluoride (PVDF) matrix as well as broadband dielectric permittivity of the nanocomposites were investigated using a dielectric analyzer. The experimental results reveal that the conversion of MWCNTs with a one-dimensional geometry to the two-dimensional GNRs has a significant impact on electrical conductivity of the nanocomposites in terms of the electrical percolation threshold and the maximum achievable electrical conductivity. These effects are attributed to the induced significantly greater contact areas along with larger number of filler-filler junctions. Accordingly, the GNR-based nanocomposites showed ~40% decrease in the through-plane electrical percolation threshold with over 3 orders of magnitude enhancement in electrical conductivity. Also, the findings show that the conductivities of MWCNT-based, and GNR-based composites are significantly increased by 230 times and 121 times, respectively, through microcellular foaming. The mechanisms of conductivity enhancement in the microcellular composites were thoroughly clarified. Furthermore, significant improvement in the real permittivity from 3.5 in the case of MWCNT-based composites to 88.3 in GNR-based samples was observed at the nanofiller loading of 2.5 vol%. Such enhancements would facilitate the development of functional polymer nanocomposites for the target applications, while minimizing the required nanofiller loading.

Oral (S09-168, Time: Tuesday 12:00, Room: Olma)

Ionic liquids as n-dopants in polymer/SWCNT composites for thermoelectric applications

Krause Beate[1], Voigt Oliver[1], Konidakis Ioannis[2], Stratakis Emmanuel[2], Pötschke Petra[1]

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In solving global energy problems, thermoelectric (TE) solutions are playing an increasingly important role. Among polymers, intrinsically electrically conductive polymers are predominantly reported, however, also conductive polymer composites (CPCs) consisting of an insulating polymer matrix and percolated electrically conductive fillers are also increasingly studied. Here TE behaviour of melt-mixed polypropylene (PP), polyetheretherketone (PEEK) and polycarbonate (PC) based nanocomposites with singlewalled carbon nanotubes (CNTs) and different kinds of ionic liquids (ILs) were studied. It was found that n-type polymer composites can be prepared from commercial p-type CNTs with relatively high Seebeck coefficients (S) when ILs are added. For PP as matrix, the highest S-values achieved in this study were +49.3 µV/K (PP/2% CNT) for p-type composites and -27.6 µV/K (PP/2% CNT+4% AMIM Cl) for n-type composites. For both PC and PEEK composites, the IL THTDP Cl leads to the highest negative S-value. In case of PEEK, the IL addition can change the S-value from 61.3 µV/K (PEEK/0.75% CNT) to -37.1 µV/K (PEEK/0.75% CNT+3% THTDP). For the PC/0.5% CNT composite, the S-value switched from 38.2 µV/K to -34.2 µV/K when 3% THTDP was added. Generally, the type of IL was decisive whether p- or n-type thermoelectric behaviour was achieved. After IL addition, a higher volume conductivity could be reached. Furthermore, ultra-fast laser time-resolved transient absorption spectroscopy (TAS) was employed to study the exciton dynamics and the corresponding lifetimes of the charge carriers within the developed PC/CNT composites upon the introduction of different ILs. . TAS results shed light on the physical origins of the TE conversion efficiency by showing how the TE values are related to the excitonic properties of the incorporated CNTs in combination with the ILs. Ref: Voigt, J. Compos. Sci. 2022, 6, 25; Konidakis, ACS Appl. Energy Mater. 2022, 5, 9770 EU funding "InComEss" GA 862597

Oral (S09-194, Time: Tuesday 14:10, Room: Olma)

Influence of annealing-induced phase separation on the shape memory effect of graphene-based thermoplastic polyurethane nanocomposites

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Shape memory polymers (SMPs) is an emerging class of smart materials, which are of special interest due to their ability to return to their original shape after being subjected to an external stimulus, such as mechanical stress, electric or magnetic fields, temperature, among others. Thermoplastic polyurethane (TPU) stands out for its easy processability and versatility, which raises its range of final shapes and applications, including membranes, medical implants, smart stents and artificial muscles. TPU is a multiblock copolymer and its morphology is composed by rigid and elastomeric immiscible segments highly dependent on thermodynamic parameters, leading to a phase separation and forming domains. The elastomeric segment (soft domain) is associated with a polyol or a long chain diol, while the hard domain refers to the intercalation of a diisocyanate and a chain extender. However, the low stiffness, tensile strength, thermal and electrical conductivity are still some of TPU limitations. In this scenario, the incorporation of nanomaterials is widely explored in the literature to obtain composites with superior final properties. In this study, 0.1 wt.% of graphene nanoplatelets (GNP) or 0.1 wt% of multilayers graphene oxide (mGO) are incorporated into TPU matrix using solution casting processing, and the contribution on the phase separation of these domains is observed. This phenomenon is even more pronounced when graphene-based nanocomposites are submitted to annealing at 110 ºC for 24 hours, suggesting a good interaction between the GO and GNP with the hard and soft segments, respectively. After annealing, the nanocomposites present better performance in SME regarding the increase on shape recovery ratio (Rrec) of around 9% compared to the non-annealed TPU. All nanocomposites maintained a high strain during SME programming, higher than that of the pure TPU, before and after annealing, indicating a direct influence of the graphene on the shape memory effect.

Oral (S09-257, Time: Tuesday 14:30, Room: Olma)

Dispersion and selective localization of carbon nanotubes in nanostructured block copolymers

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Nanostructured block copolymers (BCP) are very well suited as templates for the introduction and selective localization of nanofillers. The use of carbon nanotubes (CNT) as fillers is based on their exceptional property profile, which enables the development of functional materials with special thermal, mechanical, and electrical properties. In order to increase the compatibility between the BCP phases and the nanofiller and to enable selective placement, CNT can be modified with appropriate functional groups. Due to the small dimensions of the block copolymer nanostructures, it is necessary to use CNT with the smallest possible diameters and lengths and, when selecting the block copolymers, to give preference to materials with lamellar, co-continuous or cylindrical structures with large domain spacing. In this work, two different block copolymer systems are presented in which selective localization in one of the BCP phases was achieved by chemical modification of the CNT and by CNT shortening. The first material system consists of diblock copolymers (BCP) based on poly(methacrylate) PMMA and poly(1H,1H,2H,2H-perfluorodecyl methacrylate) (PsfMA) blocks, into which short perfluoroalkyl-modified multi-walled CNT (MWCNT) were selectively introduced via solution mixing and ultrasonic treatment. Further investigations were carried out on a styrene-butadiene based star block copolymer with shortened MWCNT. The influence of CNT addition on BCP phase-nanofiller interactions, morphology, and electrical properties of the nanocomposites were studied.

Oral (S09-290, Time: Tuesday 14:50, Room: Olma)

In-line modification of Ca-Al LDH with stearic acid

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In order to eliminate the need for pre-treatment of inorganic layered double hydroxides (LDHs), in-line organomodification of Ca-Al LDH with stearic acid was studied. Organomodification of LDHs are required in order to improve the dispersion of LDHs in composites by increasing the interlayer distance of the LDH layers as well as the polymer/LDH compatibility. The modification was performed during direct compounding of the LDH and stearic acid with polypropylene. Polypropylene-grafted maleic anhydride was used to further enhance filler/matrix interactions. The morphological properties of the resultant composites were investigated by electron microscopy. Electron microscopy was also used to characterise the dispersion the LDH in the polymer matrix. The exfoliation behaviour of the modified LHD in the polypropylene matrix was rheologically quantified with the Wagener and Reisinger power law model.

Oral (S09-327, Time: Tuesday 15:10, Room: Olma)

Fabrication of Hydrophobic self cleaning Nano-coatings for Photovaoltaic applications

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Highly transparent, water-resistant, self-cleaning, spectral-converting nano-coatings with outstanding physical properties are in high demand for a wide range of applications. Glass is frequently utilized in solar modules to shield the active components from the damaging effects of harsh climatic factors such as environmental dust, humidity, heavy rains, wind, etc . Due to the great degree of transmittance that glass surfaces possess, sunlight can penetrate into the inside of the solar modules. When this occurs, the sunlight that is taken in by the solar cells can be effectively transformed into electric energy. However, the conditions of the outside environment are typically harsh, and the presence of dust in the surrounding air creates a significant challenge. The accumulation of dust particles on the surfaces of solar modules will lower the transmittance of the protective covers, which will affect the photoelectric conversion efficiency as a result of the dust particles' ability to reflect light and absorb it. In this study thin films made of silica and silane-based composite were developed in this study to have self-cleaning and hydrophobic properties. This research focuses on the development of highly transparent, water-resistant, self-cleaning nanofilms based on silicon nanoparticles and silane-based composites. The resulting self-cleaning silica-based coating has excellent hydrophobicity and transmittance, as well as the ability to clean itself. The resulting layer has been evaluated for self-cleaning and hydrophobicity, and it has a high potential for practical applications.

Oral (S09-368, Time: Tuesday 16:00, Room: Olma)

Green Polyamide 1010 Nanocomposites Based on Hybrid Two-Dimensional Nanomaterials: Effects on Microstructure and Crystallization Kinetics

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The influence of adding different hybrid 2D nanomaterials, namely, GO, h-BN, and MoS2, on the microstructure of a green polyamide 1010 has been investigated. XRD results showed that all the samples presented only one crystalline phase, the monoclinic α phase. It was also shown that the nanomaterials' addition resulted in a decrease in the polymer crystallinity, with the lowest being obtained when h-BN and MoS2 were added. The absence of GO in this system may be the reason for that, since it would be expected that GO has a higher attraction to the polyamide chains due to good interactions between its oxygenated groups and the amide bonds, which would disrupt less the crystalline microstructure. The fillers' nucleating effect was identified by DSC analyses. It was noticed that the composites containing h-BN presented the largest increase in the crystallization temperature, indicating a more prominent nucleating effect. DSC analyses also showed that the crystallization kinetics were slowed down by the presence of nanofillers, especially in the h-BN systems, which explains the lower overall crystallinities and microstructural changes detected by XRD. Interestingly, a double melting behavior was observed. This complex melting is attributed to the Brill transition, which corresponds to the concomitant reorganization of chains that compete with melting. However, the composites presented an increase in the area under the high-temperature melting event in respect to the overall melting area when compared to the neat polymer, which suggests that the hybrid fillers might be increasing the perfection of the crystals and/or restraining the mobility of the chains after the first melting, therefore favoring its participation in the Brill transition. Since this behavior was more important for the system containing GO and h-BN, it is supposed that this specific combination of nanomaterials may present some synergistic effects less evident in the other systems.

Oral (S09-421, Time: Tuesday 16:20, Room: Olma)

Effects of stereocomplex crystallization on the electrical properties of PLLA/PDLA/PVDF immiscible blends filled with MWNTs

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Crystallization-induced segregation of nanoparticles and nanoparticle localization at the interphase or in one phase of immiscible polymer blends with co-continuous morphology are effective strategies to control the distribution of conductive fillers with the aim of generating electrical conductivity at low filler content. It is possible to further improve the electrical conductivity of co-continuous polymer blends containing conductive nanoparticles by thermal annealing at elevated temperatures. The annealing can effectively improve phase continuity and reduce the distance between nanoparticles. In this research, these strategies are combined in 50/50 (PLLA/PDLA)/PVDF blends filled with 0.1 – 1.6 wt% MWNTs to achieve high electrical conductivity and EM shielding performance. The MWNT nanoparticles localize in the PLA phase while inside this 50/50 PLLA/PDLA phase homocrystals (HCs) can form and reform into stereocomplex crystals (SCs) upon annealing at elevated temperatures. The results reveal that the electrical percolation threshold of the MWCNTs in the nanocomposite is reduced from 0.3 to around 0.06 wt% after crystallization. In addition, the SCs formed through melting and recrystallization of HCs at a higher temperature, where there is enough chain mobility, lead to a redistribution of the MWCNTs to form a conductive pathway via enhanced volume exclusion and simultaneous morphological coarsening. For instance, after stereocomplexation at 220 °C, the electrical conductivity of the nanocomposite with high concentrations of MWNTs (e.g. 1.6 wt%) increases to 0.02 S/m. As a consequence, the electromagnetic shielding effectiveness of this sample is enhanced to 11 dB which is ~50% higher than that of samples crystallized at lower crystallization temperatures. This research demonstrates that stereocomplexation at high temperatures in PLA-based blends can be used as a novel approach to fabricate high-performance conductive and EM shielding composites.

Oral (S09-571, Time: Tuesday 16:40, Room: Olma)

Fabrication of Thermally Conductive Anisotropic Polymeric Nanocomposite using Boron Nitride Nanotubes

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The growing global interest in advanced composites has intensified the demand for polymeric composites with advanced properties for various applications, from microelectronic packaging and energy storage to aerospace and biomedical. Among all novel materials for the production of advanced polymeric composites, boron nitride nanotube (BNNT) has attracted great attention due to its special characteristics, such as high aspect-ratio and exceptional thermal conductivity, as well as electrical resistivity and radiation shielding characteristics. The combination of one-dimensional structure and high thermal conductivity make possible the production of anisotropic thermally conductive polymeric composites. To take advantage of the one-dimensional structure of BNNTs to fabricate anisotropic composites, it is essential to align BNNTs in one direction and keep their aspect-ratio as high as possible. It is also critical to create sufficient interconnectivity between the nanotubes throughout polymeric matrices. In this project, aligning BNNTs inside polycarbonate (PC) matrix, using micro twin-screw compounder has been studied, characterized, and analyzed. We were able to stretch the melt and align the BNNTs simultaneously to create anisotropic sheet with 0.06 W/mK through plane thermal conductivity and 1.46 W/mK in-plane thermal conductivity by only using 11 wt.% BNNT in PC. The goal of this project is to develop highly anisotropic thermally conductive with yet electrically insulating polymeric nanocomposites suitable for applications such as microelectronic packaging, thermal management, and power generation.

Poster (S09-252, Time: Thursday 17:00, Room: Foyer)

Contribution of LDH Polymer Composites towards Sustainable Energy Resources

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With increasing environmental changes due to the wide use of fossil fuels, there is a need for continuing research on alternative sustainable energy sources. Sunlight is one such alternative energy source and, in comparison to fossil fuels, abundantly available. For solar-energy-harvesting, research into the synthesis of suitable materials is ongoing. Polymer solar cells are an application of these materials that attract researchers. However, improvements in their design and efficiency are necessary. Layered double hydroxides (LDHs) are a class of materials that have shown potential in such energy applications. After successful application of LDHs in polymers as UV-Vis stabilizers and as flame retardants, our research is now focusing on the understanding of these materials in photovoltaic applications. LDHs owe their versatility to multiple possible compositions and synthesis routes. This work highlights the synthesis and use of ternary metal LDHs in photovoltaic applications. MgAl-LDH was modified with five transition metals (Fe, Co, Ni, Cu, Zn) and two concentrations (5% and 10%) using urea hydrolysis and co-precipitation. X-ray powder diffraction (XRD) spectroscopy, energy-dispersive X-ray (EDX) spectroscopy, scanning electron microscopy (SEM) and thermogravimetric analysis (TGA) were used to characterize the LDHs. The UV-Vis absorption spectra were obtained and the optical band gaps were calculated. A new design concept was used for the preparation of the solar cells and their performances were tested using current-voltage (IV) solar simulator.

Poster (S09-254, Time: Thursday 17:00, Room: Foyer)

Preparation of novel poly(lactic acid)/poly(ethylene adipate) nanocarriers for the controlled and targeted administration of anti-Lingo-1 and anti-Nogo-A antibodies

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Innovations in the field of polymer science and technology continuously afford new drug delivery systems (DDSs) designed for the optimum administration of numerous therapeutic agents. Polymeric-based nanoparticles, compared to conventional therapeutic methods, can overcome common limitations related to the specificity of the target tissue, the release rate, and the biodegradation of bioactive molecules. The development of biocompatible and biodegradable polymeric materials that can be utilized as effective drug-carriers holds therefore a great potential, due to their low toxicity, high processability and tunable properties. Aliphatic polyesters are an important class of these materials, extensively employed in biomedical applications, such as drug delivery. Out of them, poly(lactic acid) (PLA) is a particularly promising candidate and one of the most broadly studied versatile bio-based polymers with multifunctional properties. Its biodegradability can be further tuned by combining it with other linear aliphatic polyesters. The aim of this work was to prepare novel pharmaceutical nano-formulations based on a newly synthesized poly(lactic acid) (PLA)/poly(ethylene adipate) (PEAd) copolymer for the targeted and controlled administration of two antibodies (anti-Lingo-1 and anti-Nogo-A) with proven beneficial effects regarding demyelination. Antibodies-loaded NPs were fabricated according to a double (water-in oil-in water, w/o/w) emulsion/solvent extraction method. TPGS was selected as surfactant according to recent promising studies on its brain-targeting potential. The same protocol was used to produce control batches of nonspecific IgG-loaded NPs and empty polymeric NPs. The nanoparticles were characterized in terms of size and structure, and their effect regarding demyelination after being administrated onto cuprizone induced demyelination mice was investigated.

Poster (S09-301, Time: Thursday 17:00, Room: Foyer)

Chitosan-whey protein isolate core–shell nanoparticles as delivery systems

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Chitosan (CS)-whey protein isolate (WPI) core-shell nanoparticles were synthesized through self-assembly of whey protein isolated polyanions and chitosan polycations in the presence of tripolyphosphate (TPP) as a crosslinker. The formation of this type of nanostructures with narrow particle size distribution is crucial for developing delivery systems since the functional characteristics highly depend on their sizes. To achieve this goal, the nanostructure was optimized by varying the concentrations of WPI, CS, and TPP in the reaction mixture. The chemical characteristics, surface morphology, and particle size of the nanoparticles were evaluated. Keywords: Whey Protein Isolated, Chitosan, Nanoparticles, Delivery system

Poster (S09-357, Time: Thursday 17:00, Room: Foyer)

Agricultural waste as a source of cellulose nanocrystals for extensive photonic applications

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The agricultural industry is rising annually due to the strong demand for food and meat from the world's expanding population. To make use of the organic content in agricultural waste and prevent environmental degradation, prompt and efficient treatment is crucial. The primary content of these agricultural wastes is cellulose. Cellulose, with the application of nanotechnology, has many potential uses, resulting in a spike in the research for separating nanocellulose from various agricultural leftovers. To produce nanocellulose in its two primary morphologies, cellulose nanofiber (CNF) and cellulose nanocrystal (CNC), this study compiles many manufacturing techniques, including conditioning stages, pre-treatments, bleaching processes, and, ultimately, purification. In this work, we have extracted cellulose nanocrystals from various agricultural and food wastes and utilized them as a liquid crystalline material. For extensive applications, the CNCs were tuned and mixed with mono methyl ether of polyethylene glycol at different concentrations. The conversion and extraction of the CNCs were evaluated employing FTIR, XRD, and NMR spectroscopy. The effect of modification on the morphological arrangement of the CNCs was inferred using FESEM, AFM, and TEM analysis. The liquid crystallinity of the material was assessed using rheological studies. The optical potential was evaluated by using polarized optical microscopy. The prepared mixtures were evaluated for their optical anisotropy, resulting in an elliptically birefringent material. The prepared materials' potential in sensing, smart coating, and security materials can be further investigated.

Poster (S09-375, Time: Thursday 17:00, Room: Foyer)

Cellulose nanocrystals: versatility and compatibility in different polymeric systems

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Cellulose nanocrystal (CNC) is an attractive new material due to its renewable origin, lack of toxicity, etc. Becoming a material with great potential to develop a new generation of materials for different applications, from perishable food coating to improve polymer mechanical properties. Due to the abundance of biomass in Brazil, our group is dedicated to the CNC extraction from agribusiness by-products and their application in various polymeric materials. In this abstract we will briefly describe our recent studies applying CNC in three different polymeric systems: 1) hydrophilic polymer, 2) hydrophobic thermoplastic polymer and 3) hydrophobic thermosetting polymer. For this, the following polymers were selected as representatives of each of these groups: hydroxypropyl methylcellulose (HPMC), poly(lactic acid) PLA and a methacrylate copolymers, respectively. To avoid CNC agglomeration, the present work also investigated the importance of functionalize CNC with 3-(methacryloxypropyl)trimethoxysilane (MPS), lactic acid (LA) and 3-(glycidyloxypropyl)trimethoxysilane (Glymo). Firstly, the optimized CNC extraction conditions were established, and both the CNC and functionalized CNC were characterized. Among the different characterizations carried out with the CNC composites (XPS, FTIR, TEM, XRD, TG, DSC, etc.), it is noteworthy that 1 wt% of CNC-Glymo had a nucleating effect on PLA and caused a clear change in the morphology of the cryogenic fracture surface of the material when compared to 1 wt% CNC, suggesting compatibility and chemical bonding between the nanoparticle and the PLA. In turn, 0.5 wt% of CNC-MPS in methacrylate copolymer increased microhardness by 26%, achieving results similar to commercial composites prepared with 50 wt% of silica. HPMC containing 0.5 wt% CNC decreased water sorption by 25 %. The results indicate the great versatility of the CNC and its potential to be chemically modified and adapted to different polymers.

Poster (S09-456, Time: Thursday 17:00, Room: Foyer)

Fire resistant HDPE fuel system by nano-based intumescent coating

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Nowadays, using polymers, high-density polyethylene (HDPE), and polypropylene, to name a few, for fuel storage systems is widespread in many industries like the automobile industry. The automotive fuel system is one of the abovementioned applications, using HDPE in the filling pipe and fuel tank layers, which is one of the main subsystems of the powertrain module in internal combustion engine vehicles. Despite all advantages of using HDPE like weight reduction, design freedom, and so on, some challenges like poor endurance against fire, lead researchers to propose various solutions. One of the noble methods to protect these parts against fire is using fire-resistant coating. In this article, an intumescent nano-based coating is implemented on the samples to protect the samples against fire, 3 mm Wall thickness bottles are used as specimens and the dry film thickness of the coating is about 1200-1500 microns. The main objective of this research is to study the effectiveness of this intumescent coating for the fire protection of fuel systems. Understanding how it improves the resistance of samples against fire, special flame tests using a 1000 °C Bunsen burner as a fire source are done on bottle samples. Results implied that using intumescent coating increases time to leakage over six times. To investigate the durability of the adhesion of the coating on HDPE over time and harsh conditions, PSA standard heating shock and aging tests were done on sheet samples prepared by cutting the fuel tank wall. The samples were coated by intumescent nano-based coating and examined by PSA standard processors. The effects of heat aging and heating shock on adhesion are evaluated through a pull-off test. The adhesive strength of painted plates is measured as 2.63 ±0.76, 2.25±0.72, and 3.01±0.47 for shocked, aged, and control samples, respectively. Moreover, in all cases, no impressive indication of adhesive heat degradation by was observed.

Poster (S09-489, Time: Thursday 17:00, Room: Foyer)

Cast Film Extrusion of PVDF/Carbon Nanotube/Carbon Black Composites: Filler Synergy and influence of extrusion parameters on extrudability and direction-dependent electrical conductivity

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Melt-mixed composites based on poly(vinylidene fluoride) (PVDF) and fillers with different aspect ratios (carbon nanotubes (CNTs), carbon black (CB)) and their mixtures in composites were investigated, comparing compression molded sheets with melt extruded melt-extruded sheets. The processing-induced orientation of high-aspect-ratio CNTs leads to directionally dependent electrical and mechanical properties. The differences between the directions could be reduced by the use of mixed filler systems with the low aspect ratio CB. An upscaling of melt mixing from small scale to laboratory scale was carried out for selected combinations. By combining CB and CNTs in PVDF, the electrical conductivity through the film in particular could be increased compared to PVDF/CNT composites due to additional contact points in the sample thickness. The alignment of the fillers in the two directions within the films was derived from the differences in the electrical and mechanical film properties, which showed higher values in the extrusion direction than perpendicular to it. Films up to a thickness of 50 micrometer were produced from extruded materials with 1 wt% MWCNT and 3 wt% CB by cast film extrusion while varying the processing parameters, such as the melt temperature and take-off velocity. For extruded films with 65 micrometer thickness electrical volume conductivities of 130 S/m in the x-direction, 39 S/m in the y-direction, and 4.9 S/m in the z-direction could be achieved.

Poster (S09-524, Time: Thursday 17:00, Room: Foyer)

Integration of Smart Polymers with Atomically Thin 2D Materials for Advanced Manufacturing of Hybrid Structures

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The integration of polymers and inorganic materials with complementary properties has been widely explored to solve critical scientific and technical problems. In recent years, atomically thin 2D materials, such as graphene and transition metal dichalcogenides, have been more and more used as the functional components in those hybrid systems due to their unique electrical, mechanical, thermal, and optical properties. However, it is still challenging to integrate 2D inorganic materials with functional polymers in a molecularly precise and programmable way beyond conventional nanocomposites. In this talk, I will present our recent work on integrating 2D materials with stimuli-responsive polymers for the fabrication of ultrathin and adaptive structures across different length scales. Our approach combines polymer chemistry, molecular assembly, microfabrication, and origami folding. At the micro/nanoscale, we demonstrated temperature-induced shape transformation of lithography patterned monolayer graphene and molybdenum disulfide into precise 3D microstructures for biosensing and photodetection applications. At the macroscale, we showed that polymer functionalized 2D nanosheets could be assembled into composite membranes or functional coatings with tunable mechanical properties and permeability. Our study provides valuable insights into the advanced manufacturing of next-generation composite materials with precise molecular structure and adaptive properties.

Keynote (S10-153, Time: Wednesday 13:45, Room: Monosuisse)

From melt- to solid-stage polycondensation: how to revolutionize the design of environmentally friendly polymers with advanced properties

Raquez Jean-Marie[1], Mincheva Rosica[1]

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Environmental and economic concerns associated with petroleum-based plastics waste are pushing significant efforts to the introduction of cleaner sustainable technologies. The highest priorities are methods avoiding the use of polluting and unsafe volatile solvents/chemicals; and allowing the facile replacement of the petrol-based monomers by monomers issued from annually renewable resources. With this respect, the polycondensation – a step-growth polymerization - is being attracted much attention. Polycondensation is widely used in Nature, being the main synthetic platform for natural polymers such as proteins and cellulose. In our man-made technology, the process plays an important role in the synthesis of commodity and technical polyesters and polyamides - versatile classes of polymers covering large applications going from fibers to high-performance polymers, thermoplastics and elastomers. Hence the communication will highlight the benefits of using combined melt-polycondensation to other synthetic procedures as chain-coupling or "click" reactions in order to tailor the properties of the functional (co)polyesters. The results have shown that such combined/copolycondensation can be used as a green method to design sustainable plastics going from reinforcing agents to dispersants and curable coatings. To extend the range of functional polymers, the solid-state modification (SSM) from batch into a continuous process by reactive extrusion (REx) will be discussed as well. It represents an easy-to-use and solvent-free tool as it only affects the amorphous part of the polymer, thus preserving its initial mechanical properties while enhancing its recyclability extent. This last study encompasses the design of a new process for recycling polymeric materials and offers the possibility of making polymers more sustainable and recyclable.

Keynote (S10-646, Time: Wednesday 17:00, Room: Monosuisse)

Biobased poly(butylene succinate) copolyesters for the packaging sector

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It is assumed that by rationally designing copolyesters through the ester structure and the soft segments content it is possible to adjust the material properties to specific applications, particularly for use as packaging materials. Polyesters are currently one of the most important groups of materials as they exhibit a number of desirable features. In this study, poly(butylene succinate)-co-(dilinoleic succinate) (PBS-DLS) with a 90-10 hard to soft segments ratio (wt%) was selected to assess its industrial feasibility. First, PBS-DLS 90-10 material has been successfully synthesized via transesterification and polycondensation in melt using a titanium dioxide/silicon dioxide catalyst (C-94) at a semi-pilot scale in a 3L reactor. Nuclear magnetic resonance spectroscopy (1H NMR) and Fourier transform infrared spectroscopy (ATR-FTIR) were used to confirm that the material was a copolyester and that the soft segments were incorporated successfully. Differential scanning calorimetry (DSC) results showed that the PBS-DLS 90-10 exhibited the characteristic glass transition temperature (Tg), melting temperature (Tm), and crystallization temperature (Tc) of semicrystalline materials. The melt flow index (MFI) was also measured, and mechanical properties were tested through quasi-static tensile tests. Finally, the material was extruded into ~150 μm-thick films. The obtained results confirmed that PBS-DLS 90-10 copolymer obtained from biomass substrates is a suitable and promising material for sustainable packaging applications due to its biodegradability, processability, and high mechanical performance. The work was carried out as part of the GREEN-MAP project (H2020-MSCA-RISE-2019) financed by the European Commission with contract number 872152. This scientific work was published as part of an international project co-financed by the program of the Minister of Science and Higher Education entitled "PMW" in the years 2000-2023; contract No. 5091/H2020/2020/2.

Oral (S10-007, Time: Wednesday 14:30, Room: Monosuisse)

From polycondensation to mechanochemistry: the tender for sustainability fulfilled

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A 1.5 MT in 1950 – 359 MT in 2018 - plastic production is tremendous . In EU, only 32% (115 MT) of it is recycled, 25% (90 MT) - landfilled, 43% (154 MT) – incinerated, causing environmental and economic concerns. Despite, plastics remain irreplaceable. Facing the problems University, and later Industry, took the search for sustainable plastics – "materials derived from renewable, recycled and waste carbon resources and their combinations, which at the end of life can be recycled, biodegraded or composted" , using cleaner technologies. Polycondensation – Nature-inspired - played role in the synthesis of biobased polyesters and polyamides with applications in high-performance polymers, thermoplastics and elastomers. Its version – the combined/copolycondensation (chain-coupling/click-chemistry/functional comonomers) allowed tailoring the properties of and bring reactivity to biobased polymers . Solid-state modification (a kind of mechanochemistry) rendered recycling possible . Their combination proved Industrially viable, encompassed problems, and fulfilled the tender for plastics sustainability.

Oral (S10-116, Time: Wednesday 14:50, Room: Monosuisse)

Structure property relationships for network polymers starting from in silico generated molecular distributors

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Polymer network materials are important for a wide range of applications, including drug delivery, construction composites and solar cells. The challenge is the complete understanding of the competition of chemistry and diffusivity during synthesis [1-3], including variations on the balance between inter- and intramolecular reactions as well as the topology of the individual network molecules. The present contribution shows that coupled matrix-based Monte Carlo (CMMC) simulations [1] enable to obtain the 3D positions of individual functional groups and detailed molecular information at any synthesis time. In a next step, molecular distributed properties can be acquired to input in structure-property relationships. Main focus is on step-growth polymerization, including both non-macromolecule and macromolecule building blocks [1-2]. It is shown that specific molecules, e.g. with a given number of crosslinking points of a given type and intramolecular contributions, determine the mechanical strength. In addition, surface properties are tuned by in silico design beyond the sensitivity of conventional properties/ [1] L. De Keer, K. Karsu, P.H.M. Van Steenberge, L. Daelemans, D. Kudora, H. Frisch, K. De Clerck, M.F. Reyniers, C. Barner-Kowollik, R.H. Dauskardt, D.R D’hooge ‘Computational prediction of the molecular configuration of three-dimensional network polymers’ Nat. Mater. 2021 20, 1422 [2] L. De Keer, P.H.M. Van Steenberge, F. Cavalli, D. Estipunan, A.J.D. Krüger, M.F. Reyniers, S. Rocha, L. De Laporte, J. Hofkens, L. Barner, D.R D’hooge 'The synergy of advanced experimental and kinetic modeling tools to understand the synthesis of static step-growth networks involving polymeric precursor blocks’ Macromolecules 2021 54, 20, 9280 [3] L. De Keer, P.H.M. Van Steenberge, M.F Reyniers, D.R. D'hooge* 'Going beyond the Carothers, Flory and Stockmayer equation by including cyclization reactions and mobilitiy constraints' Polymers 2021 13, 241

Oral (S10-284, Time: Wednesday 16:00, Room: Monosuisse)

Comparing techniques for determining the non-catalytic polycondensation kinetics of poly(caprolactone) diol and citric acid

Kennedy Shueh Wah[1], Choudhury Namita Roy[1], Parthasarathy Rajarathinam[1]

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Biodegradable esters prepared from poly(diol)s and acids, most commonly poly(diol) citrates, are attractive candidates for biomedical scaffolds. Combined with current bioprinting advancements, these polymers could fill a niche in creating biodegradable tissue scaffolds. The polymers' mechanical, thermal, and rheological properties influence the printability and viability of scaffolds to support damaged tissues physically and biologically. Therefore, with a view to developing such printable, nontoxic soft material, we have tried to establish the non-catalytic route of polycondensation. This work investigates the synthesis and kinetics of melt polycondensation with polycaprolactone diol (PCL diol) and citric acid to develop PCL-based biodegradable polyesters. Techniques used to track the polycondensation reaction of the polyesters include thermogravimetric analysis (TGA), nuclear magnetic resonance (NMR), and Fourier-transform infrared (FTIR) spectroscopy. The results from these analytical methods were used to create kinetic models, whose predictions were then compared with the experimental values. Future work will focus on different ratios of reactants, the characterization of the polymers' printability and mechanical properties, and the sizing of batch reactors.

Oral (S10-528, Time: Wednesday 14:10, Room: Monosuisse)

Machine Learning-Assisted Design of Charge Transfer Polymers with Full Color-Tunable Emission

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Color tuning of solid-state emissive materials is essential from a fundamental mechanistic viewpoint, as well as for practical applications. Despite enormous efforts on molecular design and engineering, a general and facile platform that offers high flexibility and broad extensibility in emission color tuning is still lacking. [1,2] Here, with the aid of a machine learning model, we developed a polymer platform with versatile color tunability via manipulation of through-space charge transfer (TSCT).[3,4] Using a single-acceptor fluorophore as the initiator for atom transfer radical polymerization (ATRP), a series of electron-donor groups containing simple aromatic moieties were introduced by facile copolymerization or post-functionalization. The resulted TSCT polymer library showed continuously tunable emission color. This was achieved by fine-manipulation of donor-acceptor interplay via simple controlled polymer synthesis. Theoretical investigations confirmed the structurally dependent TSCT-induced emission redshifts. We further demonstrated this TSCT polymer platform can be used to design stimuli-responsive materials with high-contrast photochromic fluorescence (Figure 1). Acknowledgements We acknowledge the financial support for this study from Fondation Claude et Giuliana (research project no. 1-005137) and Swiss National Science Foundation (Spark grant no. 190313). References [1] Ying, L.; Ho, C. -L.; Wu, H.; Cao, Y.; Wong, W.-Y. Adv. Mater. 2014, 26, 2459. [2] Hong, Y.; Lam, J. W. Y.; Tang B. Z. Chem. Soc. Rev. 2011, 40, 5361. [3] Ye, S.; Tian, T.; Christofferson, A. J.; Erikson, S.; Jagielski, J.; Luo, Z.; Kumar, S.; Shih, C. J.; Leroux, J. C.; Bao, Y. Sci. Adv. 2021, 7, eabd1794. [4] Ye, S.; Meftahi, N.; Lyskov, I.; Tian, T.; Whitfield, R.; Kumar, S.; Christofferson, A. J.; Winkler, D. A.; Shih, C.-J.; Russo, S.; Leroux, J.-C.; Bao, Y. Chem 2023, DOI: 10.1016/j.chempr.2022.12.003

Oral (S10-583, Time: Wednesday 16:20, Room: Monosuisse)

A Facile Approach for Preparation of Ion-Exchanged and Intrinsic Porosity-Containing Electrochromic Polymer for Enhancing Redox Switching Perfromance

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A facile approach was applied to prepare triarylamine-containing thermoset styrene-maleic anhydride copolymers (SMA) modified with ion exchange and different crosslinking structures to investigate the counter ions diffusivity of the resulting films during the electrochemical process. In this study, four novel colorless thermoset SMA with anodic electrochromism were prepared by the reaction of two triphenylamine-based diamine monomers, 4,4'-diamino-4''-methoxytriphenylamine (TPA) and N,N'-bis(4-aminophenyl)-N,N'-di(4-methoxylphenyl)-1,4-phenylenediamine (TPPA), with SMA, respectively. TPA and TPPA moieties were introduced into the polyamic acid (PAA) to generate different intrinsic porosity and ion-exchanged structures for investigating the counter ions diffusivity. The resulting TPPA-incorporated polymer films (TPPA and TPPA-TEA) revealed enhanced electrochromic (EC) performance due to the longer distance between the intermolecular chains of thermoset SMA leading to a more extensive porous structure. Furthermore, the triethylamine ion-exchanged PAA films (TPA-TEA and TPPA-TEA) demonstrated a synergistic effect on the electrochromic behaviors. Significantly, the diffusivity could effectively narrow the electrochemical redox potential difference (ΔE), resulting in a shorter switching response time while maintaining similar transmittance change (ΔT). Consequently, combining the more extensive porous structure and ion-exchange modification could promote the EC behaviors much more effectively. The resulting polymer film of TPPA-TEA exhibits a notably short switching response time (2.5 s for coloring time) and a high response speed of 29.24 s^{-1}. Thus, these findings confirm that the simple approach of combining modulable crosslinking distance and ion-exchanged structure can significantly improve EC properties while maintaining the high transparency and transmittance contrast ratio required for practical application as EC materials.

Oral (S10-630, Time: Wednesday 16:40, Room: Monosuisse)

Synthesis of Collagen Modified PLA Biocomposites for Tissue Engineering Applications

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Synthesizing biodegradable, biocompatible, non-toxic, and environmentally friendly biomaterials with good mechanical properties is of great interest for applications in tissue engineering. Within the scope of this study, starting from lactide materials, poly(lactic acid) (PLA) with suitable properties was synthesized by ring-opening polymerization, and collagen-grafted PLA copolymer production was carried out by using type I-III collagen protein. The properties of PLA, having good processability and mechanical properties, have been enhanced with type I-III collagen, which has excellent viscoelastic properties and biocompatibility, offering high bioactivity for tissue engineering applications. PLA was dissolved in dimethyl sulfoxide and reacted with collagen using dicyclohexylcarbodiimide/phosphorus pentachloride. It was confirmed by FTIR analysis that PLA and PLA-g-collagen samples were successfully synthesized. Contact angle results showed that the hydrophilicity of PLA-g-collagen samples was better than that of PLA. In vitro studies showed that the grafting method improved the cell compatibility of PLA. Keywords: poli(lactic acid); collagen; graft copolymers; biocomposites; tissue engineering Acknowledgment: This study is supported by the Scientific and Technological Research Council of Turkey (TUBITAK) with project number 3210038.

Oral (S10-684, Time: Wednesday 15:10, Room: Monosuisse)

In-line monitoring of the evolution of reaction and morphology during reactive polymer blending

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Polymer blending in a screw extruder offers an important route to produce new materials with combinations of properties not available in single polymer components. The morphology of the polymer blend and interfacial adhesion between polymer components dictate the properties of the polymer blend. Their formation is a resultant of thermomechanical and thermodynamic effects which depend on many and highly coupled parameters. For this reason, polymer blending in a screw extruder remains a black or gray box. This work aims to develop an in-line detection method to follow the evolution of reaction and morphology of a reactive polymer blend in a twin screw extruder. To that end, a reactive compatibilizer-tracer is used. It bears both reactive groups capable of reacting with its counterpart upon forming a copolymer for in-situ compatibilization of the reactive polymer blend and fluorescent labels allowing for quantifying very small amounts of interfacial reaction. This allows to study the effects of processing parameters on the evolution of the interfacial reaction and morphology of a reactive polymer blend using an in-line near-infrared spectroscope and an in-line florescence device. The processing parameters include screw configuration, feeding mode, blend composition and screw speed.

Poster (S10-215, Time: Thursday 17:00, Room: Foyer)

Tunable Microstructure and Morphology of Polyethylenes Synthesized by Surface-Tethered α-Diimine Ni2+ Complexes

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From technological and economical perspectives, commercial polymers are strongly determined by the development of a suitable catalyst for polymerization. A key element in catalyst development is the structure design of organometallic complexes. α-Diimine Ni complexes are one of the most examined late-transition organometallics in the application of catalyzed ethylene (co)polymerization. They are emerging as potential alternatives to the established Ziegler-Nata catalysis applied in the olefin industry. Recently, we have developed a new series of novel un-symmetrical α-diimine Ni2+ complexes, exhibiting high catalytic activity (29.1×106 g of PE (mol of Ni)−1 h−1) and remarkable thermal stability in ethylene polymerization. The synthesized PEs were characterized as the ultrahigh molecular weight (1.81×106 g mol−1) and various microstructures via a so-call chain-walking mechanism. The incorporation of the terminal hydroxyl group in the ligand enables the covalent immobilization of the Ni2+ complexes on the solid substrates for ethylene heterogeneous polymerization. Polymerization using these heterogeneous catalysts leads to the formation of polymers with fibrous nature. Using these new catalysts, we aim at future polymer fibers that could be generated directly in a low-temperature catalytic process.

Keynote (S11-060, Time: Tuesday 10:15, Room: Empa)

Generalized CFD/DEM Global Modeling of Polymer Extrusion

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It is a challenge the global modeling of extrusion, i.e. description of solid transport, polymer melting and melt flow. The melt flows are well known. However, the solid transport and polymer melting are poorly understood. The models of polymer melting are based on experiments to get to know the melting mechanism. These models are not general in nature since are based on a priori assumed melting mechanism, and are limited to the specific extrusion method. These are also not general due to the material processed, and geometry/operating parameters. The question arises if it is possible to solve the problem of modeling of extrusion without referring to the specific solid/melt flow mechanism, determined by the extrusion method, material type and process conditions. One can hypothesize that it is possible to model the melting/melt flow by solving the fluid dynamic equations without distinguishing the solid and liquid phases, and determining these by temperature distribution obtained as the result of computations. The solid conveying can be modeled based on granular mechanics using the discrete element method. The classic modeling of extrusion is based on the separate models for each screw section, i.e. solid transport, melting, and melt flow section, and the global model consists of these models. The concept proposed here is based on the continuum CFD modeling which does not consist of elementary models, and describes the melting/melt flow in the entire screw space. The problem of implementing the continuum CFD computations for the melting/melt flow and the discrete DEM computations for the solid conveying into the global model of the process is a challenge. The novel concepts of the melting/melt screw characteristics, and the solid conveying screw characteristics are proposed, similarly to the screw pumping characteristics applied for the melt flow section. Some examples of computations based on this concept are presented.

Keynote (S11-090, Time: Tuesday 13:45, Room: Empa)

Morphology of injection molding polypropylene parts induced by in-mold annealing: modeling and analysis

Titomanlio Giuseppe[1], Salomone Rita[1], Speranza Vito[1], Liparoti Sara[1], Pantani Roberto[1]

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It is generally recognized that high temperature treatments during solidification, namely annealing, influence the microstructure and the morphology, which in turn determine the mechanical properties of polymeric parts. In mold annealing can be thus adopted to control the mechanical performance of the molded parts. The aim of this study is to assess the effect of annealing on the morphology developed in isotactic polypropylene (iPP) injection molded parts. The in-mold annealing is obtained by controlling the mold temperature: the polymer is injected in a mold at high temperature (140°C or 160°C), which is kept for 5 minutes (first annealing step); afterward, the mold temperature is lowered and kept to a lower value (130°C) for a time compatible with the crystallization half time at that temperature (second annealing step). The characterization of morphology is carried out by optical and electronic scanning microscopy. It was found that: the temperature of the first annealing step does not influence the thickness of the fibrillar skin layer, which is, however much thinner if compared with a molded part obtained without the annealing step. The thickness of the fibrillar skin layer is also not affected by the second annealing step. In the part core, the spherulite dimensions change by effect of both annealing steps. A model that considers spherulite and fibril growth is adopted to describe the effect of molding conditions on the final morphology distribution along the part thickness. In particular, morphology is predicted (on the basis of local evolutions of both temperature and molecular stretch and of the competition between fiber and spherulite crystallization. The model, which takes as input the thermo-mechanical histories calculated by a commercial software, adequately predicts main effects of the molding conditions on the final morphology distributions.

Keynote (S11-149, Time: Wednesday 10:15, Room: Empa)

Calibration of Material Properties Using Measured Shrinkage Molding Data for Improved Part Shape Prediction

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A key objective of process simulation of thermoplastic injection molding is the accurate prediction of the final part shape. Deviation of the molded part shape from the intended design is known as warpage. In this research, we present a method to improve the accuracy of warpage prediction by using calibrated material properties. The calibrated material properties can be the modulus, Poisson's Ratio and Coefficient of Thermal Expansion. The calibration is achieved using a database of measured shrinkage molding data from a series of standardized test plaque having a variety of molding thicknesses and using a variety of process condition settings (packing pressure, melt temperature and injection velocity). Unique in this approach is that the calibration of material properties is performed on-line at the time of the process simulation rather than in an off-line calibration process which prepares static material property values. The advantage of on-line calibration is that the calibration can be based on the particular molded test cases from the database of measured shrinkage data which are most relevant for the part design and process to be simulated. For example, the material property calibration can be done using measured shrinkage samples for which the plaque thickness and process parameters most closely match the thickness and indented processing conditions of the part to be simulated. In this way, the calibration of material properties is uniquely adapted for the design which is to be simulated. Presented in this work are comparisons to actual molding data of final part shape predictions for both unfilled polymers and fiber reinforced polymer composites performed both with and without the shrinkage test calibrated material properties. This includes a thin-walled part for which a post-molding buckling response is correctly predicted when the calibrated material properties are used.

Keynote (S11-297, Time: Wednesday 13:45, Room: Empa)

60 Years of the K-BKZ Constitutive Relation for Polymers

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The K-BKZ constitutive model is now 60 years old. The paper reviews the connections of the model and its variants with continuum mechanics and experiment, presenting an up-to-date recap of research and major findings in the open literature. In the Introduction a historical perspective is given on developments in the last 60 years of the K-BKZ model. Then a section follows on mathematical modeling of polymer flows, including governing equations of flow, rheological constitutive equations (with emphasis on viscoelastic integral constitutive equations of the K-BKZ type), dimensionless numbers, and boundary conditions. The Method of Solution section reviews the major developments of techniques necessary for particle tracking and calculation of the integrals for the viscoelastic stresses in flow problems. Finally, selected examples are given of successful application of the K-BKZ model in polymer flows relevant to rheology including slip at the wall and non-isothermal flows.

Keynote (S11-454, Time: Thursday 10:15, Room: Empa)

Dynamics and self-healing of amine functionalized polyolefins

Hatzikiriakos Savvas G.[1], Yavitt Benjamin M.[1], Gilmour Damon M.[1], Zhang Ziyue[1], Moradinik Nafiseh[1], Kuanr Nirmal[1], van Ruymbeke Evelyn[2], Schafer Laurel L.[1]

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Aminated polyolefins (APO) were synthesized and used to study their dynamics through linear viscoelasticity. The rheological and mechanical properties of these materials are derived from their dynamic, reversible associative interactions between the high degree of hydrogen bonding in the secondary amine groups. Material properties are dependent on the molecular weight (Mw), ranging from a soft polymeric liquid to an entangled solid with long relaxation time. APOs exhibit autonomous self-healing ability. The cooperative impact of entanglements and associations are modeled by a tube-based model to calculate characteristics of the chain dynamics and predict their linear viscoelastic behaviour. In addition, self-healing results are explained on the basis of a theory (Stukalin, et al. Macromolecules, 2013, 46, 18, 7525–7541) developed for such systems that relates the recovery of their mechanical properties (tensile strength and ultimate elongation) with healing time.

Keynote (S11-602, Time: Thursday 13:45, Room: Empa)

Morphology distribution within injection molded parts obtained with fast cavity heating cycles and different packing pressures

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Injection molding is one of the most diffused processes for the mass production of polymeric components. The aspects related to environmental pollution make mandatory the reduction of plastic waste, especially during polymer processing. To this aim mono-materials (namely products only composed of a single type of material, instead of using a material with additives and fillers) with mechanical properties, surface characteristics, and durability addressed through the process are used. The properties of a molded part, in the case of semi-crystalline polymers, mainly depend on the morphology developed during the process. The rapid heating of the cavity surface, coupled with the possibility of maintaining the temperature for an appropriate time during the process, allows for achieving desired part performance. However, only a few works focused on the prediction of morphology development during the process. In semi-crystalline parts, the morphology is composed of spherulites at the core and fibrils at the surface: the fibril formation is marginally explored, although determining the mechanical performances. In this work, a two-steps approach is proposed to predict fibril formation in the case of a well-characterized polypropylene. The first step describes temperature and flow fields during the process; the second step adopts the outputs of the first one for describing fibril formation. Several temperature cycles are selected for the cavity surface, and two pressures are selected for the packing stage. The simulation outputs for temperature, pressure evolutions, and morphology distributions successfully compare with the experimental findings. In particular, the simulations consistently predict that the fibrillar layer thickness reduces with both the increase of cavity temperature and the decrease of packing pressure.

Keynote (S11-681, Time: Friday 10:15, Room: Empa)

Effect of Network Defects on the Microscopic and Spatially heterogeneous dynamics of Vitrimers

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A significant amount of scientific and technological emphasis has been given to the problem of recycling and reprocessing permanently crosslinked polymers known as thermosets. Vitrimers provide a sound solution to this problem owing to their ability to undergo topology rearrangements under a thermal stimulus. The presence of a dynamic covalently bonded network combines the strength of thermosets with the re-processibility of thermoplastics. Nevertheless, there is a gap in the literature on understanding the effects of factors such as bond exchange dynamics and defects in the network structure on the said topological alterations. In this study, we will use a hybrid molecular dynamics-Monte Carlo simulation to investigate the effects of various degrees of defects on the microscopic dynamics of the vitrimer matrix. The correlations between the network defects and the topology freezing temperature will be studied using the volumetric properties of the systems. Then the value of topology freezing temperature will be related to the microscopic dynamics of the network that will be characterized using the mean squared displacement and van Hove autocorrelation function of the crosslinking beads. Further, we investigate the spatial dynamical heterogeneities in glassy vitrimers under applied shear creep as prior studies show that mechanical deformation can have an effect similar to increasing the temperature and inducing dynamical heterogeneities in glassy materials. Vitrimers undergo a significant amount of creep as compared to thermosets which greatly restricts their range of applications. Thus, it is necessary to examine these microscopic dynamics in thermosets and vitrimers at temperatures below the glass transition and correlate them to the stress distribution in the materials on loading and understand the factors that lead to the mechanical failure of these materials during applications.

Keynote (S11-687, Time: Wednesday 17:00, Room: Empa)

An Improved Approach to Model the Temperature Control in Polymer Extrusion

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The rheology of polymer melts is clearly affected by temperature; thus, the temperature control system of the processing equipment must be properly designed, to assure the aimed products quality and process stability. With the current CAE tools it is possible to simulate different stages of the extrusion process. However, some strategies used process modelling still consider some simplifications, when compared with the experimental practice, which might lead to inaccurate results. For instance, in what concerns to the temperature control, in computational modelling the temperature is usually set at the flow channel surface. In practice the temperature is controlled by thermocouples that measure the temperature in a point of the metallic tool, and the heaters operation controlled by the referred thermocouple reading. Based on the authors knowledge, the errors promoted by this approach were not quantified yet. In this work the temperature control system that is employed in practice (thermocouple and heaters) is replicated in the modelling, by implementing a novel boundary condition in a multi-region flow and heat transfer solver in OpenFOAM computational library. This is expected to allow quantifying the loss of accuracy of the most common approaches, and to guide the implementation of new, and more precise, modelling methods.
ACKNOWLEDGEMENTS This work was funded by National Funds through FCT - Portuguese Foundation for Science and Technology, Reference UID/CTM/50025/2019 and UIDB/04436/2020.

Oral (S11-041, Time: Tuesday 11:00, Room: Empa)

LCA meets Moldflow: integrating sustainability metrics into plastic injection mould simulation

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LCA meets Moldflow: integrating sustainability metrics into plastic injection mould simulation The aim of this development is to combine two worlds in engineering from part design of injection-moulded thermoplastic parts to sustainability approaches using LCA (Life Cycle Assessment). While classical LCA uses data from real machines and material, this time data from process simulation using commercial software will be used instead. The LCA will be performed "cradle to gate" according to DIN EN ISO 14040 and 14044. The tool, being developed within the program TTP-LB from German Government BMWK, will produce results for all impact categories of Environmental Footprint (EF3.0). Proof of concept will be Climate Change [kg CO2 eq.] and use of resources, fossils [MJ]. The tool addresses design engineers to get an idea of environmental impacts depending on choice of material, design and processing in early design phases. This allows significantly reducing sustainability related risks and can be used to avoid sustainability pitfalls from the start. Data from AUTODESK Moldflow will be used and processed to derive input for LCA using energy consumption and material consumption. The material data information is used from material cards within AUTODESK Moldflow such as density, viscosity, heat capacity, melting temperatures, etc. Furthermore, information on the process is resulting from AUTODESK Moldflow studies, such as tempering system, system pressure, melt temperature, total mass, etc. The processing of these data as input to the newly developed tool will be realised by API programming. The aim of this development is to combine two worlds in engineering from part design of injection-moulded thermoplastic parts to sustainability approaches using LCA (Life Cycle Assessment). While classical LCA uses data from real machines and material, this time data from process simulation using commercial software will be used instead. The LCA will be performed "cradle to gate" according to DIN EN ISO 14040 and 14044. The tool, being developed within the program TTP-LB from German Government BMWK, will produce results for all impact categories of Environmental Footprint (EF3.

Oral (S11-050, Time: Tuesday 11:20, Room: Empa)

Modeling the Pressure-Volume-Temperature Behavior of Amorphous Polymers: About the Logistic Model Equation of State

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To date, accurate description of the pressure-volume-temperature (PVT) behavior in the vitreous state and at glass transition, a key feature of valid simulation of the processing of amorphous polymers, has often been subject to limitations of theories and models. Consequently, only a few models that describe the PVT behavior of amorphous polymers with limited predictive capability are implemented in commercial software of polymer processing simulation. In our research, we utilize the concepts of thermodynamics of irreversible processes to overcome these limitations. The non-equilibrium nature of amorphous polymers in the vitreous state and at glass transition is accounted for by introducing internal state variables in addition to the natural state variables of the corresponding thermodynamic potential. A result of this research is the logistic model equation of state (LM EOS). Unlike standard PVT models such as Tait's EOS, the LM EOS accurately describes the PVT behavior of amorphous polymers in the equilibrium state, the vitreous state and at glass transition without dividing the temperature domain into multiple intervals. Therefore, discontinuities are eliminated, and no conditional statements that divide the temperature domain into intervals of different model ansatzes and different model parameter-values have to be implemented in software. The LM EOS is continuously differentiable with respect to temperature and pressure, thus allowing for determination of the thermal expansion coefficient and compressibility at any valid reference state. In conclusion, polymer processing simulation could benefit from implementation of the LM EOS in commercial software. It is worth noting that a procedure to identify the parameters of the LM EOS from experimental data is available. The logistic model ansatz can also be used to determine the pressure and cooling-rate dependent glass transition temperature from experimental data.

Oral (S11-078, Time: Tuesday 10:40, Room: Empa)

Design of the plastic composter for home and educational purposes

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In the Republic of Croatia, in the last few years, awareness has started to be raised about waste disposal, especially waste separation and the education of younger people. The composting of household waste, such as food, fruit and vegetable waste, plays a significant role in separation. In this work, the design process of the small plastic composter (20 l) for the home usage and educational purposes is presented. Certain requirements on the composting space and composting mass are presented, and, along with the manufacturing and logistical aspects, all were taken in consideration when evaluating design concepts. Two different general shapes of the product were considered, and more ways of assembling were proposed and evaluated before reaching a final design. Numerical simulations of injection moulding and static structural loading were employed to evaluate design validity and assist in final design decisions.

Oral (S11-099, Time: Tuesday 11:40, Room: Empa)

Dispersion in Creeping Flows: Analytical Model

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Dispersion of solid particles (like carbon-black) carried by polymer melt flow is the outcome of the competition between stresses imparted by the fluid and the cohesion stresses of the solid particles. In this work we consider the effect of: (A) pressure and (B) shear stresses. We model the fluid flow using the Tadmor [1] unwound channel (rectangular cross section) approximation of the single screw extruder. (A) Close to the corner of the rectangular cross section, the dominant cause of rupture of the solid particles is the negative pressure p which diverges [2] as the corner is approached: $|p| \sim 1/r$, where r is the distance to the corner. Assuming the solid particle to be composed of small parts held together by the cohesion pressure pc, we expect the probability for breaking internal connections to be proportional to p/pc. Using the percolation model we determine the distribution of fragments and their average size. (B) Far from the corners the shear stresses dominate the dispersion process through erosion [3]. We assume a particle to be made of primary fragments bound together. In the erosion process a primary fragment breaks out of a given particle. Using the analytical solution for the longitudinal and transversal velocity field, we consider the erosion of particles (e.g. carbon-black agglomerates) advected by the polymeric flow. The erosion process is assumed to be a Poisson process with a probability of erosion per unit time proportional to the ratio of the largest shear stress to the cohesive stress. The system starts monodisperse (large size particles) and evolves through a poly-disperse regime at intermediate times to a monodisperse (small size particles) at late times. REFERENCES [1] Principles of Polymer Processing, Z. Tadmor and C. Gogos. [2] Analogy between Thermodynamic Phase Transitions and Creeping Flows in Rectangular Cavities, M Kaufman, PS Fodor Symmetry 12 (11), 1859. [3] Erosion in extruder flows: Analytical and numerical study, M Kaufman, PS Fodor AIP Conference Proceedings 1914 (1), 080003.

Oral (S11-104, Time: Tuesday 12:00, Room: Empa)

Modeling the bonding behavior of thermoplastic UD-Tapes during hot stamp spot welding

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In the processing of thermoplastic composites, joining UD tapes to form a layup is a crucial step. As part of the pick-and-place principle multiple tapes are welded locally by a hot stamp. The quality, i.e. the bonding strength, of the weld must be sufficient to transport the obtained tape stack to subsequent processing steps. The bonding strength is influenced by hot stamp temperature, contact pressure and time as well as rheological properties of the matrix material (viscosity and reptation time). Due to the numerous influence parameters it is hardly possible to obtain the optimum settings for a time- and energy-efficient process. To overcome this issue, a digital model of the welding process was developed that provides information on the bonding of two individual tapes during the welding process. The model includes the heat transfer between the hot stamp, the tapes, the welding table and the surrounding air. Based on the temperature rise, which affects the rheological properties of the matrix material, the degree of intimate contact and degree of healing are determined, the combination of which gives the degree of bonding. The validity of the model is shown by comparison with experimentally obtained data, where the bonding strength of the welding spots for various process parameter sets is determined by a peel off test.

Oral (S11-142, Time: Tuesday 14:30, Room: Empa)

Contactless detection of the parison geometry and wall thickness during extrusion blow moulding

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The wall thickness distribution of the blow-moulded product determines not only the economic efficiency of the process, but also its product quality. This is mainly determined by the parison geometry, i.e. its diameter, wall thickness and length. Both the extrusion of the parison and the material properties of the plastic are therefore of decisive importance for repeatable production of the parison. Process monitoring of preform extrusion is necessary to be able to measure process variations, as these are increasing due to the rising use of recyclates. Today's systems often have a high measurement uncertainty. The objective of the present study was to develop a monitoring system for the extrusion of parisons that enables both the temperature of the parison and its geometric dimensions to be measured. In order to achieve this, a measuring system based on an infrared camera was developed and tested, which enables complete and non-contact monitoring of the parison extrusion. Based on these measurements, a model was developed to determine the parison wall thickness. Extensive process tests have shown that the measuring system can measure the geometry and temperature of the parison reliably and accurately. Particularly suitable for assessing the quality of the parison extrusion are the length, the speed and the centring of the parison. Variations in the process can be accurately detected using this measured variable. The preform wall thickness modelling based on the preform temperature difference enables the calculation of the wall thickness within the test space for carbon black-filled plastics with an average error of 4.84 % for PE-HD, with an absolute error of 0.12 mm on average. To summarise, the results show that parison extrusion monitoring can be used to detect both the geometry as well as the temperature of the parison. In industrial production, the newly developed measuring system can be used to detect process variations and thus reduce defective products.

Oral (S11-166, Time: Tuesday 14:50, Room: Empa)

Modeling of Pharmaceutical Twin-Screw Extrusion Processes through Experimental Characterization of Screw Parameters

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Extrusion processes are established in several industries for processing highly viscous materials. In pharmaceutical technology intermeshing co-rotating twin-screw extruders are of particular interest to prepare new types of pharmaceutical formulations in which the drug is molecularly dispersed in a polymer matrix. One major challenge is the prediction of optimal process conditions and equipment configurations. In this context, numerical simulations are a promising tool to complement extensive series of experiments and thus reduce time and costs. Within this study, a 1D simulation software is developed that predicts pharmaceutical twin-screw extrusion processes. The software is based on physical models, originally proposed by Kohlgrüber [1] to describe the pressure and power consumption by dimensionless screw parameters (A & B parameters). Since these screw parameters are not available from literature an extruder test rig was designed by scratch to determine the dimensionless screw characteristics by measuring the pressure built-up, throughput and power consumption of various types of screw elements. This extruder test rig is geometrically similar to the Leistritz ZSE 27 and consists of a transparent acrylic barrel and a direct torque measurement at the screw shafts. The resulting screw parameters are mandatory inputs for the self-developed 1D simulation software to calculate relevant process parameters such as the power consumption, temperature profile, degree of filling and residence time. One major benefit of this software is the individual addition of models that describe pharmaceutical sub-processes such as the mixing, dispersion and dissolution of drugs within a polymer matrix. Finally, the optimal process conditions and screw configuration can be predicted for various processing tasks. [1] Kohlgrüber, K.; et al., Co-Rotating Twin-Screw Extruders – Fundamentals, Technology and Applications, Hanser Publishers, Munich (2008)

Oral (S11-167, Time: Tuesday 16:00, Room: Empa)

Simulation-based Optimization Approach for the Gate Location Optimization of Injection Molded Plastic Parts

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Injection molding (IM) is one of the most widely used polymer manufacturing process. An important design variable for determining final part quality of IM parts is the gate location, which strongly influences the filling pattern inside the cavity. However, identifying the optimal gate location that improves multiple performance indicators simultaneously is non-trivial. In this work, the simulation-based optimization approach was used for the gate location optimization problem. Here, the optimization problem is solved via response surface methodology (RSM). An automated workflow was implemented in order to establish the communication between IM (Autodesk Moldflow®) and optimization (ANSYS optiSLang®) software. Moreover, the workflow is also responsible for mapping designs generated via the Space filling Latin Hypercube sampling method onto a valid gate region. This valid gate region is a user-defined set of nodes from the 3D-mesh IM model. Optimization studies were conceptualized in order to verify different aspects of the automated workflow for both unreinforced and short fiber reinforced plastic parts. In total, three optimization goals were investigated: injection pressure, warpage and a combination thereof. The automated workflow successfully identified better gate locations for all studies, both for single and multi-gated models. In summary, the method demonstrated remarkable assistance in the identification of optimal gate locations for the injection molding process of both unreinforced and short fiber reinforced plastic parts.

Oral (S11-189, Time: Tuesday 16:20, Room: Empa)

Controlling temperature gradients to increase inter- and intralayer bond strength in fused filament fabrication: a 3D modelling study and design

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Fused filament fabrication (FFF) is an additive manufacturing (AM) technique for polymeric part production with applications in areas such as medical devices, sporting equipment and general prototyping. One of the limitations with this technique is the anisotropy intrinsic to the stepwise and directional deposition of material. This creates inter- and intralayer weld zones and voids, each of which are potential weak zones and decrease the strength of the final part. It is thus important to maximize the strength of these weld zones and minimize the voids. On the molecular scale, the bonding quality is influenced by interdiffusion, hence, chain mobility, which, under melt conditions, is governed by chain reptation [1]. It is known that the temperature strongly influences the chain mobility between polymeric phases so that its temporal evolution is an important factor to control the bond formation. Expanding on the already published work on nozzle exit flow modelling [2], the current work numerically investigates the thermal behavior during FFF deposition for four layers consisting out of two strands each and compares the influence of different process parameters such as the nozzle and bed temperature and the layer height. The simulations are based on an ABS material for which a Cross-Arrhenius model is fitted and all relevant thermo-physical properties are measured. Currently, more parameters are investigated, together with the void formation and apparent part roughness [3]. [1] De Gennes PG. Reptation of a polymer chain in the presence of fixed obstacles. J Chem Phys 1971;55:572–9. https://doi.org/10.1063/1.1675789. [2] Van Waeleghem T, Marchesini FH, Cardon L, D’hooge DR. Melt exit flow modelling and experimental validation for fused filament fabrication: From Newtonian to non-Newtonian effects. J Manuf Process 2022;77:138–50. https://doi.org/10.1016/j.jmapro.2022.03.002. [3] Van Waeleghem T, Marchesini FH, Cardon L, D’hooge DR 2022, to be submitted

Oral (S11-190, Time: Tuesday 16:40, Room: Empa)

Estimation of hygro-thermo-mechanical properties of thermoplastics via Molecular Dynamics

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The design of injection-molded thermoplastic parts at the industrial scale must consider the manufacturing process as well as the subsequent mechanical part performance under operational loading conditions. On top of the direct mechanical loads, the thermoplastic parts operate under different temperature and humidity conditions, which should become intrinsic variables of the engineering design process. In the framework of simulation-based design, the respective process-to-structure simulation workflows need material constitutive laws that incorporate dependencies on environmental variables such as temperature and relative humidity. The calibration of this kind of parametric material models require typically a considerable amount of experimental data at the coupon level, which for fiber-reinforced thermoplastics represent a non-negligible effort. An alternative approach would consist in generating virtual mechanical data of the unreinforced thermoplastic polymer under different temperature and humidity conditions. Posteriori, the thermo-mechanical behavior of the fiber-reinforced composite could be estimated using analytical or numerical micro-mechanical models. This virtual identification approach would have the potential of reducing drastically the current experimental effort in the traditional simulation-based design. Concretely, we propose to use Molecular Dynamics (MD) for estimating the hygro-thermo-mechanical properties of semi-crystalline thermoplastics. The effective properties of the semi-crystalline system are computed from MD calculations of fully amorphous and fully crystalline systems. In this work, we discuss the limitations, challenges and opportunities of this approach using a polyamide system as reference. We tackle the estimation of the glass transition of wet amorphous polyamide and the time scales for mechanical loading at the MD level.

Oral (S11-231, Time: Wednesday 10:40, Room: Empa)

Estimation of demolding forces in injection molding with simulation

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In injection molding, the demolding of plastic parts is a crucial phase, where the produced part or even worse, the mold can get damaged. When designing parts or the corresponding molds, the demolding phase is often considered only secondary. This might be especially critical when working with parts to be known to be problematic to demold. To quantify the demolding behavior of a specific part even before manufacturing the mold, a simulation of the deforming phase can deliver an important insight into the loads in the part and the demolding system. A workflow was developed, to describe the demolding process in a simulation based on both calibrated part shrinkage and surface friction. The workflow consists of a simulation, where the deformation of the plastic part at the time of being deformed is used to estimate the resulting temperature dependent surface pressures on the mold. This deformation is calculated using standard rheologic software. In order to estimate the part shrinkage more accurately, the underlying material data is calibrated using a flat circular segment, where the shrinkage in longitudinal and cross flow direction is reverse engineered. To determine the required friction values for the later demolding simulation, a separate apparatus was built to quantify the tribological behavior of various material combinations, mold surfaces and surface pressures. The device allows to imprint the mold surface onto a plastic plate by reheating this surface to melting temperature and measuring normal and frictional force at demolding temperature to calculate static/dynamic friction coefficients. As there can be slight local movements on the contact surface while measuring the static friction threshold, this coefficient is additionally calibrated by reconstructing the measurement process in a simulation. With this workflow, it is possible to estimate the resulting demolding forces of a plastic part and get a better insight in the local loads in the part and mold.

Oral (S11-238, Time: Wednesday 11:00, Room: Empa)

Warpage prediction for UD-reinforced thermoplastic extruded profiles

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[1]KU Leuven, Leuven, Belgium

Thermoplastic over-extrusion is a continuous production process in which reinforcing UD-fibers are preheated and guided at constant line speed into an extrusion die. Here the fibers are surrounded by molten polymer. After the extrusion die, the composite is cooled by a combination of calibration units and water baths. At the end of the process line, the parts are cut to length. Products made with this process have a (or multiple) reinforced zone(s) in their cross-section, allowing for improved mechanical performance with a minimum of reinforcing materials when strategically located. However, due to the mismatch in coefficient of thermal expansion between the reinforcing fibers and the surrounding polymer, excessive warpage and residual stresses after cooldown are often present in the final products. Reducing these unwanted effects is currently done by a costly and lengthy trial-and-error approach. This study presents a multiphysics (thermal-structural) simulation to predict warpage and residual stresses in an over-extruded product with complex cross-section using UD-GF/PET commingled yarns over-extruded with rigid PVC. It makes use of experimentally determined temperature-dependent mechanical properties as well as temperature-dependent thermal properties (specific heat capacity, thermal conductivity, coefficient of thermal expansion). The influence on warpage and residual stresses of process parameters changes (such as line speed and preheating temperature) as well as geometric variations (location of reinforcement in the cross section) are explored both virtually and validated experimentally.

Oral (S11-244, Time: Wednesday 11:20, Room: Empa)

Modeling of the Orientation of Liquid Crystal Polymers in a Slit Die under Wall Slip Conditions

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Thermotropic liquid crystal polymers comprise rigid chain segments, which are called mesogens. Those mesogens are oriented due to shear in a melt flow and their orientation determines the local mechanical properties of the product. A slip or no slip condition at the wall influences orientation. In this work, a new model is presented to calculate the flow orientation of those mesogens in a slit die under consideration of wall slip. For this purpose, a combination of shear flow and slip flow is assumed. The shear thinning behaviour of the polymer melt is taken into account using a power law model. The mesogens are considered as rigid rods with finite length and an initial orientation angle in the flow field. Those rods perform a differential translational movement in the flow direction during a differential time step. In addition, due to the velocity distribution and local shear rate, a differential rotation takes place. An analytical equation is obtained which enables the calculation of the orientation dependent on the position in the die and the local residence time. Moreover, a method to calculate the thickness of the highly oriented boundary layer is presented. With the no slip condition the mesogens are oriented in flow direction close to the die wall due to the high shear rate. The orientation of the mesogens decreases with increasing distance from the die surface towards the centre of the flow. When introducing slip at the wall a more uniform mesogen orientation over the die height is obtained. The thickness of the highly oriented boundary layer increases with rising residence time according to a root function. Wall slip effectuates a much slower increase of the thickness of the boundary layer.

Oral (S11-255, Time: Wednesday 11:40, Room: Empa)

Enhancing vibro-acoustic performance predictions of injection moulded lightweight metamaterial solutions by including manufacturing process simulations

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In the search for suitable manufacturing processes to mass-produce vibro-acoustic metamaterials, a novel class of lightweight and compact noise and vibration solutions, injection moulding has recently shown promising potential. These metamaterials rely on the addition or inclusion of carefully tuned resonant structures in or on a flexible host structure, which leads to targeted frequency ranges of strongly increased vibration attenuation and sound transmission reduction called stop bands. However, as the manufacturing process can lead to changes in geometry and material properties as compared to the CAD design, which is typically considered together with tabulated material properties in the numerical vibro-acoustic performance prediction models, off-design performance can result. This leads to multiple design and manufacturing iterations which are needed to close the gap between predicted and manufactured performance, which is undesirable. To address this issue, in this study, dedicated injection moulding process simulations are used to numerically predict the resulting geometry and material density distribution of produced resonators after manufacturing. By introducing these process simulation outcomes in vibro-acoustic finite element metamaterial models, accounting for the injection moulding process effects becomes possible. For a range of injection moulded tailored resonator designs, the benefits of incorporating the process effects in view of achieving more accurate stop band and noise and vibration predictions as compared to conventional non-manufacturing process informed predictions are investigated. In addition, remaining aspects for future improvement are identified.

Oral (S11-305, Time: Wednesday 14:10, Room: Empa)

A retrofit solution for substituting a conventional primary distribution by using additive manufacturing in a spiral mandrel die assembly in blown film extrusion

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In blown film extrusion spiral mandrel dies are the most common die design. This type of die assembly consists of three main components dividing the course of the melt into a 90° deflection, a n-fold star division and a spiral mandrel itself. To manufacture the components conventional subtractive manufacturing processes are used. Therefore, shaping inside the die is mostly limited to straight-line flow paths particularly in the area of the melt deflection and melt distribution. Partially, this leads to inhomogeneous flow patterns as fillets can only be integrated sparsely due to the restrictions of the manufacturing technologies. The use of additive manufacturing, on the other hand, offers the possibility of realizing channels being freely routed between inlet and outlet resulting in a gentler melt deflection. In a previous research project, a die assembly for blown film extrusion consisting of a primary and a secondary distribution was additively manufactured. Many advantages were achieved by the additive primary distribution alone through the free design of the melt channels. This paper introduces a new die design concept containing a retrofit solution for conventional spiral mandrel dies which substitutes the conventional primary distribution by an additively manufactured one. For this, the die design concept developed within the preceding project will be adapted in such a manner that it replaces the conventional primary distribution and can be combined with an existing spiral mandrel manufactured conventionally. As spiral mandrels are well established in the industry and as they are expensive components, they are retained. In this way, the advantages of both concepts, e.g. the gentle melt deflection, uniform melt pre-distribution and weld line avoidance, are combined.

Oral (S11-306, Time: Wednesday 14:30, Room: Empa)

Significance of curvature for balancing polymer melt channels in injection molding manifold systems and extrusion dies

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Natural rheologic balancing is being state-of-the-art in developing polymer melt channels for extrusion dies or hot runners in injection molding. For this, flow path lengths and channel diameters in a manifold system are defined identical on all paths and thus those channels are considered to be identical in pressure drop and consequently in cavity filling respectively in mass flow distribution. In addition, systems designed in this way are suspected to be independent of the operating point. However, the changes in direction of the melt flow due to the curvatures of the melt channels are nearly always completely neglected. Depending on the space available or the manifold system in general, the conditions can be such that melt sub-channels differ completely from one another regarding their overall course. Despite natural rheological balancing by means of flow paths of equal length and diameter, unequal mass flow distributions or cavity fillings occur in such geometries. The aim of this paper is to investigate the influence of channel curvature on the balancing of melt channels. For this purpose, the curvature collectives along the neutral fibers of the melt sub-channels are evaluated and considered during the design process. The overall objective is a more specific balancing with respect to the mass flow distribution. The results indicate the necessity of considering the channels' curvature in the designing process in order to achieve a homogeneous melt distribution.

Oral (S11-317, Time: Wednesday 14:50, Room: Empa)

The influence of material properties and process parameters on energy consumption in the single-screw extrusion of PVC tubes

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Extrusion is one of the most widely used but energy-intensive processes for shaping polymers. It is thus fundamental to understand which are the process conditions that enable a cost-efficient operation of the extruder machines, especially given the high increase in the electricity cost in recent years. However, the energy required by the process is highly affected by both system variables and material properties in a complex manner. The aim of this work is thus to further investigate the correlation between the process settings and the material properties and the extrusion demanded energy. An extensive experimental campaign was conducted, testing different type of polyvinyl-chloride (PVC). The active, reactive and apparent power, as well as the power factor were recorded for both the motor of the extruder and the entire machine during continuous single-screw extrusion for different process conditions. A regression model was developed to correlate the specific energy consumption with both the material properties and the process settings, thus providing a useful tool for the estimation of the cost of the final manufactured product.

Oral (S11-338, Time: Wednesday 15:10, Room: Empa)

A Tailored Modelling Approach to Predict the Three-Dimensional Flow of Polymer Melts in Helical Screw Channels

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High-performance screws such as barrier, double-wave, and energy-transfer screws are becoming more and more popular for the polymer industry to meet the steadily increasing requirements on productivity and melt quality in single screw extrusion. A special feature of these screws are regions with deep channels and multiple flights, where the channel curvature and the flight flanks affect the melt flow significantly. Hence, the traditional flat plate model, which neglects both effects, becomes invalid in these regions. Aiming for a more realistic description of the local flow conditions, we present a novel calculation routine, in which the balance and constitutive equations for creeping flows are formulated in a helical reference frame, scaled to a dimensionless representation, and solved numerically using the finite element method. The modified equations include the three-dimensional flow pattern inside a confined helical screw channel, coupled with the shear thinning behaviour of the melt using a power law model. Focusing on fully developed flow conditions in channels of constant dimensions allows to reduce the flow domain to short segments of unit length. Furthermore, the flow is treated as isothermal and incompressible. As a result, computation becomes faster and requires less memory compared to a full computational fluid dynamics (CFD) simulation. This makes the routine particularly suitable for comprehensive parametric design studies, in which the influencing factors may be varied systematically in a wide range and in numerous levels to derive precise process models using data processing algorithms. By predicting the dimensionless flow rate and viscous dissipation rate for selected industrial use cases, we found a pronounced influence of curvature and aspect ratio in deep channel sections. Additional full CFD simulations support the plausibility of the modelling approach.

Oral (S11-358, Time: Wednesday 16:00, Room: Empa)

Coupling Fiber Orientation and Polymer Flow in Molding Simulations of Composites

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Flow of composite polymer melts which contain fiber reinforcements are known to exhibit anisotropic rheology behavior dependent on the orientation of the fibers. In this work, the anisotropic effect of fiber orientation on the polymer rheology has been incorporated into a numerical simulation program of molding processes of fiber-reinforced polymer composites to fully couple the analysis of fiber orientation and polymer flow. As the measurements of the material data were performed on the composite, the isotropic viscosity of the matrix resin is first decomposed from the measured composite viscosity. The model parameter called the Particle Number, which controls the strength of the fiber orientation effect on the polymer rheology, is determined by the aspect ratio and the volumetric fraction of fibers as well as fiber orientation and varies during the molding process and throughout the part. The validation against experimental data shows that the fully coupled analysis achieves an improvement in the predictions of injection pressure, filling pattern, and fiber orientation compared to a traditional analysis with polymer rheology which does not depend on fiber orientation.

Oral (S11-397, Time: Wednesday 12:00, Room: Empa)

Response of Shear Thickening Fluids Impregnated fabrics for Soft Body Armor

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The impact resistant of high tenacity fabrics is improved by using shear thickening fluid (STF).In presence of STF, the Kevlar fabric is found to prevent the bullet penetration more effectively. To understand the mechanism of the prevention of projectile penetration, quantum of work is very less. There are a good number of experimental works showing the effect of STF on the prevention of the projectile penetration through its composite with high tenacity fiber. This requires the development of simulation methodology to reduce the number of experiments. In the continuum approach of simulation, a single layer fabric–STF system has been developed. One needs to develop continuum simulation for multilayer system along with STF. Here, yarn pullout simulation and ballistic impact simulation to have a better understanding of how the energy is absorbed by the fabric will be discuss. A comparison will be shown between a neat fabric and STF impregnated fabric using simulations. The simulations are done in LS Dyna software which provides explicit finite element analysis. The results are in coordination with the hypothesis that with addition of STF in the Kevlar fabric, improves its ability of protection against ballistic impact with respect to non-STF Kevlar. For a single layer of fabric, the STF fabric provides less penetration than non STF fabric, i.e. the velocity decrease in STF-fabric is more than that in the non-STF fabric. Multilayered ballistic impact simulations are done to find the number of layers at different coefficients. Results showed that as the friction coefficient increases, the number of layers required decreases. The STF Kevlar fabric has a range in higher friction coefficient and thus requires less number of layers than normal Kevlar fabric as observed in simulations. It is concluded that with STF, the present body protection armor is improved and thereby increasing the safety of the person.

Oral (S11-398, Time: Wednesday 16:20, Room: Empa)

Numerical Simulation of the Pressure, Power and Mixing Characteristics of Twin Screw and Planetary Roller Extruders

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Extruders are applied in several industries to plasticize, convey and mix highly viscous fluids. The extruder screws are characterized by their pressure and power characteristics which allocates them to various processing tasks. Furthermore, the mixing efficiency of a screw configuration is particularly relevant when designing extrusion processes. However, the dispersing and homogenizing represents a challenge due to the sometimes high viscosity ratios between the continuous and discontinuous phase as well as the generally high viscosity of the media that is processed. In industrial practice, co-rotating twin screw extruders are mainly used for these demanding mixing tasks, but special designs such as the planetary roller extruder are also established. In the context of this work, a method is presented to predict the process behavior of intermeshing screw machines by numerical calculation of dimensionless parameters for the pressure, power and mixing characteristics. Therefore, three-dimensional flow simulations were applied using the finite element method. A mesh superposition technique was implemented to take into account the rotation of the intermeshing screws and the resulting variation of the flow geometry. The potential of the proposed simulation method is confirmed on the basis of a validation with experimental data for conveying and kneading elements of the twin-screw extruder. Based on this, process parameters are determined for various configurations of the planetary roller extruder, which were not previously available for this special type of extruder. Finally, the compilation of dimensionless parameters for the pressure, power and mixing characteristics of both extruder types enables a comparison of the processing behavior between them. Furthermore, different screw configurations are considered, facilitating a systematic and efficient design of extrusion processes.

Oral (S11-401, Time: Wednesday 16:40, Room: Empa)

Validation of a multiscale simulation for precise warpage prediction of injection moulded semi-crystalline parts

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The simulation of the injection moulding process of semi-crystalline thermoplastics is widely used and the software is commercially available. Typical use cases for process simulations are melt flow optimisation, tool design and finding process parameter sets for the production. In recent years, injection moulding simulations also provide the prediction of shrinkage and warpage. These simulations, however, neglect the variations in local mechanical properties caused by inhomogeneous solidification and the resulting locally different microscopic structure. In order to account for the locally inhomogeneous solidification and the resulting local microstructure, and hence to improve the precision of the warpage prediction, a multiscale simulation chain is developed and presented here. The multi-scale simulation chain predicts the shrinkage on the part scale taking into account the mechanical property distribution, which is derived based on the local microstructure using a two-stage numerical homogenisation scheme. The numerical homogenisation is able to consider the local degree of crystallisation, which is considerably reduced within the layers near the surface of the component, resulting in a reduction of the mechanical properties. To show the applicability and precision of the multiscale simulation chain, an experimental validation is presented using injection moulding plates, of which the geometry is measured with the GOM system from Carl Zeiss GmbH and compared with the numerical results.

Oral (S11-442, Time: Thursday 10:40, Room: Empa)

Coat hanger die design through constant shear rate flow network method and adaptive deflection analysis

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A design methodology for polymer coat hanger sheeting extrusion dies is presented in this paper. This methodology integrates a coupled fluid flow and die deflection analysis. The main aim of extrusion die design is to create a fluid distribution manifold facilitating a uniform flow at the die exit. The flow of highly viscous polymers in narrow slits of extrusion dies causes significant pressure drops, which in turn generate a nontrivial die lip deflection. In the proposed design methodology, the die lip is partitioned into strips of Euler beams for the die deflection analysis subject to a fluid pressure obtained from the fluid flow analysis. The fluid body in the fluid analysis was updated using the deflection results while the applied pressure in the deflection analysis was updated using the fluid analysis results. A limitation is set for maximum allowable lip deflection by increasing the die body thickness. The effects of polymer melt flow rate and die body thickness were studied in connection with the deflection. Computational fluid dynamics simulations were performed as a comparison of various designs. The proposed methodology created a die design which exhibits 33.6% improvement in uniformity index compared to a previous design where the die body deflection is not considered in the design.

Oral (S11-447, Time: Thursday 11:00, Room: Empa)

Optimizing the mixing quality of a single screw extruder with CFD simulations

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Polymer mixing with a single screw extruder is a common process in industry. The design of a screw geometry is essential to obtain the right mixing quality in an extruder. However, the screw geometry is often not yet fully optimized. This study is aimed at comparing mix quality of several common mixers quantitatively with computational fluid dynamics (CFD) simulations. The mixing quality was determined with the distribution of tracer particles (simulations) and a tracer fluid (experiments). These distributions were quantified with Shannon entropy calculations and residence time distribution (RTD). The simulations and experiments are in agreement with each other. Therefore these CFD simulations can be used as a validation tool for selecting mixing elements. Furthermore these simulations were used to determine which of the studied mixing sections is the most optimal mixer.

Oral (S11-450, Time: Thursday 11:20, Room: Empa)

Investigation of the Particle Distribution in Injection Molding of Mineral-Filled Polyamide Using CFD Simulation

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The appearance of the interior and exterior of modern cars is characterized by a variety of chromed parts. Due to the lightweight construction requirements to reduce fuel consumption or to maximize the range of electric vehicles, the use of metallic parts is adversarial. Typically, these parts are injection-molded parts made of ABS or PC/ABS and subsequently electroplated. If the requirements on mechanical properties increase, mineral-filled polyamide 6 (PA6) is often used. The particles and their distribution are primarily responsible for the surface quality of the electroplated parts. Unfortunately, the rejection rate of electroplated PA6 is up to 30 %. Therefore, the particle distribution in the boundary layer was investigated in this paper for different operating points carrying out computational fluid dynamics (CFD) simulations using ANSYS Fluent. The simulations were executed for a microscopic section in the boundary layer and the boundary conditions were derived from a macroscopic simulation assuming continuous material properties using Sigmasoft. Furthermore, the particle distribution was set via user defined function (UDF). Significant process parameters for the particle distribution in the surface layer were determined and correlated with surface parameters from real experiments.

Oral (S11-455, Time: Thursday 11:40, Room: Empa)

Finite Element modelling of solid-phase tube die-drawing: production of Shape Memory Polymer for bone-fixation devices

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Solid-phase polymer processing, such as die-drawing, is an established technique that produces highly oriented polymer product in large section. In the solid-phase tube die-drawing process, a hollow cylindrical polymer billet is pulled through a fixed die and mandrel set, at elevated temperature, to produce a stretch in the axial or hoop direction (or both). As the polymer tube cools beyond the die-exit, the induced orientation of the polymer chains is locked in. The degree of orientation depends on the drawing temperature and speed, die/mandrel geometry, and the material characteristics. For semicrystalline polymers, such and PP, tube die-drawing is used to permanently enhance the mechanical properties of the polymer (e.g., to manufacture pressure pipes). For an amorphous polymer, such as PETG, it is instead used to induce a large Shape Memory effect. Here, heat triggers a section of the die-drawn tube to revert back to its original (non-die-drawn) shape. One major application is in orthopaedic fixation devices, where body-temperature triggers the shape-change, generating the required fixation-force shortly after surgical implantation. Here we model the tube-drawing process within the commercial Finite Element software Abaqus/Explicit. The simulations are not trivial as they involve large deformations and complex surface interactions. To properly represent the strain-hardening and strain-rate sensitivity of the deforming polymer, a rate-dependent elastic-plastic material was used. This was parameterised using data from tensile (free drawing) tests conducted to large strains. The Finite Element predictions agree well with tube drawing experiments in terms of geometry, stretch and drawing force. The model elucidates the stress/strain fields within the polymer, allows us to explore the effect of different die/mandrel geometries, and helps us to optimise the process.

Oral (S11-459, Time: Thursday 12:00, Room: Empa)

Experimental validation of a simulation model for the non-return valve performance during the injection phase of an injection molding cycle

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A CFD model which simulates the flow of a polymer melt during an injection molding cycle is developed using the simulation software Ansys Fluent. With this model the closing behavior of a ring-type non-return valve during the injection phase is investigated. The flow conditions in the barrel from the metering zone of the screw to the nozzle tip are considered based on a viscous, compressible and non-isothermal polymer melt flow. In a quasi-static approach, the velocity of the non-return valve sliding ring is determined based on the equilibrium of forces acting on the sliding ring. The closing behavior of the non-return valve is determined by analyzing the closing movement of the ring and the mass which is pushed back into the screw chamber. The simulation model is validated by performing injection molding experiments for two thermoplastic polymers (PP and HDPE) with varying injection flow rates. Experimental data for validation is obtained by three melt pressure sensors which are installed in the barrel of the injection molding machine. The accuracy of the simulation model is analyzed by comparing the melt pressure occurring behind the non-return valve in simulation and experiment. The simulation results are in good agreement with the experimental data for the materials and setting parameters considered.

Oral (S11-462, Time: Thursday 14:10, Room: Empa)

Modeling the pressure-throughput behavior of double wave zones by means of network analysis and heuristic melt-conveying models

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The aim of the present work is to study the extrusion performance of double wave screws and present a modeling approach based on network analysis for calculating the pressure-/throughput behavior. Wave and energy-transfer screws exhibit wave-like channel depth profiles (valley/crest sequences and flight undercuts) with the purpose of inducing solid bed break-up, dispersion of solid agglomerates and enhancing dispersive and distributive mixing by creating extensional and wedge flows. Experiments were performed on a well-instrumented 35/34D and 60/33D single-screw extruder with barrier and wave screws, varying materials, and screw speeds. The replacement of the barrier zone with a combination of a compression and a double wave zone resulted in a considerable performance improvement. Furthermore, a simulation routine based on network analysis is presented to calculate the non-isothermal melt dominant pressure-/throughput behavior of wave and energy-transfer zones. In combination with recently developed melt conveying and dissipation models the simulation results are in excellent accordance with the experimental results.

Oral (S11-475, Time: Thursday 14:30, Room: Empa)

On the prediction of parameters for the glass-rubber model for polyethylene terephthalate (PET) based on observed data in the injection stretch blow moulding (ISBM) process

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The ISBM process is a common production process for bottles and containers made from polyethylene terephthalate (PET). The forming of these containers begins with preforms, that are heated and subsequently formed by stretching and blowing. Process settings, material properties as well as the geometric shape of the preform define the deformation behaviour. Finite Element Analysis (FEA) is a common tool for virtual prototyping, but meaningful predictions require a meaningful numerical representation of the process. The ISBM process is not only governed by process parameters, but also by material behaviour. If different material grades are available, this should be considered for virtual prototypes. Consequently, a suitable material model and the identification of its material parameters are challenging but also key to success for FEA. In this contribution, new methods for automating both the experiments and the calibration of the material model are presented. Additionally, the feasibility of establishing a prediction model of material parameters based on observed process data is analysed by employing an artificial neural network. The glass-rubber model firstly introduced by Buckley et al. [1] has been used and extended several times and has proven its suitability for representation of PET in stretch blow (moulding) processes. Consequently, the glass-rubber model (in its adoption from Zimmer [2]) is also suitable for this contribution to illustrate and evaluate the new methods for experimental calibration. The validity of the prediction model is examined by a sensitivity and distinctiveness analysis, as process parameters like the air pressure and the initial temperature are influencing the shape history as well. 1: https://doi.org/10.1016/0032-3861(95)99429-X 2: http://dx.doi.org/10.17877/DE290R-7923

Oral (S11-479, Time: Thursday 14:50, Room: Empa)

Discrepancies in Injection Molding Simulation and Reality: An Analysis

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Numerical process simulation is an established development tool for the design of injection molding processes. However, commercially available simulation software focuses primarily on the filling process in the mold and makes various simplifications. Due to these simplifications, the non-consideration of disturbing influences, as well as the deviating boundary conditions within the simulation, the result of the real process often deviates from the simulation result. To enable an injection moulding simulation that is closer to reality, the aim of this work was to carry out an analysis of the current discrepancies between a real injection moulding process and the simulation of this process. For this purpose, a design of experiments was carried out in the simulation and compared with the corresponding injection molding experiments. Relevant process variables such as the specific pressure or the cavity pressure as well as the parts weight were used as target variables for the analysis to compare the weight from the experiments with the estimated weight from the simulation.

Oral (S11-480, Time: Thursday 15:10, Room: Empa)

Reducing the discrepancies in Injection Molding Simulation and Reality

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Due to the ever-increasing demands of governments, companies and the general public for resource efficiency and reduction of the CO2 footprint in industrial processes, numerical process simulation is becoming an increasingly important part of the design of injection molding processes. A well-known and major problem is the existing discrepancy between real processes and the representation of these processes in the simulation. The objective of this work was to simplify the complex simulation models and reduce them to the relevant process parameters (melt temperature, packing pressure, packing time, injection speed and mold temperature). For this purpose, mathematical conversion models were used to set up equations for the simulation and real process that contains the relevant process variables and were expanded with an adjustment factor. These equations have the part weight as the target variable in the simulation and in the experiments. The equations were applied to the selected tool-material combination of the experiments in order to reduce the differences between simulation and reality. From this, a conversion model was developed that can convert the simulation results into machine-usable parameters. These adapted parameters were applied to a design of experiments. Subsequently, it was checked whether the discrepancy between reality and simulation could be reduced. Remaining differences were then analyzed and it was checked whether all relevant parameters had been converted and whether it was necessary to consider further simulation parameters. It was found that there is still a discrepancy between the simulation and reality, but that it is possible to reduce this deviation. It is necessary to adjust the process parameters and use data from the machine (injection profiles, etc.) to achieve a realistic representation of the process in the simulation.

Oral (S11-519, Time: Thursday 16:00, Room: Empa)

Modeling Large Amplitude Oscillatory Shear Deformation in Semi-crystalline High-Density Polyethylene

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Plastic polymer recycling is a major global challenge. Two key processes have paved the way for plastic polymer recycling: mechanical recycling and chemical recycling. The mechanical recycling method is widely appreciated due to its environmental and economic impacts. Recently, Yao et al., in their mechanical recycling process experiments, optimized the mechanical properties of recycled high-density polyethylene (HDPE) near their virgin HDPE material and proved that the recycled plastics are physically degraded. However, the microstructure information is missing so, molecular dynamics (MD) simulations and non-equilibrium molecular dynamics (NEMD) simulations are performed, and subsequently, their corresponding mechanical properties are evaluated to correlate with the aforementioned experimental observations. The experimental conditions are expressed in MD simulations by performing static and dynamic deformations. In NEMD, for the dynamic sinusoidal deformation, the SLLOD equations of motions with Lees-Edwards boundary conditions were used to mimic the large amplitude oscillatory shear (LAOS). Three different deformation rates (100%, 500%, and 1000%) are applied in both static as well as dynamic deformations, to see their effect on chain orientation in the semi-crystalline structure at 300K. This molecular information study could serve as a guide in principle to optimize or design new polymers for industrial or economical aspects. The characteristic model densities (ρ in g.cm-3) ranges are 0.90-0.94, which are the averages of the amorphous and crystalline regions, evidence for the formation of the semicrystalline structure in the recrystallization process. The lamellar thickness (~70-100 Å) is marginally underestimated than the experimental values (110-167 Å). The lamellar formation in all recrystallization methods is random and the mechanical properties due to the morphological changes are anisotropic representing a heterogeneous morphology formation in the recrystallization. Consequently, the shear treatment followed by relaxation has shown improved mechanical properties, this is consistent with the experimental observation obtained from the experimental study.

Oral (S11-537, Time: Thursday 16:20, Room: Empa)

Fatigue life analysis of POM gears with transient material modelling

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The today's standard calculation methods for investigating the load carrying capacity of polymer gears (such as VDI 2736) are based on the same assumptions as for steel gears. Due to strongly varying material properties of polymers regarding stiffness level, nonlinearity and rate dependency, the predicted lifetimes of polymer gears are inaccurate. In the current study a rate dependent nonlinear viscoplastic finite element (FE) modelling of POM allows quantifying the material influences not considered by standard assumptions for metal parts. By deploying such a nonlinear material model for POM, a POM-steel gear pairing is investigated and additionally validated on a gear test rig. An accelerated approach for a repeated transient FE modelling of the gear meshing process makes it possible to investigate critical stress and strain states over consecutive cycles. It turns out that a cyclic increase of (plastic) strains occurs in the polymer gear teeth, thus the angular error of the corresponding steel gear increases in addition. Through an extrapolation of this angular error arbitrary subsequent cycles can be simulated in reasonable time. In this way the local stress and stain states of critical failure causing cycles can be analysed. This forms the basis for a fatigue life analysis of POM gears. In combination with stress- and strain energy-based failure criteria the admissible number of cycles of POM gears for various loading parameters is predicted and validated against experimental results obtained from the gear test rig.

Oral (S11-539, Time: Thursday 16:40, Room: Empa)

Static mixers producible by additive manufacturing: Operating point specific geometries through automatic optimisation

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In the extrusion of plastics, the thermal and material homogeneity of the plastic melt at the die entry are of high importance for extrudate quality. While static mixers are widely used to improve the melt homogeneity, previous attempts at optimisation for reduced pressure loss and improved mixing had to be performed by hand and based on human experience, limiting the degrees of freedom and efficiency. Additive manufacturing enables new degrees of freedom in the design of static mixers, for example for the integration of temperature control channels. A new automatic optimisation method based on the open source software OpenFOAM was developed. Using immersed boundary methods, new target functions in the pre-existing routine adjointShapeOptimizationFoam and an additional algorithm checking the suitability for additive manufacturing and repairing the geometry during run-time were developed. This allows the low-cost design and manufacture of individualised static mixers. Each optimisation is performed for a specific operating point, characterised by the throughput and material as well as the specific temperature inhomogeneities at the mixer inlet. The new algorithm is used to optimise an existing static mixer based on an X-type geometry with integrated oil channels, maximising the heat exchange between oil and melt. Experimental validation shows that with this optimisation algorithm, a pressure loss reduction of 10% could be achieved. The core melt temperature was reduced by 6 °C, improving the thermal homogenisation as well. While the main advantage of this method is the rapid optimisation for a specific operating point, the trials also showed positive results in off-design operating points. A quantitative analysis of the operating point specific optimised mixing geometries is presented, where the performance characteristics of the static mixer optimised for one operating point are compared to the performance of the same mixer geometry in a different operating point.

Oral (S11-556, Time: Friday 10:40, Room: Empa)

Study of Residence time and Filling ratio inside Counter-rotating continuous mixer using Partially filled flow simulation

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Twin-screw extruders and continuous mixers are used in plastic molding process to improve the quality, homogeneity and transportability of plastic materials. The control and optimization of mixing state are expected not only to enhance the added value of plastic products but also to improve energy efficiency of mixers. Therefore, rotor shapes and operating conditions that make large influence on mixing conditions have long been the subject of research. To optimize the mixing process, it is important to reveal the behavior of the melt plastic inside the mixers. Although numerical simulation has been used to reproduce the inside of mixers, there are few cases in which the partially filled state inside a mixer is simulated. This is because the flow path geometry of the mixer is complicated, and it is difficult to capture unsteady shapes of the free surface. In this study, the Element-free Galerkin method, a type of mesh-free simulation technique, was used to reproduce the partially filled condition inside a mixer, and the residence time distribution, which is known to have a significant effect on the mixing performance, was investigated. Partially filled flow simulation of a counter-rotating continuous mixer was conducted to investigate the effects of rotor shapes and operating conditions on the filling ratio and residence time distribution. As a result, although the overall filling ratio varied with rotor shapes and operating conditions, it was found that there was no local change in filling ratio, and the behavior of the filling ratio along to the flow direction was identical for each rotor shape. In addition, the results of marker particle tracking suggest that the experimental estimates of the filling ratio may be modified, and that the melt plastic flow behavior, residence time distribution, and filling state are different when the average residence time is similar.

Oral (S11-569, Time: Friday 11:00, Room: Empa)

Effect of a shear flow on the rupture of a thin polymer film

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Nanolayer coextrusion allows processing films composed of thousands of layers of alternating polymers with individual layer thicknesses below 100 nm [1]. It has been observed that below a critical layer thickness (around 10 nm for PS/PMMA nanolayered systems), the layers tend to lose their integrity, i.e. they break spontaneously during the process [2]. These layer break-ups have been attributed to small interfacial perturbations that are amplified by van der Waals forces [2]. The objective of this work is to give new insight into the stability of thin polymer films under shear, in order to pave the way to a better control of the nanolayer coextrusion process. To do so, a first 2-D finite-difference numerical scheme for the resolution of the thin film equation was set up taking into account capillary and van der Waals (vdW) forces. The results suggest that shearing during the flow in process acts as a stabilizing factor and tends to delay or suppress the appearance of layer breakups [3]. In the 3-D system, it seems that unidirectional shear only leads to a minor delay in the rupture time. While perturbations are suppressed in the direction of the shear, they grow freely in the transverse direction. References. 1. M. Ponting, A. Hiltner, E. Baer Macromol. Symp. 2010, 294, 19 2. A. Bironeau, T. Salez, G. Miquelard-Garnier, C. Sollogoub, Macromolecules, 2017, 50, 4064 3. K. Kadri, J. Peixinho, T. Salez, G. Miquelard-Garnier, C. Sollogoub, Polymer, 2021, 235, 124283

Oral (S11-595, Time: Friday 11:20, Room: Empa)

Validated Modelling of Complex Geometry Dies for Extrusion Foaming of Starch-Based Mixtures

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Biobased materials offer a good alternative to the widespread use of fossil-based plastic. Foamed plastics are used in a variety of industries such as packaging, automotive, marine, and aerospace may result in the sea, soil, and air pollution due to insufficient waste management systems. One of the most abundant biobased materials is starch derived from natural sources such as potato, tapioca, and corn starch. This class of materials can be handled as a thermoplastic material and employed in the foaming extrusion process by adding additives (such as water and glycerol). Starch foams as a biobased, and biodegradable product could be a promising alternative to expandable polystyrene (EPS), or expanded polypropylene (EPP). However, their application remains difficult due to issues related to moisture content and irregular swelling at the die exit, both of which affect the final product properties and well-defined shape of an extrudate. Typically, to obtain the required shape of the extrudate, a die with an approximate shape is created and iteratively adjusted based on the quality of the extrusion until the shape matches the desired one, which is still considered an art. The aim of this work is to develop and validate a fluid dynamics model for starch-based mixtures flowing in a die and relate the shape of the foamed extrudate cross-section with the die geometry and flow conditions to reduce trial and error iterations. The pressure drop and velocity profiles are evaluated and compared to experimental data. Moreover, the die swell phenomenon is investigated by changing the land length of the dies to tune the residual normal stresses at the die exit for controllable dimensional accuracy.

Oral (S11-597, Time: Tuesday 15:10, Room: Empa)

Developing HME-Based Drug Products Using QbD and Emerging Science

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Hot-melt extrusion (HME) is a continuous manufacturing process that uses co-rotating intermeshing twin-screw extruders. The process primarily produces amorphous solid dispersions of poorly soluble active pharmaceutical ingredients when used in pharmaceutical applications. Being a continuous manufacturing technology, HME-based drug development typically requires prohibitively high amounts of API for successful formulation screening and early process development. Effectively solving the challenge of formulation development, scale-up, and transfer to production presents a critical challenge for the pharmaceutical industry, especially for HME. Our group has focused on developing scientific tools that allow for fast and minimum-risk development of HME-based formulations using several advanced tools. These include advanced material screening, small-scale formulation test beds, the design of small-scale processes, and the scale-up to GMP production of clinical batches. Since, to this date, most of the process setup and scale-up activities are performed experimentally and empirically, one of the goals of our group was to create in silico tools for a rational, science-based process setup and scale-up while addressing other important aspects, such as an API degradation and overall product quality. Formulation and process development will be discussed and analyzed. A detailed analysis of the different extruder elements and equipment scales, based on the results of detailed smoothed particle hydrodynamics (SPH) simulations, will be discussed. Our work includes computing the shear rate distribution, viscosity distribution, melt temperature distribution, local residence time distributions, pressure, and power characteristics of conveying, kneading, and mixing elements. A detailed process analysis based on mechanistic 1D simulations will be performed, comparing various configurations and equipment scales in the context of scalability and resulting process quality.

Oral (S11-620, Time: Friday 11:40, Room: Empa)

Digital twin of the profile extrusion process

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According to the current state of the art, the profile extrusion process is often optimized by the “trial and error” method. This is on one hand the case for the design of extrusion dies, but especially for the cooling section including the calibration tools. The biggest disadvantage of the "trial and error" method is that each optimization iteration is time-consuming and, moreover, often involves high financial outlays. These can manifest themselves in form of new tools, machine and working hours, assembly and cleaning efforts and eventually the production downtime. One sophisticated way to avoid these problems is to be able to predict the behavior of the extrusion line or the profile, respectively to detect problems ahead of time by using numeric calculation tools. For many applications, this is already state of the art; most injection molds, for example, are designed and configured with the aid of simulation. In profile extrusion, the design of extrusion dies and downstream are still based on experience and costly commissioning and optimization iterations. For several years now, the Institute of Materials Technology and Plastics Processing IWK has been conducting intensive research into ways of virtually simulating the extrusion process from the extruder through flow simulations in the die tool to the finished and calibrated profile. The holistic approach and interrelated consideration of the entire production line, including the mutual influences in a simulation, is unique to date. Ultimately, the profile geometry achieved, including shrinkage and warpage, can be virtually reproduced and thus predicted for given process parameters and tools. As a result, manufacturers can significantly increase the efficiency of their development process and improve the quality of the product.

Oral (S11-672, Time: Friday 12:00, Room: Empa)

Product lifecycle management in injection molding

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Initial situation Everyone is talking about digitalization, the smart factory and Industry 4.0, and these topics are also becoming increasingly prevalent in the field of injection molding. The topic of product lifecycle management (PLM) is becoming increasingly important. In theory, PLM records important product-related master data that accumulates during the life cycle of a product. In practice, however, it has become apparent that, particularly in the development of plastic components realized by injection molding, only a fraction of the relevant data from process-specific pre-development and subsequent production can be recorded. Data consistency within PLM By developing a database-based software solution, all important tasks are to be mapped and the relevant data generated in the process is to be recorded and made centrally available. These are for example: Component design (material selection, article specification and drawing); Process design (part number and process parameter determination, tool design); Tool sampling (process settings, sensor data, part dimensions); Production (quality control, setting data variations, tool maintenance) Software demonstrator OPTx In cooperation with the Institute for Software (IFS), a first software demonstrator (OPTx) has been developed, which already maps many of the complex interrelationships. In addition to intuitive operation, OPTx is characterized in particular by the consideration of different versioning, such as from the article specification as well as the possibility of continuous and holistic computer-aided process optimization.

Oral (S11-680, Time: Tuesday 14:10, Room: Empa)

Elasto-vsicoplastic modelling of polystyrene in the rubbery state

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In this study, a micromechanical inspired elasto-viscoplastic modelling approach is developed to model the thermomechanical behaviour of a high impact polystyrene (HIPS) in the rubbery state. The proposed model consists of a nonlinear generalized Maxwell model in series with a second dissipative element. Each of the Maxwell elements incorporates an elastic eight chain spring in series with a dashpot based on a hyperbolical law, which is a simplification of the double kink model of Argon. While the generalized Maxwell model considers the relaxation spectra for glassy-like polymers, the additional dissipative element is a small modification of the approach of Bergström and thus considers the reptation based energy dissipation. The model is designed to simplify to the three-dimensional generalized linear Maxwell model in the limit of small deformation. Furthermore, the viscous modelling is temperature activated and follows the Williams-Landel-Ferry (WLF) approach in the limit of linear viscoelasticity. The simulation is implemented into Simscape, a framework of MATLAB to simulate physical systems based on the principle of energy flow. The implementation provides fast and easy modelling, calibration and simulation of any rheological experiment. However, due to the low numerical effort, even without advanced computer hardware, modelling, calibration and simulation is not as time-consuming as with finite element or finite volume method. The model is fitted by oscillatory shear experiments and uniaxial elongational testing. To validate the performance of the model, it is implemented into a VUMAT for finite element simulation of a thermoforming process with Abaqus/Explicit.

Poster (S11-018, Time: Thursday 17:00, Room: Foyer)

Computer simulation of stretch blow molding and structural analysis of PET bottle and comparison with experiments

Lyu Min-Young[1], Choi Dong-Hae[1]

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There is increasing demand to reduce the weight of PET bottles to decrease plastic waste. To manufacturing weight reduced PET bottles, research on the stretch blow molding process is essential. In this study, experiments and computer simulations were performed on the stretch blow molding process. The computer simulations were performed using non-isothermal model. Experimental and simulation results were compared. Structural simulation of PET bottle was performed using the results of stretch blow molding which was a blow-structure coulped simulation. The simulations shown in this study were useful for designing and manufacturing improved PET bottles.

Poster (S11-295, Time: Thursday 17:00, Room: Foyer)

Capillary Flow Behavior of a Gum Acrylonitrile Butadiene Rubber

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Rubber compounds are known to be very shear-thinning and possess a strong degree of viscoelasticity. A series of rheological measurements for a Gum Acrylonitrile Butadiene (NBR) rubber are carried out using a moving die rheometer for the characterization of storage and loss moduli and a high-pressure capillary rheometer in order to study the effect of different die characteristics and temperature. The flow simulations are performed for axisymmetric (capillary dies) extrusion. The flow simulations are performed using a viscous (Cross) and an integral (K-BKZ) rheological constitutive equation (the PSM model). The effect of capillary die angle ranging from 180o to 60o is examined and no vortex activity is found in a wide range of apparent shear rates (or flow rates). Some conclusions on the pressure drop in the capillaries are drawn regarding the ability of the integral K-BKZ model to capture the viscoelastic behavior of NBR rubber. These values highlighted the importance of viscoelastic modeling to correctly predict pressure drops of an unfilled gum rubber especially in contraction flow-dominated geometries.

Poster (S11-449, Time: Thursday 17:00, Room: Foyer)

Computational fluid dynamics analyses of ultrasonically aided extrusion

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The use of circular materials (recycled polymers) within the field of extrusion is difficult due their often-inconsistent viscosity related to variations in composition and molecular weight of the recycled polymers. A constant adaptation of the extrusion process is needed to compensate for these changes in viscosity. Plastic profile production with recycled polymers can be made more efficient and enhanced with ultrasonic vibrations to manage the extrusion die pressure. Extrusion dies often have complex geometries and therefore wave propagation inside the die is also complex. Ultrasonic waves in a non-Newtonian polymer melt were modelled with Ansys CFD (computational fluid dynamics). It was found that both shear waves and pressure waves induce high shear rates resulting in shear thinning and therefore a reduction of die pressure. Furthermore extrusion experiments were performed. The die pressure reduces significantly (up to 34%) and rather quickly (1 bar/s). This short response time makes ultrasonically aided extrusion a promising tool for managing die pressure with recycled polymer extrusion.

Poster (S11-543, Time: Thursday 17:00, Room: Foyer)

Cavity balancing optimization of injection moulds via three-dimensional, shape-morphing flow leader generation.

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Better part quality can be achieved by improving the cavity balance in injection moulds. However, identifying an optimal thickness distribution that alters the melt front shape such that it reaches the mould's boundary at exactly the same time, continues to be a major challenge. Traditional parametric CAD approach allows for quick and easy geometry adjustments. Yet, they require human input, which can limit the design space greatly. On the other hand, shape-morphing methods require sophisticated algorithms for generating suitable nodal displacement fields. Nonetheless, highly complex geometries can be achieved. In this work a shape-morphing method for balancing the filling of the mould cavity via fill time dependent nodal displacement field is presented. The method is based on the work of Y.C. Lam and L.W. Seow (2000) and consists mainly of three steps. First, the longest flow path (LFP) between the gate and the last filled node is determined using the hill-climbing algorithm. Secondly, the coordinates of the LFP nodes are updated based on a fill time-dependent rule. This fill time data is obtained from an injection moulding (IM) simulation of the unaltered initial geometry. Lastly, the coordinates of neighbouring nodes to the LFP are updated based on a quadratic factor of the distance between neighbouring node and LFP. All updated node coordinates obey a predefined maximum thickness constraint in order to avoid mass accumulations. Initial results show that the shape-morphing method is capable of improving the cavity balancing in IM moulds.

Poster (S11-683, Time: Thursday 17:00, Room: Foyer)

Shear-Induced Dynamical Heterogeneities in Soft Particle Glasses

Khabaz Fardin[1], Pable Hrishikesh[1]

[1]University of Akron, Ohio, USA

Soft particle glasses (SPG), which are jammed beyond the random close-packing fraction of equivalent hard spheres, show rich rheology under shear flow. The softness of particles allows them to compress to volume fractions larger than the random close-packing of the equivalent hard-spheres and form flat facets at the contact between particles. In this regime, the thermal energy contribution becomes minor, and the contact forces are the dominant source of dynamics and govern the morphology and macroscopic rheology. These fluids demonstrate yield stress behavior; they flow according to the Herschel-Bulkley (HB) relationship. We will use our particle dynamics numerical method to disentangle the interrelation between the softness, size distribution, volume fraction of the particles, and strength of the flow on the microscopic dynamics and rheology of SPGs using computational techniques. A direct relationship between the microscopic dynamics and macroscopic properties of these suspensions will be established using an efficient computational method in shear flow. The shear flow curves and trajectories of the suspensions will be analyzed to identify domains with heterogenous motion and link the length scale of these domains with the stress-strain behavior at low and high shear rates. Furthermore, the simulations will provide a direct relationship between the stress distribution in the suspensions and their nonaffine dynamics.

Poster (S11-716, Time: Thursday 17:00, Room: Foyer)

Extrusion Blow Molding Simulator: Towards the Digital Transformation of the Packaging Industry

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In the packing industry extrusion blow molding is an extremely important manufacturing process, where the need to optimize product development processes has motivated the use of computational tools. However, unlike other plastics processing operations, such as injection molding, the existing modeling tools for extrusion blow molding present several limitations, the most restrictive of which is that it is not possible to simulate all process stages in an interconnected manner, which has significantly limited the impact of modeling tools on this process.

The Extrusion Blow Molding Simulator project, is based in a collaboration of competences between the Logoplaste Innovation Lab and the University of Minho, aims to develop computational tools capable of simulating all stages of the extrusion blow molding process, using appropriate models to mimic the behavior of plastic materials. The codes developed are being validated with data collected in experimental trials, both at the lab and industrial scales.

This presentation will describe the simulation tools under development and the experimental work carried out to evaluate them, as part of the Extrusion Blow Molding Simulator project.

Acknowledgements:

This work was funded by the EBM Simulator project (POCI-01-0247-FEDER-047166) and by National Funds through FCT - Portuguese Foundation for Science and Technology, Reference UID/CTM/50025/2019 and UIDB/04436/2020, the PhD grant 2020.08395.BD. The authors also acknowledge the support of the computational clusters Search-ON2 (NORTE-07-0162-FEDER-000086) and Minho Advanced Computing Center (MACC).

Poster (S11-717, Time: Thursday 17:00, Room: Foyer)

A Computational Framework to Model the Selective Laser Sintering Process

da Costa Teixeira e Castro João[1], Nóbrega João Miguel[1], Costa R.[1]

[1]University of Minho, Guimarães, Portugal

Additive manufacturing (AM) processes allow for complex geometries due to their layer-by-layer building principle. The unneeded external tools make the processes cheaper and suitable for small quantity productions and iterative designs. Powder bed fusion (PBF) is a widespread AM technology that resort to high-density energy sources, such as lasers, that, when employed, the process is referred to as selective laser sintering (SLS). Although seemingly simple, the SLS process comprises multiple complex underlying multi-physics phenomena that hinder its controlled reproducibility. Using the trial-and-error experimental method in such intricate environment is challenging and the support of computational modelling can provide numerous advantages to its optimization.

The present work is embedded in a larger project that aims to develop an accurate simulation tool for the SLS process, within an open-source framework, that allows assessing the most relevant process parameters impact. To achieve that, the powder bed must be represented realistically, with adequate particle spatial and size distributions, using an appropriate simulator, LIGGGHTS. As a complement, the solver icoReactingMultiphaseInterFoam, available in the OpenFOAM computational library, was further adapted to better represent the most relevant physical phenomena involved in the SLS process. At the end, employing both tools, a representative section of the powder bed is used to simulate the sintering progression and assess the influence of the different relevant process parameters on it, while also validating the results accuracy.

ACKNOWLEDGEMENTS

This work was funded by National Funds through FCT - Portuguese Foundation for Science and Technology, Reference UID/CTM/50025/2019 and UIDB/04436/2020, and project SIFA - Sistema Inteligente de Fabricação Aditiva (POCI-01-0247-FEDER-047108). The authors also acknowledge the support of the computational clusters Search-ON2 (NORTE-07-0162-FEDER-000086) and Minho Advanced Computing Center (MACC).

Poster (S11-718, Time: Thursday 17:00, Room: Foyer)

An Open-Source Computational Framework to Support the Design of Complex Profile Extrusion Dies

Baptista Oliveira Marta Cláudia[1], Srivastava A.[1], Oliveira Vidal João Paulo[2], Sacramento A.[2], Guerrero J.[3], Nóbrega João Miguel[1]

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Numerical tools can provide an alternative route to design/optimize the extrusion forming tools even though most of the profile extrusion forming tool designers prefer to follow the time-consuming experimental trial-and-error approach. This is due to the high cost involved in acquiring commercial modeling tools and the lack of knowledge and skills in numerical modeling.

The main objective of the project, in which this work is integrated, is to establish new and improved design procedures for profile extrusion dies, implemented with the aid of free/open source software, that are able to run efficiently in High Performance Computing facilities. The improvements on the design procedures, as a result of this work, are expected to motivate industrialists to follow this route.

The proposed profile die design methodology integrates an appropriate solver to model the polymer melt flow inside the extrusion die, developed within OpenFOAM computational library. The implemented code comprises a temperature dependent viscosity model to calculate the non-isothermal flow of generalized Newtonian fluids, which follow an Arrhenius Law coupled with the Bird-Carreau constitutive model.

Within OpenFOAM, the solver was implemented in simpleFoam, which is a steady state, isothermal and incompressible flow solver. The temperature field is calculated by incorporating the solution of the energy equation conservation into the calculation sequence of simpleFoam. Moreover, at the end of each iteration the viscosity field is calculated with the updated temperature and shear-rate fields.

The results obtained with the developed solver are presented, comprising the velocity, pressure and temperature fields, for industrial relevant profile extrusion dies. These results evidence the ability of the solver to deal with complex geometries.

ACKNOWLEDGEMENTS

This work was funded by National Funds through FCT - Portuguese Foundation for Science and Technology, Reference UID/CTM/50025/2019 and UIDB/04436/2020. The experiment "HPC4PE - High Performance Computing for Profile Extrusion" has received funding from the European High-Performance Computing Joint Undertaking (JU) through the FF4EuroHPC project under grant agreement No 951745. The JU receives support from the European Union's Horizon 2020 research and innovation programme and Germany, Italy, Slovenia, France, Spain.

Poster (S11-719, Time: Thursday 17:00, Room: Foyer)

Resorting to High Performance Computing to Optimize Profile Extrusion Dies

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Theoretically, plastic profiles can have any cross section, tailored for a specific application. However, the usually employed design methodologies, based on experimental trial-and-error approaches, are highly dependent on the experience of the designer and require a large amount of time and material resources to achieve the desired product. These difficulties are obviously more evident when the desired profile has a complex geometry and there is a lack of previous experience with similar geometries or materials. The most relevant component of an extrusion line is the extrusion die, which should be designed to promote an even flow distribution at its outlet, a very difficult to achieve for complex profiles usually required in the most demanding applications. Accordingly, the support for simulation tool is expected to improve the design process, reduce the overall plastic profiles design costs obtain increased quality profiles and reduce the time to market.

This work describes the development of an appropriate computational automated workflow based on open-source and free access computational libraries, that are able to run in high performing computing (HPC) systems, implemented to support the optimization of profile extrusion dies. The main simulation codes that can run on HPC systems, were implemented in OpenFOAM and coupled to Dakota for optimization purposes. The implemented computational framework was tested with different industrial case studies. With this approach it is possible to assess hundreds of trials in the timeframe of a day, which showed to be clearly advantageous to allow exploring several design possibilities and, as desired, identify the most appropriate solutions.

ACKNOWLEDGEMENTS

This work was funded by National Funds through FCT - Portuguese Foundation for Science and Technology, Reference UID/CTM/50025/2019 and UIDB/04436/2020. The experiment "HPC4PE - High Performance Computing for Profile Extrusion" has received funding from the European High-Performance Computing Joint Undertaking (JU) through the FF4EuroHPC project under grant agreement No 951745. The JU receives support from the European Union's Horizon 2020 research and innovation programme and Germany, Italy, Slovenia, France, Spain.

Plenary (S12-804, Time: Thursday 09:10, Room: Sulzer Chemtech)

The complexity of redesigning the plastics system for circularity

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Recent studies show that a polymer circularity of around 80% could be reached (2050 horizon) if several technologies and circularity levers are used in a complimentary way.

As such, there is much more to plastics recycling than how a single recycled polymer will behave during processing. A whole system surrounds how plastic products are collected at end of life and sent to a potential diversity of sorting and recycling technologies. What goes where is not only determined by composition and quality but also by factors like waste management systems, policy and legislation, economic and ecological footprint, available tonnages, market demand and continuously advancing state of technology. Not all technologies are suitable for all polymers and input specifications differ per recycling pathway.

This lecture will take us into this complex landscape of plastics circularity and identify the boundary conditions we must respect as well as the challenges that are for us polymer scientists to tackle.

Keynote (S12-020, Time: Tuesday 10:15, Room: Bruker AXS)

Vitrimerization: A Novel Concept to Recycle Thermoset Waste via Dynamic Chemistry

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Vitrimerization is a newly developed concept to convert permanent crosslinked thermoset networks into vitrimer type dynamic networks via a simple, one-step method without depolymerization. Vitrimerization relies on designing a strategy to induce re-formability and healing in permanent chemically crosslinked polymer networks by using exchangeable chemical bonds that will lead to dynamic crosslinked networks. Key to the success of the strategy is establishing a process whereby exchange catalysts transform waste thermoset polymers into recyclable vitrimers. In vitrimerization, a mechanochemical process is used to form exchangeable linkages via ball milling the thermoset network with an appropriate catalyst. In this presentation we illustrate the use of vitrimerization for recycling epoxy and rigid polyurethane foams. We also show how the recycled thermosets can be used in combination with various nanofillers to manufacture nanocomposites with tailored properties. The vitrimerization approach is a low-cost, eco-friendly and scalable method that can be effectively implemented to address current challenges in recycling thermoset polymers. This simple and practical concept of recycling thermoset polymers without depolymerization provides a new strategy toward elimination of thermoset waste.

Keynote (S12-021, Time: Tuesday 13:45, Room: Bruker AXS)

Which circular economy for PLA? Technological, environmental and socio-economical points of view

Massardier Valérie[1], Sandei Benjamin[1], Barbaut Lea[1], Brette Olivier[1], Lazaric Nathalie[2]

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The development of biosourced and biodegradable plastics, with ability to be recycled, seems to be a relevant alternative to limit our dependence on fossil fuels as the amount of waste is increasing every year as a result of the increase in global plastic consumption. However, conventional plastics are so well established that it may be difficult for a new polymer to emerge and compete with them. The objective of this work is to study the scientific, technological and socio-economic barriers to the development of one such biosourced and biodegradable plastic, Polylactide (PLA), whose production remains marginal despite the benefits from its biosourced origin and its biodegradability. For this, we studied the influence of temperature and extrusion time, as well as the impact of successive thermomechanical cycles on the thermal degradation of PLA. This allowed us to see if this polymer could be recycled mechanically, with a simple process, at the end of its life. In addition, the socio-economic aspects of a circular economy of PLA were analyzed to determine wether the development of PLA industrial recycling could be relevant.

Keynote (S12-107, Time: Friday 10:15, Room: Bruker AXS)

Hybrid Chemical and Mechanical Approaches to Advance Plastic Film Recycling

Sobkowicz Kline Margaret J.[1], Ferki Olivia[1], Jamalzadeh Mansoureh[1], Martey Shawn[1], Chen Wan-Ting[1], Masato Davide[1]

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Over a third of global plastics production goes into single-use packaging, but a far smaller percentage of this packaging is successfully recycled or composted. The plastics recycling rate remains low due not only to infrastructural and geopolitical issues, but also because contamination and complexity in multi-material packaging degrades the quality of secondary feedstocks. Linking innovations in separations, catalysis, and product redesign with advanced processing capabilities in the plastics supply chain is key to scaling circular economy innovations. We use state-of-the-art extrusion approaches for recovering value from flexible films, a packaging waste stream that is particularly challenging to recycle. Multilayer barrier film is composed of different polymers that are not miscible, leading to interfacial weakness. The first case study describes a compatibilization scheme using gamma irradiation of PET as an in-situ approach to strengthen the interface when it is blended with polyethylene. Results show that branching and free radical initiation lead to reinforcement in the blends. A second case study investigates twin screw extrusion with oxidizing catalysts to induce structural modification in polyolefins. Two reactive extrusion schemes are demonstrated: maleic anhydride grafting and zeolite catalyzed branching. These approaches show promise for upcycling and viscosity modification, respectively. Spectroscopy, rheology, and mechanical testing are applied to determine causal relationships between process parameters and recyclate quality. Results from this work will inform process intensification through coupling of mechanical and chemical recycling approaches for more efficient recovery of value from waste plastics.

Keynote (S12-156, Time: Wednesday 10:15, Room: Bruker AXS)

Verification of the Effect of Processing History of Recycled Polypropylene through on-line Measurement of Melt-spinning Behavior

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It has been discussed that there is a structural change of polymer melt on a mesoscopic scale after applying melt processing for the production of polymer products. This may deteriorate the properties of the products from recycled polymers. To verify this concept, on-line measurements of the spinning behavior of virgin and recycled polypropylene (PP) were performed. For this purpose, model recycled PP was prepared through the crashing of injection molded plaque. Equipment for the simultaneous on-line measurements of diameter and velocity of the spin-line with the capability of automatic tracking was applied for the precise measurements of spinning behavior. It was revealed that the crystallization behavior of recycled PP was slightly suppressed in comparison with the virgin PP in a low-speed region. Oppositely, the crystallization behavior of the recycled PP was promoted in the high-speed region, where so-called neck-like deformation occurred. By decreasing the extrusion temperature, the region of take-up velocity with the suppression and promotion of crystallization shifted to lower speeds. It was concluded that the selection of processing conditions with the minimum memory effect of processing history is preferred for the fabrication of polymer products of better characteristics from recycled polymers.

Keynote (S12-198, Time: Tuesday 17:00, Room: Bruker AXS)

Towards effective upcycling of polypropylene: evaluation of the introduction of a chain extender on the processability and mechanical properties of recyclates

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The new circular economy policies developed in the recent years due to the increasing environmental awareness of society are changing the traditional way to design plastic products, in order to provide greater durability, reuse and high-quality recycle. The circular economy model suggests a rational utilization of the resources and promotes the valorization of the plastic materials already in circulation through recycling strategies. However, the market data of the last years indicate that the global economy of plastics is largely linear, with the disposition (with no recovery after their utilization) of more than half of the total amounts of plastic products produced per year. Therefore, there is an urgent need of increasing the recycling rate of plastics, to make the plastic industry more sustainable and compliant with circular economy policies. Among the different recycling technologies, mechanical recycle is the preferred option from both energetical and ecological point of view, as it ideally allows fully replacing the pristine polymers with materials recovered form wastes. Nevertheless, mechanical recycling strategies are really effective if closed-loop processes are realized. Actually, the degradation phenomena occurring during the re-processing significantly affect the polymer processability, also causing a progressive deterioration of the final properties of the secondary materials, with a consequent economic disadvantage of recycled polymers compared to their pristine counterparts. In this work, the effect of multiple re-processing cycles on the final properties of pre- and post-consumer polypropylene (PP) scraps was investigated. The materials were subjected up to ten re-processing operations in a twin screw extruder, and the rheological behavior, crystallinity degree and mechanical properties were evaluated as a function of the processing cycles. Furthermore, the influence of the introduction of a commercial chain extender additive was also evaluated.

Keynote (S12-366, Time: Wednesday 17:00, Room: Bruker AXS)

Multiblock Copolymers for Compatibilization of Mixed Stream Recyclates

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Due to phase separation of individual components, mechanical recycling of mixed polymer waste streams generally results in an unsuitable material with substantially reduced performance. However, when an appropriately designed compatibilizer is used, the recycled blend can have competitive properties to virgin materials. In its current state, polymer blend compatibilization does not compete with traditional waste management in terms of cost, but we believe that with further development and optimization, it can be made competitive. Historically, these compatibilizers have been diblock copolymers or in situ generated graft copolymers, but recent progress shows there is great potential for multiblock copolymer compatibilizers.1 In this talk I will describe the performance of some of the multiblock copolymers developed in the University of Minnesota Polymer group. Two of the most important mixed streams are polyethylene terephthalate (PET) with polyolefins and polyethylene (PE) with polypropylene (PP). Recently synthesized PET-PE multiblocks have been found to compatibilize PE in PET even added at levels < 1%.2 Similar levels of PE-PP multiblocks3 and PE with PP grafts4 are effective to create tough PE/PP blends. 1. Self et al., JACS Au 2022, 2 (2), 310–321. 2. Nomura et al., ACS Appl. Mater. Interfaces. 2020, 12 (8), 9726–9735 3. Eagan et al., Science 2017, 355 (6327), 814–816 4. Klimovica, K.et al. ACS Macro Lett. 2020, 9 (8), 1161–1166.

Keynote (S12-399, Time: Thursday 10:15, Room: Bruker AXS)

How surface treatment and/or reactive agents allow closed loop recycling of PLA/Flax and PLA/Bamboo reinforced composites to be performed ?

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Polylactic acid (PLA) composites with 10 wt% flax (FF) and bamboo (BF) fibers were prepared by melt mixing. Alkali pretreated fibers were treated by 3-aminopropyltriethoxysilane (APTES)[1-2]. A grafted copolymer with epoxy groups (Joncryl ADR 4468[3], acting as chain extender was used alone or in combination with the silane treatment.The influence of silane and/or Joncryl on the composite materials was investigated on the mechanical, thermal and thermomechanical properties of injection molding biocomposites. The adhesion of the fibres to the matrix was shown using SEM and FTIR. XPS indicated that chemical bonds were formed between silane and fibers. The combined used of Joncryl and silane in the corresponding composites (PLAJ-SFF and PLAJ-SBF), allowed a significant improvement of the thermal stability and interfacial adhesion to be achieved. With the silane-treated fibers, both tensile modulus and strength of the corresponding composites were improved. When adding Joncryl alone or in combination with silane, these mechanical properties improved further.Closed loop recycling was performed after hygrothermal ageing to simulate the lifetime of the composites and before grinding and reprocessing.All composites without modified fibres exhibited pull-out phenomena after recycling steps. The presence of Joncryl seemed efficient to maintain tensile strength, but mainly for PLA/Flax composites. Only the silane/Joncryl combination allowed the tensile strength to be conserved beyond 2 cycles of ageing and reprocessing. Bibliography [1] P. Sabarinathan et al, , Int. J. Biol. Macromol. 163 (2020) 2457-2464. [2] T.J. Lu et al. Compos. Pt. B-Eng. 51 (2013) 28-34. [3] J.M. Lopez-Cuesta et al.Materials (2019) 12(3):485.

Keynote (S12-426, Time: Thursday 13:45, Room: Bruker AXS)

Synergistic Effects of Hybrid Phosphorus Flame Retardants in Mechanical Recycling of PET

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Polyethylene terephthalate (PET) fabrics containing flame retardants (FRs) are not recycled mainly because the FR is a hazardous component. Downgrading of PET during mechanical recycling is another cause. So, PET fabric waste is often incinerated for energy recovery. To address this issue, a series of chemical, thermal, and rheological experiments were performed on several PET/FR compounds containing a DOPO derivative (DOPO-PEPA, shortened DP) and/or a phosphonate compound (Aflammit PCO 900, shortened AF). ICP-OES, NMR, and GPC were used to study the phosphorus components and characterize the polymer. Time-resolved frequency sweeps were implemented to assess the rheological behavior of PET/FR compounds. It shows that the DP/AF hybrid can stabilize the viscosity at a processing temperature of PET over long time scales. Based on this intriguing performance, to study the FR effects on the recyclability and processability of PET, repeated extrusion trials were carried out to imitate recycling. The recycled PET incorporated with hybrid FRs shows better mechanical behavior than PET/AF compounds after recycling. Since hybrid FRs hinder the degradation and improve the recycling quality of PET/FR compounds, it is significant to achieve a molecular understanding of this appealing synergistic FR effect. Density functional theory calculations and molecular dynamics simulations were performed to study the decomposition mechanisms of FRs. With the insight into phosphorus chemistry, it is promising to predict the processing behavior and improve the quality of recycling PET fabrics containing phosphorus FR.

Keynote (S12-676, Time: Wednesday 13:45, Room: Bruker AXS)

In-Process Melt Separation of PET/PE Blends. Part 1: Chain-Extension and Coalescing of PET

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[1]Case Western Reserve University, Ohio, USA

Multilayer, multicomponent films are widely used for their barrier properties, with more than 40 million tons of all plastic being produced annually being composed of multilayer polymer systems. This creates an enormous challenge as recycling of these multilayer systems is not technically feasible at large scales. In fact, the component plastics often have differing recycling pathways which prevents these films from being recycled. This work is the first part of a more general project aimed at developing a novel chemical-mechanical approach that leads to achieving in-process melt separation of polyethylene/polyethylene terephthalate (PE/PET) blends in twin-screw extrusion for posterior individual recycling. Herein, we use various levels up to 1.00% w/w of pyromellitic dianhydride (PMDA) as a PET chain extender at 260 °C and demonstrate that significant chain-extension occurs up to 0.5% w/w/ of PMDA, while chain-branching becomes dominant above this PMDA level. The corresponding increase in PET viscosity, and consequently blend viscosity ratio, induces coalescence of the PET droplets, which can then be mechanically filtered out of the main PE melt stream. This work further studies the coalescing ability of the chain-extended PET vs. that of branched PET and determines the optimal PET modification levels to maximize droplet coalescence within the typical processing window in twin-screw extrusion of 2-3 minutes.

Oral (S12-019, Time: Tuesday 11:00, Room: Bruker AXS)

Processing induced modification of commercial and waste PET and its effect on enzymatic depolymerization

Chang Allen[1], Patel Akanksha[1], Mastromonaco Abigail[1], AcostaDiaz Mauricio[1], Abid Umer[1], Ayafor Christian[1], Wong Hsi-Wu[1], Xie Dongming[1], Sobkowicz Kline Margaret J.[1]

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Plastic waste pollution is an increasingly vital problem to remedy as generation of plastic continues to far outpace recovery and recycling efforts. Enzymatic depolymerization of plastics has drawn significant attention as a low energy cost, environmentally friendly, and potential heterogenous waste treatment option. The influence certain material properties (e.g., crystallinity, glass transition temperature, surface area, etc.) have on enzymatic depolymerizations have been loosely studied by some of the broader community, but the bulk of the work in this area has tended toward enzyme engineering. Here, we present a process engineering-based approach to modifying, characterizing, and comparing material properties and depolymerizations of various commercial and post-consumer recycled PET sources. In general, enzymatic depolymerization of post-processed substrates showed improvements, some more drastic than others. Thermal analysis of as-received and post-processed substrates support the notion that decreases in crystallinity led to larger improvements in depolymerization. This trend held constant over various PETs and copolyesters, however, the influence of glass transition temperature became increasingly important when Tg was farther away from the operational 65 oC of the depolymerization reaction.

Oral (S12-025, Time: Tuesday 11:20, Room: Bruker AXS)

New approach for high-quality extrusion with low energy consumption in post-consumer recycling applications

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On the way to a closed loop in the plastics industry, highly qualified recycling technologies especially targeting post-consumer material streams are necessary. In times of CO2 taxes and increasing energy costs, the energy input per kg polymer (= specific energy input SEI) gains in importance. For this reason, we have developed a new machine concept for double filtration, focussing on low energy consumption and high material quality. The concept is based on the existing TVEplus® with a pre-conditioning unit, melting section, filtration (coarse), mixing section and melt degassing combined with a short melt buffering section and a melt pump prior to the second filter (fine). The melt pump is more effective in pressure build-up than a conventional screw and decouples the pressure demand of the second filter from the pressure at the screw tip. This results in a reduced screw length, lower melt temperature and lower energy consumption (~10% for the whole recycling line). The advantage in conveying efficiency and in reduced dissipation of a melt pump will be shown theoretically, as well as in experimental results. In contrast to existing double filtration systems available on the market the temperature peak in the extrusion system is shifted from the tip of the extruder screw (before the second filter) to the mixing section ("plus section") upstream of the degassing position. This optimized temperature profile along the extruder screw counteracts the subsequent outgassing of melt components after the degassing zone and gives a clear advantage for the quality of the melt and the recycled pellets. Another advantage of the plant design is the particularly advantageous configuration of the melt pump in the TVEplus® process sequence: filtration > degassing > melt pump. Thanks to this arrangement only pre-cleaned and degassed melt, flows through the melt pump and the service life of this component is increased. The new machine configuration brings us one step nearer to a closed loop and offers the opportunity to increasingly replace virgin material with high-quality recycled materials.

Oral (S12-027, Time: Tuesday 11:40, Room: Bruker AXS)

Challenges in Large Scale Single Screw-Extrusion in Polymer Recycling- from High Quality Application to Chemical Recycling

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To reduce running costs and to improve production capacities with reduced amount of space trends in recycling force the development of high throughput machines. Improved sorting and washing processes as well as digitalization and therefore high levels of automatization go hand in hand with large scale extrusion in polymer recycling. Therefore, this work presents modeling concepts to cover the different demands in terms of plastics recycling. Both the high-quality PET recycling market for bottle-to-bottle recycling and the chemical recycling applications for mixed polyolefin waste demand for high throughput capacities of more than 3.000 kg/h. The main challenges of scaling machinery and processes to high throughputs will be presented in this work as special scale-up rules for polymer recycling were developed. Scaling extruders means balancing conflicting requirements as the common scale-up factors for throughput are different than those for the melting capacity. Especially in times of CO2 taxes and increasing energy costs the energy consumption of the extrusion systems must be given priority. The developed scale-up rules were applied for PET as well as polyolefin extrusion systems up to 6.000 kg/h. Extensive experimental studies were performed in order to validate the theoretical scale-up rules. In these experiments various operating points were investigated and common extruder process parameters, like mass throughput and energy consumption were measured. To also validate the restrictions for melting capacity the melting performance was measured using ultrasonic measurements. To enable a quality assessment regarding thermal homogenization the melt-temperature distribution at the screw tip was measured. This work presents new scale-up rules which consider the demands of polymer recycling machinery and therefore assist the development new recycling plants with higher throughputs and lower energy demands. The application of these rules enabled the development of energy saving PET bottle-to-bottle recycling systems for up to 6.000 kg/h production. The scale-up rules are further applied to polyolefin recycling plants which are now capable of up to 4.000 kg/h.

Oral (S12-028, Time: Tuesday 12:00, Room: Bruker AXS)

Investigation of the influence of sorting on the property profile of colored polyethylene recyclates

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Plastics recycling is crucial for the transition into a sustainable circular economy in the plastics industry. Proper sorting is one of the essential tools to enable the production of high-quality recyclates. However, the sorting process entails several challenges that may hinder the development of plastics circularity. The aim of this study is to understand the influence of sorting on certain properties of recycled polyethylene. A high-density polyethylene (PE-HD) dominated post-consumer feedstock was used in this study. Amongst other pretreatment steps, the feedstock was color-sorted into five different colors, namely, two achromatic (incl. white and black) and three chromatic colors (incl. green, blue, and red). Afterwards, to produce recyclates with the specified colors, the color-sorted feedstock in form of flakes were re-granulated using an industrial recycling extruder with filtration and degassing systems. To characterize the property profile of the resulting recyclates, several measurements including melt flow rate (MFR); Charpy impact tests; and tensile tests were performed. A significant variation in the MFR values as well as the mechanical properties of the materials was observed. Moreover, to further investigate the influence of sorting, mixtures of the colored recyclates with various ratios were produced. The mixtures were compounded using a twin-screw compounder to generate new blends, which to a certain extent can simulate possible recyclates that could result from an unsorted feedstock. Thereafter, the same characterization methods were performed on the different blends to determine the changes in the material properties if the feedstock were unsorted. Additionally, as an attempt to test the applicability of existing mathematical mixing rules, the material properties of the color sorted recyclates were used to calculate the property profile of the mixed fraction.

Oral (S12-048, Time: Tuesday 14:10, Room: Bruker AXS)

Recycling of non-metallic fraction in waste printed circuit board for an electronic plastics circular economy

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Energy and resource recycling are required to solve worldwide energy shortages and reduce adverse environmental impacts. Reducing electronics waste is critical given the rapidly growing demand of electronic hardware. It is very challenging to recycle the vast amount of waste electrical and electronic equipment produced annually to minimize environmental pollution while creating value with the recycled material. Among electronic waste materials, printed circuit board (PCB) is the most attractive portion due to valuable materials that can be leached from it (i.e., precious metals). The basic structure of PCB is the copper-clad laminate consisting of 30%wt metallic fraction and 70%wt non-metallic fraction of glass fiber and brominated polymeric substance. The metallic fraction of waste PCB is welcomed by recycling industries, and precious metals recovery from PCB has become one of the most profitable businesses. However, the non-metallic fraction draws less attention due to its complicated recovery and lower economic benefits. Herein, two typical chemical recycling technologies, pyrolysis and chemical leaching, are introduced to depolymerize the non-metallic fraction of waste PCB into smaller valuable molecules. Gaseous products formed are mainly CO2, CO, bromobenzene, and some lower hydrocarbons (C1-C2), which had high heat value and could be used as fuel. Naphtha, asphalt, and other fractions like phenol and its derivatives, are detected in pyrolysis oil. Bromine content significantly decreased in solid residue after both de-polymerization methods according to elemental analysis. These two clean and non-polluting technologies offer a new way to separate valuable materials from waste PCBs and prevent environmental pollution.

Oral (S12-057, Time: Tuesday 14:30, Room: Bruker AXS)

Strategies for Improving Product Properties of Thermoformed Multilayer Cups with Contents of Post-Consumer Polypropylene Recyclate

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Thermoformed products bear great potential for the application of recycled materials when using multilayer structures for incorporating post-consumer recyclates. For achieving the tailored property profile, the shrinkage behavior, and the mechanical properties of the thermoformed cups and thus the used materials are of utmost importance. Therefore, recyclates must fulfill the corresponding requirements. Four different high-quality recyclate materials were selected for the processing of multilayer thermoformed products with top layers of virgin material. For comparison, a reference cup was made from virgin material. Various strategies for improving the product properties when using recyclates were evaluated. First, the influence of the amount of top layer material and the impact of adding virgin material to the middle layer was investigated with one selected recyclate. Furthermore, another selected recyclate was used for the evaluation of the variation of the absolute film thickness. Tests were performed on material level (thermo-analytical and mechanical properties) as well as on product level (shrinkage diameter and top load). On material level, the thermo-analytical measurements of the materials revealed polyethylene (PE) contaminations in all recyclates. The recyclates have different mechanical property profiles compared to the standard virgin material and lower stiffnesses were obtained. On product level, thermoformed cups with recyclates showed less shrinkage compared to the shrinkage of the virgin material cup. Most cups with recycled content achieve lower values than the reference cup. All tested strategies to adapt the film setup achieved better results in shrinking behavior and top load. Hence, (i) using a higher share of top layers, (ii) adding virgin material with high stiffness to the recyclate middle layer, and (iii) increase the absolute film thickness led to superior product performances. However, the latter strategy is economically not feasible.

Oral (S12-140, Time: Tuesday 14:50, Room: Bruker AXS)

Overcoming Gaps in Mechanical Recycling by Additive and Color solutions

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The Presentation will focus on today’s major gaps in mechanical recycling related to colorants and additives. To be able to develop tailor made solutions for plastic recycling, Avient invested in an Innovation Center for Mechanical Plastic Recycling (CycleWorks) where the entire recycling process can by run in a lab to pilot scale which allows to make in depth studies of the polymer behavior and decomposition under the influence of various parameters like e.g. additives, colorants, other polymer residues or processing conditions. The first part of the presentation will show a detailed analysis of mechanical recycling of Polyolefins in a multi loop process to simulate the effect of the polymer degradation after up to 5 recycling cycles of a packaging material. As the content of PCR in packaging articles is continuously increased, it becomes more and more important to understand how to best protect the polymers during the recycling process to have as less as possible accumulation of degraded and colored material, leading to a higher PCR quality and hence bring the industry one step closer to a truly circular economy. Analytical methods such as MFR, Gel Counting, Oxidation Induction Time (OIT), Oxidation Onset Temperature (OOT) and color index have been used to characterize virgin and recycled polymers and to benchmark the results against recycled polymers including standard stabilization packages and novel tailor-made stabilization formulations. In the second part the presentation will highlight the problems arising from gray PCR grades which can hinder a consistent and high-quality coloration of products containing PCR. A newly developed color simulation software can help to predict achievable colors based on gray starting material. Lastly the problem that black packaging articles cause in sorting lines will be described and a new solution based on NIR transparent black formulations will be presented.

Oral (S12-141, Time: Tuesday 15:10, Room: Bruker AXS)

Mechanical and microstructural characterization of paint powders filled thermoplastic polymers from recycling

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Today, plastic waste has become a major obstacle to greener technology. It is necessary to reduce its environmental impact by limiting its landfilling, incineration or erratic dispersion in the environment. The recycling process is a critical issue for the plastics industry in response to legislative and media pressure to protect the environment. It seems important to find recycling pathways that would turn plastic waste into a recoverable resource. The objective of this work was to analyze the mechanical properties of thermoplastic polymers filled with recycled paint powders. The study was conducted using a pure polypropylene and two grades of recycled polypropylenes denoted as NPP, RPP1 and RPP2, respectively. The blends were obtained using a twin screw extruder. The effect of the powders on the thermal properties of the blends was studied by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The addition of the powders was shown to have no significant effect on the thermal stability and crystallinity of the polymers. Injection molding was used was used to fabricate samples for tensile and Charpy impact tests. It was found that increasing the powder content increased the tensile properties of NPP and RPP1. It was also shown that powders caused a slight improvement in the elastic modulus and a slight decrease in the tensile strength of RPP2. The ductility of all the polymers decreased with the addition of the powders. The morphology of the tensile samples was observed by scanning electron microscopy (SEM) to analyze the fracture surfaces and relate the mechanical properties to the microstructure of the mixtures. While the recycling procedure caused chain scission of the PP phase, the studied blends maintained acceptable mechanical properties. These results showed that blending PPR with paint powders is a feasible way to reduce plastic waste and generate mechanically performance materials.

Oral (S12-160, Time: Tuesday 16:00, Room: Bruker AXS)

Evaluating the degradation influence of reprocessing cycles for injection moulded PETG grades

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In the scope of the mechanical recyclability of polyesters, the analysis of the processability after several reprocessing cycles and the quantification of the degradation during these reprocessing steps is of great importance. This paper therefore aims to study the influence of several reprocessing cycles on the thermal and physical properties of injection moulded PETG, bearing in mind its promising sustainability from multilayer packaging [1]. Three grades have been investigated, each having a different melt flow index ranging from 7-22 (240 °C, 2.16 kg). Reprocessing was done by shredding injection moulded produced parts and use these shredded parts as granulate to again injection mould. This way, the same material was reprocessed and after every processing step, the parts were fully characterized. Between every processing step, the material was dried to avoid degradation due to hydrolysis. This way, alterations in properties could be completely attributed to degradation during processing. To obtain information on the melt behaviour, MFI and inherent viscosity measurements were performed and discoloration of the samples was evaluated using a spectrophotometer. Thermal properties were determined with DSC and information on functional groups formation could be obtained with FTIR. In addition, tensile and impact tests were performed to quantify the decrease in mechanical properties when reprocessing. Chain scission, being the main degradation mechanism for (co)polyesters, refers to the rupture of polymer chains resulting in a decrease of the average molar mass. However, to verify if there are traces of cross-linking during the degradation, SEC was also performed. Significant changes between the different PETG grades are identified and discussed. The high viscosity grade especially, behaved much different showing more degradation during processing, compared to the low and medium viscosity grades. [1] L. Trossaert, M. De Vel, L. Cardon, M. Edeleva, Polymers, 2022, 14, 196

Oral (S12-172, Time: Tuesday 16:20, Room: Bruker AXS)

Chemical Disassembly and Recycling of Electronic Devices Manufactured with Biosourced Polymers

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Charmeau Jean-Yves[1], Cabrera Michel[1]

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Nowadays, the world production of plastics represents 360 Mt of wastes per year and could achieve 1000 Mt in 2050. Many methods exist to handle the end of life of these plastics: disposal in landfills, incineration, composting and two recycling techniques, mechanical and chemical. The mechanical recycling is the most developed industrially and chemical recycling is still to this day not very available on an industrial scale because of the higher cost; it represents 1% of the recycled plastics. We are interested in a disassembly and recycling method for plastronic devices manufactured using the In Mold Electronics (IME) process. IME consists in three main processing steps of thermoplastic polymers: screen printing, thermoforming and over-molding by injection. To that extent, electronic components and conductive ink are encapsulated in a polymer shell (thickness 3 mm). Plastronic devices are commonly made with petroleum-based polymers like PolyCarbonate (PC). There is an interest to substitute PC by a more sustainable alternative that could allow easier chemical recycling. In this work, we will present a method combining both mechanical and chemical recycling which made it possible to disassemble plastronic objects made of Poly(Lactic Acid) (PLA), a biosourced polymer. There are two main steps in our study: the disassembly of the electronic circuit from the polymer shell and the chemical recycling of the polymer. For the first step, a chemical dissolution is considered. Several solvents are tested to determine an environmentally friendly alternative with a sustainable life cycle. For the recycling step, two chemical reactions are compared (alcoolysis and hydrolysis). Materials and energy balances are discussed. Several analytical techniques are used (NMR, GC-MS, GC-FID, SEC) to characterize the products obtained during and after dissolution and depolymerization. Preliminary LCA is presented.

Oral (S12-203, Time: Wednesday 10:40, Room: Bruker AXS)

Investigation of the Fiber Length and its Distribution during the Recycling of Endless Fiber Reinforced Thermoplastics

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Fiber reinforced plastics show exceptional mechanical properties paired with low densities and are common for the mass production of lightweight components. Despite the increasing shortage of resources, the amount of reused fiber-reinforced waste and offcuts is still unsatisfactory. The mechanical recycling of fiber-reinforced waste and offcuts shows great potential, as necessary recycling equipment is already available and both fibers and matrix can be reused. In this study, a mechanical recycling process is developed and the steps along the process chain are investigated. Since injection molding of fiber-reinforced thermoplastics is a well-established process, the usage of the fiber-reinforced waste as a substitute for virgin material is investigated. The main challenge during recycling is the preservation of the fiber length, since the mechanical properties largely dependent on it. The offcuts and end-of-life components are shredded and further processed by a twin screw extruder to produce fiber reinforced granulate, which is further processed in an injection molding machine. The resulting fiber lengths and distributions are analyzed along the entire process chain. At first, the influence of fiber volume content, barrel temperature and screw speed during compounding on the fiber length is investigated. Due to increasing fiber-fiber interactions, an increasing fiber volume content shows the greatest influence on the fiber length. Furthermore, a low shear rate and a low melt viscosity are beneficial for maximizing resulting fiber lengths. During injection molding, the influence of initial fiber length, back pressure and screw speed is investigated. Gentle parameter settings and a process chain adapted to the processing of fiber-reinforced waste have a significant effect on the properties of the injection molded part. The utilization of offcuts and fiber-reinforced waste is a promising approach towards a circular economy.

Oral (S12-242, Time: Wednesday 11:00, Room: Bruker AXS)

3D printing fused filament fabrication (FFF) with recycled PP

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The Circular Economy Package in 2015 set for Europe specific key targets including recycling of municipal wastes at the level of 65% until 2030, recycling of packaging waste at the level of 75% until 2030, and reduction of landfilling to a maximum of 10% until 2030. In 2020 only 32% of polymers were collected for recycling, while 25% were sent to landfill and 43% were incinerated. The plastic recycling has therefore become one of the main challenges of environmental protection and waste valorization as it is recognized as an effective method to reduce the use of new natural resources through the recovery of materials. The use of waste or recycled thermoplastics for the development of 3D-printed parts is an emerging sector. Several case studies will be presented including the recycling of surgical masks and the recycling of industrial mixed polyolefin to demonstrate the possibility of exploiting recycled PP for the formulation of filaments that are suitable for FFF 3D printing processes. A Taguchi design of an experimental method was also used to optimize the processing conditions of blends showing adequate rheological and thermal characteristics for FFF. Blends containing till 50 wt.% of r-PP were selected for the production of filaments and for the subsequent 3D printing through FFF. The morphological observations allowed for verifying the quality of the produced samples, confirming the effectiveness of the proposed method in achieving FFF-printable materials. Finally, the mechanical characterization of the printed samples showed that adequate tensile properties were achieved compared to samples printed using commercial filaments. Overall, the obtained results demonstrated the possibility of using recycled polymers for additive manufacturing processes, therefore, allowing the valorization of a waste material through its exploitation for an advanced and sustainable manufacturing approach.

Oral (S12-246, Time: Wednesday 11:20, Room: Bruker AXS)

Pack2theLoop – Closing the circle of polyolefin packaging

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The integration of recyclates, made of post-consumer (PCR) plastic packaging waste, into the production of new equivalent products is still a major challenge and represents a promising approach to increase the required recycling rate of 50 %.

Therefore, the project Pack2theLoop, aims to recycle plastic packaging composed of polyolefins and polystyrene which are then applied in the production of new food and cosmetic packaging, closing the value-added loop.

The innovation goals are (1) the development of quality ensured recyclates from PCR packaging, ensuring the mechanical processability and the chemical and biological safety assessment, (2) the demonstration of the closed loop in course of defined Use Cases, (3) establishing "design for/from recycling" as the key to future and recyclable packaging, (4) as well as the development of a common language of the plastics/waste management/recycling sector through cooperation along the entire "plastics" value-added cycle.

Initial results from five sorting analyses of separate waste collection in Austria at three different sorting plants showed that rigid PE-HD, PP and PS account approx. 14 %, where PP is the largest amount followed by PE-HD and PS. In addition, exemplary samples from the yellow bag were analysed for their safety concerning the presence of mycotoxins potentially present in recyclates. Here no health effect could be identified, however, remains of labels showed cause for concern and will be further investigated. Selected samples from the waste stream recycled by the industrial partners were thermally, rheologically and mechanically characterised. Contamination tests with different amounts of PE-HD and PP showed good compatibility when PE-HD is the contaminant, while PE-HD mixed with small amounts of PP showed a significant decrease in elongation at break (εB). Further investigations relate to changes in PCR material properties as a result of multiple processing as well as the use of stabilisation additives.

Oral (S12-250, Time: Wednesday 11:40, Room: Bruker AXS)

Optimization and recycling of biocomposites : a case study on polybutylene succinate reinforced by maple fibers

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In this work, the properties of polybutylene succinate (PBS) reinforced with maple fibers (MF) was investigated in terms of tensile, flexural and impact strength. In the first part, the effect of MF content between 0 an 40% wt. was investigated to determine the optimum fiber content without coupling agent addition. In the second step, the optimum found in the first step (30% wt.) was used to optimize the concentration of maleated polypropylene (MAPP) used as a coupling agent between 0 and 15% wt. Finally, the optimized PBS-MF-MAPP formulation (70-30-10) was used in a closed loop recycling process to determine the changes in mechanical properties with respect to the number of time the material was recycled. In all cases, the compounds were prepared by twin-screw extrusion and injection molded to prepare the samples for characterization. These processes were selected to be more representative of an industrial scale production for these materials.

Oral (S12-262, Time: Wednesday 14:30, Room: Bruker AXS)

Investigation of enzymatic hydrolysis conditions on PET properties

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Polymers such as polyethylene terephthalate (PET) have good mechanical and barrier properties. However, their ubiquitous usage also introduces several problems: Monomers are obtained from non-renewable resources, and an increasing amount of non-degradable waste accumulates [1]. PET is used in the textile industry, often as poly-cotton blends. A comparatively novel approach to the problem posed by the mixed materials is the use of enzymatic degradation to remove the lower percentage component, allowing for fiber-to-fiber recycling of the PET [2]. For this, we have established a standard experiment based on a post-industrial PET/cotton blend that is able to remove the cotton. This process requires several steps: shredding, alkaline pre-treatment and finally the enzymatic hydrolysis, which is performed at 50°C and a pH of 5 for a reaction time of 48 hours. The question now is how these enzymatic process influences the PET. Therefore, the aim of this work was to investigate the different parameters on the properties of PET for fibre spinning. Laboratory experiments have shown that it is possible to produce PET fibres using a Göttfert HKR RH6000. The parameters used were a melt temperature of 260°C, an apparent shear rate of 100 s 1 and a 0.5mm nozzle, resulting in a fibre diameter of 0.1mm and a tensile strength of approximately 56 MPa. This simulated melt spinning process as well as the intrinsic viscosity (IV) of the material is used to assess the impact of each step of the process on the PET properties, using a 100% PET fabric as model organism. References [1] Roland Geyer, Chapter 2 - Production, use, and fate of synthetic polymers. in Plastic Waste and Recycling (Ed: Trevor M. Letcher), Academic Press 2020, p. 13. [2] B. Piribauer, A. Bartl, Waste management & research the journal of the International Solid Wastes and Public Cleansing Association, ISWA 2019, 37, 112.

Oral (S12-273, Time: Wednesday 14:50, Room: Bruker AXS)

Dihydroxy polyethylene additives for compatibilization and mechanical recycling of polyethylene terephthalate/polyethylene mixed plastic waste

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Single use plastics present a major challenge for sustainability as they are primarily either incinerated or accumulate in landfills and the environment. To improve their sustainability, technologies must be developed to return these materials to the market to extend their useful lifetimes and reduce resource waste. Mechanical recycling, which involves melt reprocessing waste plastic materials into new products, is an attractive option. Due to the near universal thermodynamic immiscibility of the most widely used plastic materials, waste streams must be sorted before recycling by melt reprocessing; however, this sorting process is expensive, often imperfect, and unable to handle multicomponent materials, meaning polymer-polymer blends are a common outcome. Without any additives, these blends are typically brittle and unsuitable for applications requiring toughness. Block copolymer compatibilizer additives can toughen blends by localizing at polymer-polymer interfaces and promoting adhesion between the two domains. In this work, we demonstrate the success of molecularly simple hydroxy telechelic polyethylene (HOPEOH) reactive additives for in situ compatibilization of polyethylene terephthalate (PET) and polyethylene (PE) mixed waste. Compatibilization is determined through tensile testing and quantification of particle size distributions. Blends containing HOPEOH show greatly improved mechanical properties, achieving an ultimate strain at break comparable to the virgin PET material, even at very low loadings (0.5 wt%) and without any added catalyst. Moreover, dispersed phase particle sizes decreased and were less prone to coarsening in blends containing HOPEOH, as observed by scanning electron microscopy and quantified by atomic force microscopy. Importantly, these additives were shown to have the same potency in blends prepared with post-consumer PET and PE suggesting their applicability as drop-in additives for already existing mechanical recycling applications.

Oral (S12-274, Time: Wednesday 15:10, Room: Bruker AXS)

Material recycling of post-consumer flexible polyethylene packaging waste

Boz Noyan Ezgi Ceren[1], Venkatesh Abhijit[2], Boldizar Antal[1]

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The growing concern in society has led to ambitious targets set by EU directives for an increased rate of recycling on plastic packaging waste, however the challenges involved in material recycling require better understanding on the development of the performance of the material. The purpose of this work was to study the properties of post-consumer flexible polyethylene packaging waste, using material sorted on a large scale from two different sources. Unwashed, laboratory-scale washed, industrial-scale washed, and industrial-scale washed and melt-compounded (industrially recycled) materials were used. The unwashed and washed flakes were melt-compounded on a laboratory scale with a twin-screw extruder using two different temperature profiles and two screw configurations. The pellets were then injection moulded into a shape of a frame with the purpose to obtain different material structures: sections with a mixed molecular orientation, a unidirectional flow and a weld line. The characteristics of the pellets were analysed with GPC, MFR and capillary rheometry. Moreover, the degradation status, thermal and mechanical properties of the samples were assessed. The washing reduced the polymer molecular mass and the melt viscosity, making the material susceptible to further degradation during melt-compounding and more so at a higher temperature. The Young's modulus and tensile strength were affected by the washing but not by the compounding temperature or screw configuration, while the elongation at break were affected somewhat both by the washing and compounding temperature but not by the screw configuration. The moulded samples made of unwashed, laboratory-washed and industrial-washed materials had a stiffness, a tensile strength and an elongation at break as expected of conventional polyethylene grades available. The industrially recycled samples, however, had a lower stiffness, a slightly higher tensile strength and a significantly greater elongation at break. This significantly different mechanical properties of the industrially recycled material were probably due to the degradation at recycling processing, possibly also to the additives in industrial melt-compounding.

Oral (S12-276, Time: Wednesday 16:00, Room: Bruker AXS)

Vitrimerization of Crosslinked Poly (Ethylene-Vinyl Acetate): A Mechanochemical Approach for Recycling Thermoset Polymers

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Crosslinked poly (ethylene- vinyl acetate) (EVA) is extensively used in a wide range of applications including solar cell encapsulates and athletic shoes. However, recycling large amount of EVA thermoset wastes is still a global challenge. Converting a permanent crosslinked network into a dynamic exchangeable network through a vitrimerization process is a cost effective and environmental-friendly operative method for recycling thermoset wastes already present in the market. In this work, crosslinked EVA is converted to a vitrimer via a mechanochemical approach using a catalyst and a feedstock of hydroxyl groups. The vitrimerized EVA can be reprocessed at least three times at temperatures as low as 120 °C (via extrusion and compression molding) without addition of extra catalyst or hydroxyl groups and without loss in the mechanical properties. This study shows that converting crosslinked EVAs into vitrimers using a mechanochemical approach is effective for different types of crosslinked EVAs with different topology and vinyl acetate content enabling EVAs waste to be recycled and reprocessed into value-added products.

Oral (S12-296, Time: Thursday 12:00, Room: Bruker AXS)

Incremental cost analysis and exploration of an improved mechanical recycling process for post-consumer flexible plastics

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To increase the volume of recycled plastics, technological advancements are necessary to deliver high-quality regranulates (r). The so-called quality recycling process (QRP), proposed by CEFLEX, applies this strategy on post-consumer flexible plastic (PCFP), which is generally considered to be a difficult-to-recycle fraction, as only 17% of the fraction is recycled and eventually ends up in low-value-applications. In material recovery facilities, the biggest part of PCFP is sorted into bales that can, according to the 'Duales System Deutschland (DSD) GmbH' standards, be referred to as the DSD 310-1 and DSD 323-2 bales; these bales are respectively rich in PE film and PO film. QRP targets both bales and subsequently results into two improved, rPE Film Natural and rPP Film, and two drop, rPE Flex and rPO New, fractions. As proven by previous research, QRP achieves a similar process yield to conventional PCFP recycling, as well as higher quality – in terms of both polymer and transparency grades – for the two improved fractions. QRP attains this by introducing extra processing steps, such as additional sorting, hot washing, and deodorization, as compared to conventional mechanical recycling. Nonetheless, the question of economic feasibility remained unanswered. In this article, we present an incremental cost analysis, which can act as a decision-making tool for exploration. Accordingly, this article applies a combined methodology from engineering and management accounting, i.e. mass flow analysis and the two-stage cost allocation method, to assess the associated cost of improving these quality parameters. This results in insight into the cost of different processing strategies - i.e. extra cost for improved recycling as compared to classic recycling - the difference in costs in terms of input bale quantity versus the amount of output granulate, as well as the origin of the incurred costs. Furthermore, unused capacity and processing line optimization will be discussed.

Oral (S12-312, Time: Wednesday 16:20, Room: Bruker AXS)

Mechanical Recycling of Polyethylene and Polypropylene Wastes via Organometallic Complexes

Girişken Çağla[1], Kurucu Oguz Alp[1]

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Global warming and depletion of sources have caused to arise attention to circular economy and sustainability in recent decades. As is known, mechanical recycling is still more cost-effective and environmentally friendly option than other recycling methods and it is also crucial part of the circular economy and the sustainability. Since seperation and sorting of polyethylene (PE) and polypropylene (PP) waste mixtures is still challenging and costly, upcycling of plastics by compounding in presence of various compatibilizers considered to be promising method for recycling. In this study, titanium and vanadium based novel multi-functional compatibilizers were synthesized to obtain compounds that have mechanical properties comparable to pristine PP/PE blends, single PP and single PE. PP and PE with and without multi-functional compatibilizers were compounded and molded by using high shear mixer and injection molding, respectively. %0,3 wt titanium based compatibilizers with 8-carbon chain which studied before in the literature, improved elongation at break by 165% compared to pristine PP/PE blends. Since vanadium posses much more electron affinity than titanium, the best result was obtained for %0,3 wt vanadium-based compatibilizers as %328 strain followed by %0,3 wt titanium-based compatibilizers with 18-carbon chain as 284% strain. Furthermore, as a general trend vanadium based compatibilizers was more effective than titanium based compatibilizers. New and novel multi-functional compatibilizers, especially vanadium-based ones, were effective to obtain materials that have desired properties. They might be useful particularly to obtain substitue materials for plastic film applications. However, a lot of experiments are still required for detailed understanding and future perspective.

Oral (S12-330, Time: Thursday 10:40, Room: Bruker AXS)

The influence of multiple mechanical recycling on the properties of glass fibre reinforced composites made from organosheets

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Continuous-fibre reinforced thermoplastics are attractive materials for automotive industry to fulfil their quest for weight reduction. Low density alongside with short cycle times and high mechanical properties offer these so-called organosheets a wide range of applications. [1] At the end of their life, however, these parts can hardly be recycled. A way to recycle them nonetheless is to shred them and use these chopped fibres as filler in new compounds. Though, the (multiple) processing of glass fibres in mechanical recycling poses a challenge as the fibres break due to shear and stress and recycling may have an end at some step. The aim of the work was to investigate the integration of shredded organosheet as filler component in glass fibre reinforced PP with three different fibre contents, with the highest fibre content corresponding to 100% shredded organosheet (i.e. 45 vol.% of glass fibres in the composite). Three compounds with 45, 30 and 15 vol.% of glass fibre were each subjected to several recycling sequences and the influence of the recycling steps (shredding, compounding, injection moulding) on fibre length and composite performance was evaluated. Additionally, we compared our experimental results with the results from available models for the property prediction of fibre-reinforced composites. We see, that due to the recycling step, a degradation of fibre length and composite properties is happening, which is also in correlation with the fibre content. With our research, we hope to contribute increasing the recycling rate of glass fibre reinforced thermoplastics and to show that it is also possible to preserve the value of these materials instead of thermally recovering them. Acknowledgments The authors are grateful to Talib Al-Hashmi for carrying out part of the lab work. References [1] Stock, A.; Egger, P.: Hybridteilfertigung – Organobleche verlassen das Hochpreissegment. MM Composite World 2011/02, 12–15.

Oral (S12-342, Time: Friday 10:40, Room: Bruker AXS)

Inks as a potential source of contamination for PE film recycling

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Flexible plastic packaging is the fastest-growing category, but its recycling rate is very scarce due to its complex composition. One of the biggest hurdles impeding the recyclability of flexible packaging is the presence of contaminants. Inks are very different from the carbon structure of polyolefins. Thus, they can interfere during recycling processes and adversely impact the quality of the recyclate. Therefore, a fundamental understanding of the chemical interaction between the polymer and the other packaging components is urgently needed to improve the relationship between inks and reprocessing. This study focuses on the investigation of macromolecular degradation of ink components, i.e. binder and pigments. It is aimed to gain knowledge on the physical and chemical behaviour of the ink components during an industrial recycling process. This is achieved by 1) determining the kinetic degradation profile of typical inks predominantly used by industry, such as Polyurethane-based (PU) and Nitrocellulose-based (NC) binders, pigments, e.g. copper phthalocyanine through TGA-FTIR measurements performed under typical extrusion conditions, 2) determining the potential VOCs formed from the inks during the extrusion via analytical techniques, e.g. GC-MS, and 3) investigating physical and mechanical properties of the extruded plastic films containing the studied ink components. This will provide valuable insights into the effects of ink components on the recyclability of flexible films to improve flexible packaging recyclability towards achieving higher recycling rates.

Oral (S12-371, Time: Friday 11:20, Room: Bruker AXS)

Impact of Recycled-Polypropylene Viscosity on the Embodied Energy of Injection Molded Parts

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Energy consumption in injection molding is affected by processing conditions and polymer properties. Understanding the relationship between these parameters enables calculating the embodied energy associated with injection molded parts. This research investigates the correlation between polymer rheology and injection molding energy consumption for recycled polypropylene. The secondary feedstock was fully obtained from a post-industrial film waste stream. The recycled polymers were modified via high-speed extrusion to reduce the molecular weight and control the rheological properties. The experiments focus on the characterization of thermal and mechanical energy drawn by the injection molding machine while monitoring the polymer states during the cycle. The data collected from the experiments are used to calculate the embodied energy as a function of the polymer rheology and processing conditions. The results analyze the relationship between polymer rheology, processing, and product characteristics.

Oral (S12-386, Time: Thursday 11:20, Room: Bruker AXS)

Kinetics of Poly(ethylene terephthalate) Fiber With Different Morphologies Glycolysis in Ethylene Glycol

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In 2021, the annual production of polyester in China reached 53.63 million tons, while the recycling rate is less than 20%, a large number of polyester waste textiles are burned and landfilled, causing serious pollution of the environment and a great waste of resources. Chemical methods can depolymerize the polyester component of waste polyester textiles into diethylene terephthalate (BHET) monomer, which can be repolymerized after purification, and is a potentially effective method to achieve true closed-loop recycling of polyester raw materials. In our work, at 190 °C, the glycolysis of waste PET was studied using excess EG as the glycolysis agent (6:1) and zinc acetate as the catalyst, focusing on kinetic aspects were studied. The main interactions between the different aggregate state structures such as crystallinity, orientation, and so on were investigated to optimize the yields. Good yields of bis(2-hydroxyethyl) terephthalate (BHET) were obtained for monomeric terephthalate under optimal conditions.

Oral (S12-409, Time: Thursday 11:40, Room: Bruker AXS)

Insights into the Mechanical Deformation Behavior of High-density Polyethylene Contaminated Polypropylene

Seyed Khabbaz Hamid[1], Gooneie Ali[1], Ragaert Kim[1], Demets Ruben[1]

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Cross-contamination of plastics is an inevitable consequence of current sorting technologies, which rarely separate plastics into 100% pure fractions. It is a major problem that affects the mechanical properties of recycled plastic, thereby precluding the full implementation of a closed-loop recycling system. The purpose of this study was to investigate the effects of cross-contamination on the tensile deformation and mechanical behavior of polypropylene (PP) contaminated with HDPE, two plastics commonly found in sorted packaging wastes. Four types of PP, varying in molecular structure and viscosity, were melt-blended with three types of HDPE at waste-representative concentrations. In the case of polypropylene homo- and co-polymer, a localized stress concentration near HDPE dispersed phase increased the probability of matrix shear yielding, resulting in a decrease in yield strain values. However, this effect was not observed in the PP block copolymer due to the changes in the structure of core-shell dispersed particles, leading to improved interfacial adhesion. The plastic deformation of all compositions was governed by a competition between the shear yielding of the PP matrix and the progressive opening of microvoids through decohesion between two phases. Nevertheless, each composition followed a different scenario. PP copolymer was more robust at 10% of high-viscosity HDPE contamination. This can be attributed to the higher degree of intercrystalline links in the imperfect PP crystals. Low-viscosity PP homopolymer maintained its tensile ductility only with well-dispersed low-viscosity HDPE. PP block copolymer displayed a pronounced improvement in its tensile elongation and impact strength with the addition of high-viscosity HDPE, given the encapsulation of HDPE in its multi-layered core-shell structure.

Oral (S12-410, Time: Thursday 14:10, Room: Bruker AXS)

The role of the hard segment content in the foaming of thermoplastic polythiourethanes with supercritical CO2.

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Nowadays, the development of new smart polymers easy to recycle is widely investigated, being the polythiourethanes one of the most promising alternatives within the family of polyurethanes. In addition, the utilization of supercritical CO2 as a blowing agent in polymers foaming is also considered an interesting green alternative when physical polymer foaming is needed. Because of that, in the present work the foaming of thermoplastic polythiourethanes using supercritical CO2 was carried out. Due to the great influence of the internal structure of those materials in their final properties, three films with different hard segment content (23 wt%, 33 wt%, 43 wt%,) were synthesized, characterized, and foamed. After characterization, it was confirmed that no bands in the wave lenght corresponding with isocyanate or thiol appeared, corroborating that all reagents have reacted completely. Moreover, the influence of the hard segment was specifically noticeable in the case of mechanical properties. Because of the increase in hard segment content, polymers were able to resist higher stress as well as became less elastic. On the other side, once the foaming tests were performed, it was seen that not only the expansionability depends on the hard segment content, being this lower as the hard segment content increases, but also the effect of temperature on the expansion ratio also depends on it. On the other side, when certain value of foaming pressure is reached, in this case 150 bar, the influence of temperature on the cell density is negligible.

Oral (S12-453, Time: Thursday 14:30, Room: Bruker AXS)

Effect of recycling on reprocessed PHB modified with virgin PHB and characterization of thermal and mechanical properties

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Bioplastics as an alternative feedstock to conventional materials have been recognised by the United Nations in 2021. As circular economy ideals state that the material has to be kept in the loop, the mechanical recycling of these plastics needs to be explored further. Polyhydroxybutyrates (PHB), which are both biobased and biodegradable, have a promising time to come in the packaging market. They belong to the family of Polyhydroxyalkanoates (PHAs) and are produced within micro-organisms. The current work addresses the gap in mechanical recycling potential by investigating the properties at each stage from once extruded (E) PHB blended with virgin (V) material in two ratios (E20V80 and E80V20) and further extruded 5 times (E5) in a twin-screw extruder. At each stage, some material was used for thermal testing and injection moulding of test specimens for mechanical testing. The tensile strength remained almost constant for the series E80V20, and the E5 only dropped ~6% from that of the E80V20. Strain at break decreased from E80V20 to E5, which was corroborated by Charpy Notched Impact results showing a progressive brittleness of the samples with a 40% reduction at the end of E5. E20V80 showed similar trends to the E80V20 series, but with higher values in the impact strength as expected due to the higher amount of virgin material and only differed in the tensile strength in which an increase was observed from the E20V80 to the E1. The thermal stability of the materials was assessed by the degradation temperature of TGA measurements, which did not change even after 5 reprocessing cycles. DSC studies showed that the degree of crystallinity from the 1st heating scan marginally increased as the cycles progressed from 41% to ~44%. The melting temperature did not vary a lot with each cycle and was ~175°C. We demonstrate quantitatively and qualitatively that the utilization of recycled PHB into packaging is an area of growth and importance for the circular economy.

Oral (S12-461, Time: Thursday 14:50, Room: Bruker AXS)

Effect of Reprocessing Method on the Molecular Structure of an Impact Copolymer Polypropylene

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The degradation of polypropylene during reprocessing reduces the molecular weight, and consequently, limits the performance of the reprocessed (recycled) resins. Improvements in stabilizers have reduced the levels of degradation, enabling greater reuse of reprocessed resins. This study investigated the influence of the reprocessing technique on the degradation of a more stable impact copolymer polypropylene. The polypropylene was reprocessed using twin screw extruder and quad screw extruder with the different screw speeds. The reprocessed materials were characterized for their rheological and thermal properties as well as level of stabilizer remaining in the polypropylene. In addition, the reprocessed samples were fractionated using a suitable organic solvent to separate homopolymer matrix and rubber fractions; the relative changes in molecular weight were determined from the rheological properties of soluble and insoluble fractions. Results are discussed with respect to screw speeds since the quad screw extruder enables greater throughput for repelletizing polymers.

Oral (S12-476, Time: Thursday 15:10, Room: Bruker AXS)

Reincorporation of plastic wastes in an integrated circular process: strategies towards sustainability

Borges Carolina[1], Araújo Andreia[1], Santos Raquel M.[1]

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Over the last 70 years, the plastic industry has grown exponentially due to both the variety of plastics available and the relatively cheap production from petroleum. Hence, they have become vital materials for several everyday life applications. Also, their excellent mechanical and barrier properties, together with lower density and chemical inertness turn them into highly demanded materials in different industries, including packaging, building and construction, automotive and medical. However, in today´s scenario the ever-increasing amount of plastic is a matter of concern, giving its impact on biodiversity and contribution to climate change. Hence, industries are becoming increasingly interested in managing, recycling and recovering their waste. In this work, the properties of industrial plastic-based residues were assessed for subsequent reincorporation of the material. For the effect, formulations were developed in a twin-screw extruder for further characterization, while maintaining rheological behaviour. Spectrophotometric analyzes were performed on the CIELab color space to adjust compounds' colors and their mechanical and thermal behaviours were also evaluated through tensile tests and DSC analysis, namely. In this way, it was possible to develop products with higher added value, decreasing primary resource consumption but increasing secondary raw material shares. Thus, circular economy and sustainability are highlighted, in a world where progressive efforts are being done accordingly.

Oral (S12-485, Time: Thursday 16:00, Room: Bruker AXS)

Determination of polyethylene (PE) and polypropylene (PP) content in post-consumer recycled flexible plastics using machine learning assisted differential scanning calorimetry (DSC)

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One of the main challenges of working with post-consumer recycled plastics is often their unknown composition, both in type and amount of different plastics in the blend. This composition influences the processability of the regranulate and also the performance of the final produced item. The existing techniques (both at flake and regranulate levels) to determine the composition are generally either low in accuracy or they are time and/or cost -intensive. The issue is paramount in case the plastics are difficult to separate, like PE and PP, as they both float in a sink-float sorting procedure and not all the recycling facilities are equipped with near-infrared sorting modules. This study presents a novel way of determining the composition at regranulate level by means of DSC. The current technique uses machine learning and is trained to be able to determine the composition of recycled flexibles with an error of less than 0.5 wt%.

Oral (S12-535, Time: Thursday 16:20, Room: Bruker AXS)

Performance Analysis of Solid-State Shear Milling of Waste Plastic Particles

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Solid-state shear milling is increasingly used for polymers solid state compatibilization, particularly useful in recycling commingled waste plastic particles into fine powders that can be extruded or moulded without pellitization to avoid additional heating that degrades the polymer. The resulting products have been found to have mechanical and physical properties significantly better than those of polymers recycled by conventional techniques and sometimes rivaling even those of virgin polymers. The reason for this is the 3D scissor size reduction that occurs between the bevelled ridged geometry of the engraved pair of discs. During operation, the discs face each other in rotation and under load, trapping cell spaces between them that change in shape, volume and position in both radial and circular directions. High pressures, shear, elongation and reorientation ensue in these spaces filled with particles resulting with a rapid breakage of particles. As they break, the particles move towards and discharge at the brim of the pan. Since its development, the S3M has been the subject of several studies. The materials that have been milled include polymer pellets (polystyrene, blends of polypropylene/polyamide 6 and polyethylene), recycled plastic waste particulates (fiberglass-resin from waste printed circuit boards and automotive plastic shredder residue). In addition, particles that could aid milling have been trialed, iron powders with polypropylene and salt crystals with polyethelene. The operating parameters that have been changed in these studies include the rotating disc rotational speed, the applied load and the number of passes through the mill. This presentation will bring together all this valuable data and extract from them operation equations that predict the fractions milled within a size and the minimum particle size achieved. These equations can then be used to analyse the performance of the S3Mill, optimise operation and develop better future designs.

Oral (S12-562, Time: Thursday 11:00, Room: Bruker AXS)

Effect of mechanical recycling on short glass fibre reinforced polyamide 6,6 from post-industrial waste

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Recycling post-industrial plastic waste offers a series of advantages compared to post-consumer recycling, specifically: (1) the waste stream is not contaminated by other materials, (2) its composition is generally known and stable, and (3) limited degradation. This makes it a commercially viable and effective recycling strategy, which is nowadays exploited by many manufacturers. Nonetheless, there’s currently a lack of technical knowledge on the effects of recycling on materials with a complex composition, such as glass fiber-reinforced plastics. For these materials, in fact, compliance with certain dielectric and self-extinguishing properties is often required, as well as good mechanical behavior. This study aims to fill this gap by analyzing the effect of the integration of recyclates into injection-molded parts in percentages ranging from 0% to 20% wt. The material of choice is a polyamide 6,6 composite containing 30% wt. of short glass fibers. The recycled granules are produced by grinding injection molded parts and subsequently extruding the resulting ground material. These are then mixed with virgin granules in different percentages by weight (5, 10, 15, and 20%) and the mixtures are used to produce the specimens to be tested. A wide characterization of the mechanical, thermal, rheological, and dielectric behavior of the different materials is performed to verify compliance with the stringent requirements for this class of materials. This approach will help to further increase the level of technical knowledge on the post-industrial recycling of glass-fiber reinforced polyamide, providing manufacturers with useful data for the industrial implementation of the process. The benefits deriving from the introduction of closed-loop recycling are evident: not only from an economic point of view (material savings) but also from an environmental perspective, as in fact, mechanical recycling has significantly lower impacts than the production of virgin plastic.

Oral (S12-568, Time: Friday 11:00, Room: Bruker AXS)

Glycolysis of sorted end-of-life polyurethane wastes

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Polyurethanes (PU) occupy the sixth position of the most used plastics in the world, due to their multitude of applications. Their high demand is finally translated in a large amount of waste, the majority of which ends up in landfills causing an environmental problem. The PUReSmart project seeks ways of transitioning from the current linear lifecycle of PU products to a circular economy model. To achieve this objective, glycolysis is proposed for the chemical recycling of these materials, being able to recover the raw materials from end-of-life PU wastes, such as the polyol and the isocyanate. The glycolysis of the foams were carried-out at pilot plant and demonstration scales, minimising the excess of glycolysis agent maintaining the splitting of the phases. The scraps feeding system and the further separation devices were improved for getting a superior quality of both polyol and isocyanate rich phases. The glycolysis product obtained presented split-phase, where the upper phase (UP) was composed mainly by the recovered polyol and the bottom phase (BP) by the reaction-by products, the excess of glycol and slight losses of solubilized polyol. After the glycolysis process and a subsequent purification process, the recovered polyol obtained presented similar properties to commercial polyols, being possible to use it as replacement in the synthesis of new PU foams. On other hand, the PUReSmart consortium succeeded in recovering the isocyanate from the BP with properties similar to commercial isocyanate. Finally, different polyurethane foams were synthesized, using both recovered materials (polyol and isocyanate) as a total replacement for the commercial ones. The PU foams obtained presented adequate properties, complying with the specifications for which are designed.

Oral (S12-606, Time: Friday 11:40, Room: Bruker AXS)

Effect of molding pressure on lamellar structures and mechanical properties of mechanical recycled low-density polyethylene

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Mechanical recycling is the most suitable process of plastic based on the study from life cycle assessment. On the other hand, heat and steady shear from mechanical processing affected the degradation of mechanical properties of recycled plastic products. From our previous studies, it can be found that mesoscale lamellar structure of plastics had a strong correlation with mechanical properties and processing conditions. Especially in high-density polyethylene (HDPE) which consisted in linear chain structure, the lamellar structures were decreased with high steady shear. Interestingly, it can be successfully regenerated by the addition of dynamic shear in mechanical recycling process. As a result, mechanical properties of HDPE mechanical recycled products were improved as similar as virgin HDPE. In other way, mechanical recycling process of low-density polyethylene (LDPE) has still been challenging. This is because its long-branching chain structure obstructs the regeneration of lamellar structure caused by dynamic shear. As a result, the mechanical properties of mechanical recycled LDPE are difficult to improve. In this study, the addition of pressure in molding process is performed in order to study the effect of additional pressure on the regeneration of lamellar structures of mechanical recycled LDPE. The thickness and ratio of lamellar structures will be evaluated and correlated with the changes of mechanical properties and mechanical recycling process conditions. This study is expected to win an obstacle of LDPE mechanical recycling approach in order to prolong the lifespan of LDPE.

Oral (S12-628, Time: Wednesday 14:10, Room: Bruker AXS)

In-Process Melt Separation of PET/PE Blends. Part 2: SC CO2-assisted separation of depolymerized PET

Vecchi Steven[1], Hampton Lauren[1], Kone Ezra[1], Ghassemi Hossein[1], Schiraldi David[1], Maia João[1]

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Multilayer, multicomponent films are widely used for their barrier properties, with more than 40 million tons of all plastic being produced annually being composed of multilayer polymer systems. This creates an enormous challenge as recycling of these multilayer systems is not technically feasible at large scales. In fact, the component plastics often have differing recycling pathways which prevents these films from being recycled. This work is the second part of a more general project aimed at developing a novel chemical-mechanical approach that leads to achieving in-process melt separation of polyethylene/polyethylene terephthalate (PE/PET) blends in twin-screw extrusion for posterior individual recycling. Herein, we use various levels up to 8% w/w of ethylene glycol (EG) to depolymerize PET at 260 °C and demonstrate that significant chain depolymerization occurs with a reduction of PET molecular weight (MW) greater than 95%. The corresponding decrease in PET MW allowed for the PET to be depolymerized to a level that was extractable with the aid of SC CO2. The resulting PET wax separated from the PET/LLDPE film was further depolymerized into monomeric form with the aid of zinc acetate. This work further studies the effect EG%, time, temperature, screw design, feed rate, and screw speed have on the depolymerization in a twin screw extruder. Two different reagents EG and BHET (Bis(hydroxyethyl)terephthalate) were used to look at the depolymerization efficiency, with EG being the more efficient depolymerization reagent.

Oral (S12-678, Time: Friday 12:00, Room: Bruker AXS)

Ecological and economical evaluation of available recycling technologies for polyester-containing textiles

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Due to new legislation, rising disposal costs and technological innovations, the recycling of textile waste will change significantly in the next years. Various end-of-life scenarios for textile waste are currently being developed or are already in use. The choice of economically and ecologically sustainable end-of-life scenarios, such as mechanical, thermomechanical and different chemical recycling processes, is challenging and depends highly on the waste stream quality. Therefore, textile polyester input streams and their detectability using near-infrared (NIR) is investigated. NIR trials show that certain fibres types, such as elastan, or thin surface coatings made of PU cannot be detected and therefore cannot be sorted out before the materials enter the recycling process. By using life cycle assessment (LCA) modelling, chemical recycling processes are evaluated in comparison to state-of-the-art processes for polyester-containing used textiles. The focus of the comparison is on tearing, regranulation, solvent-based separation as well as glycolysis, hydrolysis and methanolysis. In addition, the most promising chemical recycling processes, namely glycolysis and hydrolysis, are investigated simulatively and experimentally for their mass and energy balance, cost structure and sensitivity to textile-typical impurities. The investigations show a high dependency between the process feasibility and the (sorted) input stream. Some bottlenecks of the processes depending on input stream are loss of chemical auxiliaries absorbed by natural fibres or loss of product in a decoloration step. The developed model enables researchers and companies to classify their existing or to-be-developed technologies and/or textile waste streams in the textile recycling market.

Oral (S12-690, Time: Wednesday 12:00, Room: Bruker AXS)

Design and Production of Packing Films in line with Recycling

Rodrigues Pedro A.[1], Barros Carolina[1], Carneiro Olga S.[1], Cruz Vasco[1], Machado Ana Vera[1]

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Polyolefins are the synthetic polymers with the highest commercial success, accounting for more than 47 % of Western Europe's total consumption, approximately 24.1 million ton per year. They present a combination of physical properties (flexibility, strength, lightness, stability and impermeability) that are ideally suited to a wide variety of applications, such as food packaging. Owing the increase on the production of commodity plastics, as well as, on the landfill rate in the last two decades of the 20th century, they are currently the most common and persistent pollutants worldwide. Once such material became part of the natural ecosystem, its long-lasting duration has a huge contribution to environment contamination, representing, for instance, a major threat to marine ecosystem. Plastic packaging is a priority area when it comes to designing for recycling. Since product development is one of the fundamental elements for improve recycling levels, it has been estimated that design improvements to decrease the cost of recycling packaging waste plastic. The activities developed in this work through Product Design will contribute to respond to the research/innovation challenges applied to the design phase of the article/product/packaging, aiming to improve its general roundness. Decrease the incorporation of multi materials in products/packaging to improve recyclability. At a PE matrix several nano and microparticles were add in the melt to achieve the same film performance as with multilayer polymers. The results obtained until now, demonstrate that it is possible to follow this path to develop more sustainable packaging film.

Oral (S12-698, Time: Tuesday 10:40, Room: Bruker AXS)

Towards a circular economy for carbon-fiber reinforced polyetheretherketone (CFRPEEK) composites: reusing, recycling and remanufacturing

Santos Raquel M.[1], Borges Carolina[1], Chícharo A.[1], Araújo Andreia[1], Silva J.[1]

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Traditional fiber-reinforced polymer (FRP) composites are known for their remarkable in-plane stiffness- and strength-to-weight ratios, being very attractive for highly demanding industries with stringent requirements. Moreover, the growing demand for fuel efficiency and greenhouse gas emissions reduction, in different mobility sectors, will contribute to an increase in the composites market since the main driver is the highest performance achievable with lightweight materials. Currently, the main disposal routes for FRP composites are through landfill and incineration, which are at the bottom of the waste management hierarchy. Sustainable and profitable recycling methodologies, in line with circular economy concepts, need to be developed and implemented since a considerable increase of generated FRP composite wastes is expected through the i) decommissioning of end-of-life (EoL) wind turbine blades, ii) retired commercial aircrafts, and iii) mass production of thermoplastic-based composites in the automotive industry. In this work, commercially available carbon-fiber poly(etheretherketone) tape material was considered for the re-design and manufacturing of two aeronautic demo cases by using different processing technologies, including hot-stamping with over-injection (demo1: panel) and compression molding (demo2: bracket). The generated CFRPEEK composite wastes, from the fabrication of these components, were milled and reused for the development of novel compounds using a co-rotating twin-screw extruder. Different fiber sizes and loadings were incorporated into PEEK pellets aiming to study their influence on the mechanical, thermal, electrical, and rheological properties. Fractured surfaces after quasi-static measurements were observed by scanning electron microscopy (SEM) to evaluate the matrix/fiber interface. Afterwards, aiming at studying the potential thermo-oxidative degradation and recycling feasibility of this material, CFRPEEK composites were submitted to different thermomechanical cycles (5, 10, and 15). The most suitable compound, based on experimental results, will be further applied to develop another demo case by injection molding (demo 3: bracket). Lastly, the three demo cases will be assembled by using ultrasonic welding technology, forming an aircraft window frame structure.

Oral (S12-710, Time: Tuesday 16:40, Room: Bruker AXS)

Chemometrics and Machine Learning Techniques for Polymer Processing: Sustainability and Quality Improvements

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Scaled-up polymer processing requires more efficient process analytical technologies via chemometrics and machine learning. This work demonstrates the usefulness of chemometrics and machine learning techniques for the applications of (i) a polymerization unit for polyester products and (ii) polyamide fibre manufacturing process. For multi-step polymerization units, imprecise switching of phases can waste a significant amount of time and energy for the unit, which gives negative plant sustainability and costs. Additionally, automation for phase switching is rarely used due to the challenges of batch-to-batch variance, sensor instability, and various process uncertainties. Here, we demonstrate that by using an approach which includes optimized noise removal methods and neural architecture search, the real-time reaction completion could be precisely tracked. A novel phase switch index is proposed to act as a digital phase switch and is shown to be capable of reducing total reactor operation time. The combined analytics gave a potential of 5.4% reaction batch time saving, 10.6% reaction energy savings, and 10.5% carbon emissions reduction. For the operator, this method also saves up to 6 h during the end discharge of the reaction. For polyamide fibers manufacturing process, we demonstrate the usefulness of chemometrics and machine learning via data fusion of process information and portable spectroscopy. A recipe-based pre-processing optimization algorithm was used to predict mechanical properties of polyamide fibre products under different processing conditions. Pre-processing optimization reduces data artefacts in portable spectroscopy and improves the quality monitoring of polyamide fibre manufacturing process. Furthermore, the technique can be applied in a multi-block manner to select effect spectroscopy techniques (IR, NIR, Raman, etc.). This work demonstrates the high-value applications of chemometrics and machine learning towards polymer processing, giving positive contributions to sustainability and quality improvements.

Oral (S12-723, Time: Wednesday 16:40, Room: Bruker AXS)

Polymer composites based on non-metallic fractions of electronic waste materials as fillers

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The rapid development of electrical and electronic equipment has given birth to the vigorous development of printed circuit boards (PCBs), exacerbating the waste problem. PCBs are one of the most challenging cases for recycling as they consist of polymers, metals, and glass fiber. In this work, PCBs were recycled and re-used as potential hard fillers for polymer composites. The metallic part of the PCBs was leached out and the non-metallic fraction was ground to a particle size smaller than 250 µm. Polypropylene/PCB composites were prepared by introducing 10 to 30 wt.% of ground PCB using an internal batch mixer at 200 °C at a rotor speed of 100 RPM for 5 min. Surface treatment was performed using a silane coupling agent, (3-aminopropyl) triethoxy silane, in the liquid phase to promote the compatibility of the filler with the polypropylene matrix. The surface-treated particles were washed with hot ethanol, dried in a vacuum oven at 40 °C for 24 hours, and thermogravimetrically analyzed to confirm and quantify the surface treatment. The presence of amine groups was confirmed using Fourier transform infrared spectroscopy. Rheological studies of polypropylene/PCB composites in shear strain sweep tests showed higher viscosities and dynamic rheological moduli for higher filler loadings, indicating higher network formation and more dissipation in the composite for both treated and untreated particles. Moreover, polymer composites with silane-treated particles showed higher viscosities than the composites with untreated fillers, revealing a better binder-filler interaction as a result of silane surface treatment. The impact resistance of polymer composites was studied for treated and untreated fillers and compared with pure polymer. Although adding the ground PCBs reduced the impact resistance of the polymer, composites based on the silane-treated fillers were still comparable with pure polymer, showing there is a promising solution for recycling and reusing PCBs.

Poster (S12-004, Time: Thursday 17:00, Room: Foyer)

Textile Sustainability by Up-cycled Clothing: Economic and Environment Impact

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Nowadays living without clothing is approximately impossible. Although clothing indicator of personality and social status of person, as well as provide comfortable and protective situation for wearer. Also one of the most important industries in the world is textile and fashion industries because of their world economic and global environment impacts. In the textile production cycles, to produce a short time used clothes extracted huge amount of non-renewable resources and then due to clothing underutilization and the lack of recycling are mostly landfilled or incinerated. Furthermore, the annual greenhouse gas emissions from textiles production, hazardous substances, releasing plastic microfibers and contributing pollution in the environment, indicate the catastrophic outcomes in future. Fibers and fabrics are kept and never ending up as waste, after that they re-enter the economic cycles. The current proceedings should be concentrated on all aspects of sustainability with the global approach; such an approach makes a purpose of new textile economy for all of stakeholders.

Poster (S12-026, Time: Thursday 17:00, Room: Foyer)

Recycling of waste tire by pyrolysis to recuperate carbon black: An alternative reinforcing filler

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Thermal pyrolysis of waste tires is an industrially beneficial method for material and energy recovery. Pyrolytic carbon black (CBp) is considered to be the secondary main product of this process. An eco-friendly and economically feasible autoclave reactor-based thermal pyrolysis method has been employed for the pyrolysis of waste tire tread. The average yields obtained for CBp and pyrolytic liquid are 48.2 wt% and 46.3 wt%, respectively. The detailed characterization of pyrolytic carbon black fillers and the comparison of their properties with that of commercially available carbon black (mainly N330) was carried out. The thermal properties were studied by thermogravimetric analysis (TGA) whereas, XPS and XRD were employed to investigate the surface characteristics. This study includes several conventional analyses like CHNS, DBP oil absorption, Iodine adsorption, and pH measurement for better comparison between the black samples. The CHNS analysis shows that CBp has higher sulfur and lower carbon content. Furthermore, FESEM and TEM analysis reveals surface attachment with ash in CBp which is further justified by TGA results. Zinc oxide (ZnO) plays as an accelerator in sulfur vulcanization and is added during the compounding stage of the tire manufacturing process. XRD analysis confirms the presence of ZnS on CBp surface converted from ZnO during the high-temperature conversion process as reported previously by other researchers. It is anticipated from obtained results that pyrolytic carbon black will offer a cleaner and more sustainable alternative filler for rubber products relative to conventional blacks.

Poster (S12-334, Time: Thursday 17:00, Room: Foyer)

Assessment of the Environmental Stress Cracking Resistance (ESCR) of Recycled High-Density Polyethylene (HDPE)

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Environmental Stress Cracking Resistance (ESCR), an essential property for packaging materials such as High-Density Polyethylene (HDPE), considerably decreases in recycled materials. This study aims to determine the effects of some possible root causes of the decrease in ESCR of recycled HDPE. The effects of reprocessing, the addition of Polypropylene (PP), and the molecular weight distribution of HDPE on ESCR were investigated. Design of experiments and analysis of variance were employed to statistically evaluate the results. Samples were prepared by melt blending and sheet extrusion. Lab-scale ESCR testing was performed on notched specimens and compared with mechanical and rheological analysis. The results showed that the presence of an HDPE grade with monomodal molecular weight distribution, as well as the addition of low levels of PP (less than 3 wt.%) have a negative effect on ESCR. Moreover, reprocessing reduced the ESCR drastically (66% after 3 extrusion cycles), and chain branching/crosslinking reactions were dominant.

Poster (S12-361, Time: Thursday 17:00, Room: Foyer)

Influence of sample preparation of post-consumer and virgin polypropylene on the reproducibility of oxidation induction time and oxidation induction temperature

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Regardless of whether recycled material or virgin material is used, polymer processing companies depend on an economically advantageous first evaluation of the incoming material. The "oxidation induction time/temperature" (OIT), measured with a differential scanning calorimeter (DSC), can be used to classify the different feedstock materials in terms of thermo-oxidative stability. Polymers investigated are thermally treated in the presence of oxygen and an exothermic reaction is recorded in watts over time (isothermal) or temperature (dynamic). However, the correlation of stability with a time- and temperature-dependent degradation is not always reliable, because the OIT is significantly dependent on different sample preparation techniques. The ISO-11357-6 OIT standard [1] has proven to be insufficient in terms of validity and reproducibility. The aim of this work was to investigate the OIT of isotactic polypropylene, both isothermal and dynamic, concerning the influence of sample preparations. The experiments include different preparations of sample geometry, available product state (injection moulded/ extruded component), sample-to-pan surface area as well as preparatory DSC-measurement programs deviating from the ISO norm standard. These experiments show a wide range of results even though only the preparatory measures of the sample were modified. Concluding the arguments presented, the inadequacies of the OIT standard should be queried, considering their fluctuating outcomes. However, several approaches for an improvement of reproducibility materialized, during the OIT research and will be presented as a norm-standard alternative for a valid polypropylene characterization concerning oxidative stability. [1] DIN EN ISO 11357-6:2018-07 Plastics - Differential scanning calorimetry (DSC) - Part 6: Determination of oxidation induction time (isothermal OIT) and oxidation induction temperature (dynamic OIT) (ISO 11357-6:2018)

Poster (S12-457, Time: Thursday 17:00, Room: Foyer)

The effect of residual polyethylene on mechanical and rheological properties of recycled Polypropylene

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Polyolefin materials (polyethylene (PE) and polypropylene (PP)) are at the top of the world's plastic list and are the largest components in municipal waste streams. Different types and amounts of PP residues exist in recycled PE and vice versa. This type of residue in recycled polymers is a prominent factor that limits the recyclates' applicability. Therefore, knowing the exact contamination amount is crucial to predict its processability and mechanical properties. In this regard, a nominal amount of PE or PP content in their recyclates is acceptable to assure the recyclate quality. To this moment, many researchers benefited from various experimental methods to measure recycled materials compositions. One of the successful methods which are employed to analyze the recyclates is differential scanning calorimetry (DSC), which by measuring the melting enthalpies, one can estimate the type and amount of residues in recyclates. Moreover, spectroscopy analysis techniques like Fourier Transform Infrared spectroscopy (FT-IR) and Nuclear magnetic resonance (NMR) are widely employed to measure contamination. For this reason, in this study, the amount of PE residues in PP recyclates is quantitatively assessed by DSC and FT-IR methods and compared to virgin homopolymer PP with high crystallinity. The amount of PE calculated by these methods is between 3 – 6 %, which is acceptable for recycled PP. It is believed that the presence of PE residues in PP recyclates has negative impacts on some properties of recycled PP. However, recycled PP showed more thermal and environmental stability compared to homopolymer PP. The virgin PP showed a higher rate of thermal degradation and higher sensitivity of viscosity to shear rate, temperature, and inert environments. Furthermore, we assume that PE content enhances the toughness of PP recyclates up to 55%, which is measured by the Charpy impact test.

Poster (S12-541, Time: Thursday 17:00, Room: Foyer)

Recycled tire fibers as reinforcement in elastomers

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The global rising concern of decreasing non-biodegradable wastes accumulating in landfills has led to an increasing demand for polymer recycling. In this regard, recycled tire rubber is one of the best examples, which can be used for various products such as speed bumps, car bumpers, barricades, recreational areas, mats, etc. However, only 65% of recycled tire is crumb rubber, the other 20% and 15% of tires are steel and waste textile fibers (polyester/nylon), respectively. While textile fibers have the lowest content, it has the lowest density, takes up more landfill space and is stored forever due to its non-biodegradability. Even though a large number of articles are available on recycled ground tire rubber (GTR), very few have focused on recycled tire fibers (RTF) due to processing hurdles. For example, considerable amounts of rubber particles is still attached to the RTF that cannot be easily separated. Therefore, this study proposes an approach to determine the RTF compositions using thermogravimetric analysis (TGA)/calcination. Then, the RTF was used as a reinforcement in thermoplastic elastomer composites such as ethylene-vinyl acetate (EVA), polybutylene adipate terephthalate (PBAT) and polyolefin elastomers (POE). The promising results obtained from this study offer possible applications for RTF reinforcements as their addition to the elastomeric matrix substantially improved the tensile and flexural moduli, while limited decrease was observed in the strengths. Furthermore, the effect of RTF content on the mechanical and physical properties was related to the molding conditions (compression vs. injection).

Poster (S12-711, Time: Thursday 17:00, Room: Foyer)

Investigating the effects of hybrid phosphorus flame retardants on the mechanical recycling of PET fibers

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It is challenging to recycle flame-retarded PET fabric waste, and one of the reasons is the decline of mechanical properties and flame retardancy. This study focuses on the mechanical recycling of PET fibers containing phosphorus flame retardants, which is significant regarding the recycling of halogen-free flame retarded thermoplastics. Two phosphorus flame retardants, DOPO-PEPA and Aflammit PCO 900 (AF) were chosen as additives for PET fibers. DOPO-PEPA was proved to own lubrication effects on the melt viscosity of PET, while AF showed polycondensation/branching effects in the long-term rheological characterization tests. PET/FR compounds containing DOPO-PEPA and/or AF were produced in the mini-compounder. Repeated extrusion cycles were carried out to study the processing behavior and recyclability of PET/FR compounds. Following this, mechanical testing was done on the compounded and recycled PET/FR fibers to demonstrate the recycling potential. More chemical, thermal, and rheological tests including GPC, NMR, and ICP-OES were implemented in this study. In addition, a molecular understanding of phosphorus chemistry in the FRs was attempted using density functional theory calculations and molecular dynamics simulations.

Keynote (S13-310, Time: Wednesday 10:15, Room: Mettler Toledo)

Flame retardant modification of PA6 via reactive extrusion

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Though reactive extrusion is a well-established technology in polymer industry to synthesize or modify polymers, it is rarely used to flame retard polymers. This due to the fact that the reactive extrusion process is complex and lack of availability of suitable reactive flame retardant components. Flame retardation of polymer via reactive extrusion process can be considered sustainable approach as it avoids the use of organic solvents and reduces processing steps. In this presentation, our approach to flame retard polyamide 6 (PA6) via novel reactive extrusion process will be discussed. Divinyl phosphine oxide and piperazine as suitable reactive monomers was reacted via in situ Michael addition reaction to form physical networks of phosphine oxide macromolecules inside PA6 during the extrusion process. The extruded PA6 was chemically characterized, analyzed for rheological, thermal and fire properties. The modified PA6 was further converted into textile fibers and analyzed for leaching behavior and mechanical performance. In addition to earlier stated advantages, via such approach migration and leaching out of the flame retardants in thin film and fiber applications can be avoided.

Keynote (S13-689, Time: Wednesday 13:45, Room: Mettler Toledo)

Upcycling marine waste

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Synthetic plastics have joined all sectors of human activity since the early XX century, entering our daily routines and taking over from traditional materials. Their low density and characteristics such as electrical resistivity, mechanical and barrier properties, together with the low cost and easy processing, made plastics ubiquitous. The vast volume of plastic wastes abandoned in the environment or loaded into wastewaters is carried into sewers and streams, ending up in larger rivers and finally into the ocean. The European Green Deal published in 2019 commits to the implementation of a circular economy action plan with a particular focus on plastics recycling. The circular economy approach implies a balance between production, use and controlled end-of-life of the plastics, where recycling will decrease the waste plastic towards zero. However, the plastic waste that has been accumulating along the past decades without control is a huge problem for which no absolute solutions could be designed yet. Recycling is the process in which waste is collected, separated, cleaned, and reprocessed to produce a new product or become a secondary raw material. More recently, a novel kind of approach is emerging, which is upcycling rather than recycling. This new process allows us to transform plastic waste into value-added materials. Furthermore, it plays a critical role in environmental conservation, answering how to overcome the problem of plastic waste in marine environment. Thus, this work investigates and develops solutions by direct incorporation of different amounts of plastics in marine into the same polymeric matrix by reactive extrusion. Different compatibilizers and blends with dissimilar compositions were prepared from marine waste and then characterize. The results show that it is possible upcycling marine waste.

Oral (S13-080, Time: Wednesday 10:40, Room: Mettler Toledo)

Radiation cross-linking of polyamides for highly loaded structural parts

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Highly stressed injection molded parts like e.g. gear wheels are currently often made of standard polyamides. However, the properties of commercially available materials sometimes show an insufficient performance. According to the research publications, materials or parts with better fatigue and wear resistance can be realized e.g. by increasing the molecular weight or by cross-linking. Especially cross-linking by electron beams (β-radiation) is a promising and rather simple way to improve relevant mechanical properties and parts performance. Nevertheless polyamides show some barriers in processing of adequate compounds and yielding whished results. This presentation gives indication how to circumvent these barriers. A case study shows own results for an application.

Polyamides like PA6 or especially PA66 cannot be crosslinked without additives. Typically triallyl-cyanurate (TAIC) based, reactive agents have to be added in percent-range to reach a >40% interconnection rate after β-radiation of >=100 kGy. To avoid losing to much of reactive groups for crosslinking of especially PA66 it is important to carry out the compunding and injection moulding process the right way. That means special guidance must be worked out for both processes.

To analyse the performance of this technology injection moulded test samples were mechanically characterized (standard tensile test, impact tests, dynamical stress tests) and gear wheels were tested. Furthermore chemical and microscopic analysis were performed, to evaluate the potential of the unique behaviour of such radiated material.

To reach high performance in heavily stressed applications, tailor made materials have to be developed, and interdisciplinary interactions between teams from material researchers to application specialists and sales are necessary.

Oral (S13-157, Time: Wednesday 11:00, Room: Mettler Toledo)

Acylated industrial lignins as new biobased antioxidant additives for polypropylene stabilization

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Due to its versatility and low cost, polypropylene commercial demand is increasing, however since it is sensitive to oxidation, antioxidant stabilizers are needed to decrease damages during formulation and use, and thereby increasing the long-term stability of the polymer [1]. Lignins, complex and heterogeneous assemblies of phenolic oligomers in planta, representing 15-30% of the dry weight of vascular plants, are recovered as the major byproducts of lignocellulosic biorefineries [2]. The presence of numerous phenolic groups in lignins grants them a valuable antioxidant activity that may be valorized in industrial applications as biosourced additives [3]. In this work, we want to study the incorporation of industrial soda lignins as additives in polypropylene (PP) and investigate the antioxidant protection and mechanical properties of the resulting material. The main problem relies however in the poor affinity of lignins with apolar polymers, leading in general to a very heterogenous distribution within the polymer matrix. To circumvent this problem, lignins were selectively acylated with fatty acids (C6 and C18) to improve their compatibility with apolar matrices, while preserving the phenols free, and so their antioxidant activity. Raw and modified lignins were thereafter blended with PP in different amounts (1 to 3 wt.%) through two different processes: internal mixer and twin-screw extruder. Lignin/PP films were obtained by compression molding, and they were characterized by optical (or fluorescence) microscopy, then by dynamic mechanical analysis, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC) under nitrogen, before determining their oxidative stability under oxygen flow. The results demonstrate that lignins delay polypropylene oxidation and that the protective effect increases not only with lignin content but also through fatty-acid acylation. [1] Chung, T. C. M. (2019). doi.org/10.1021/acs.macromol.9b00855 [2]Obydenkova, S. v. et al (2019). doi.org/10.1016/j.biortech.2019.121805 [3] Vachon, J. et al. (2020) doi.org/10.1016/j.jcomc.2020.100044

Oral (S13-206, Time: Wednesday 11:20, Room: Mettler Toledo)

Importance of PLA Grade on Its Reactivity with Joncryl Chain Extender

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Polylactide (PLA) is an aliphatic polyester that is bio-based, biodegradable and has good mechanical properties to replace commonly used petroleum-based polymers such as polyethylene terephthalate (PET) and polystyrene (PS). Even though PLA has promising properties, it also suffers from low melt strength, slow crystallization kinetics, high amounts of degradation during processing. To compensate for these shortcomings, chain extension and branching via reactive processing has been shown to be a good option without altering the final properties of PLA, Joncryl ADR being one of the most promising among these chemical modifiers. In this study, five different PLA grades with various copolymer content and molecular weight (Mw) were reactively processed with two different Joncryl to clarify the effects of PLA grade on its reactivity. Samples were processed using internal melt mixer to closely monitor the processing times and were analyzed using small amplitude oscillatory shear (SAOS) and differential scanning calorimetry (DSC). Rheological experiments illustrated that low Mw and low copolymer content allowed higher reactivity with Joncryl while providing less increase in complex viscosity when compared to high Mw and high copolymer content PLA. Low Mw provides more end groups to react with Joncryl but weaker branch interactions while high Mw PLA had lower reactivity but dramatic increase in melt properties. Also, low copolymer content allowed higher complex viscosity in neat samples but high copolymer content showed higher complex viscosity with Joncryl. The DSC analysis revealed that addition of Joncryl lowered the crystallization and melting temperatures while also reducing the overall crystallinity which was more pronounced in low copolymer PLA than higher ones. In lower addition percentages, high functionality Joncryl had more dramatic effect on both rheological and thermal properties.

Oral (S13-302, Time: Wednesday 11:40, Room: Mettler Toledo)

Developing polymeric low-pressure hydrogen storage tanks for mobile applications

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Hydrogen is recognized as a significant potential energy source and energy carrier of the future. On the one hand, storing hydrogen is a challenging task due to its low volumetric density, on the other hand, a particular type of hydrogen in the form of a liquid can be used to store large quantities of hydrogen at ambient pressure and temperature in thermoplastic tanks. But storing hydrogen (in liquid form) for a longer time in polymer tanks affects the physical and chemical properties of the liner. In the current automotive industry, high-density polyethylene (HDPE), polyamides, and some thermoplastics were already been used in existing fuel tank applications. However long-term exposure to fuels leads to the permeation of hydrocarbons into the plastics and resulting loss of mechanical properties. Additionally, facing material shortages and limited supplies of resin leads to an increase in the cost of the material. Therefore, alternative material is searched especially for hydrogen fuel tank applications. In this study, we compared two semi-crystalline thermoplastics, which were exposed to a selected liquid organic hydrogen carrier (LOHC) at 25°C and 60°C for 100 to 500 hours in an enclosed chamber, regarding their mass uptake in these materials. A short analysis is also carried out using differential scanning calorimetry (DSC), and thermo-gravimetric (TGA) to know the influence of LOHC. A surface modification treatment, organic fillers that act as a barrier are also applied on these samples in order to avoid the diffusivity of liquid hydrogen into the polymers. In future work, a suitable manufacturing process is to be chosen (injection molding, blow molding) in a cost-effective manner on replaceable thermoplastic tanks in high volumes for mobile applications. At the same time, these tanks are suitable for recycling and reuse at the end of the product life.

Oral (S13-309, Time: Wednesday 14:10, Room: Mettler Toledo)

Enabling reprocessability and recyclability of epoxy thermosets via reactive incorporation of phosphonate moieties

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Increasing amount of fossil-based plastic waste ending up in our environment is one of the most pressing issues of the oncoming decade. Thus development of re-usable polymers with a prolonged useful lifetime heralds the switch for a transition towards a more circular economy. We design and synthesized novel dynamic phosphonate ester bonds rich thermoset via solvent-free one-pot and two-step synthetic pathway, which demonstrated exceptional reparability, recyclability and flame retardancy. The covalent incorporation of phosphonate in the thermoset matrix introduced sufficient dynamic P-O ester bonds, and promoted the exchange of network strands under moderate heating condition, resulting in scratch reparability and recyclability. Due to the presence of phosphorus in the structure, the thermoset also exhibited excellent flame retardancy in fire tests. Based on the multi-functionality, the application of the thermoset as fire protective coating on wood samples and manufacturing fiber reinforced composites have been explored.

Oral (S13-321, Time: Wednesday 12:00, Room: Mettler Toledo)

Effect of Different Compatibilizers on Mechanical and Thermal Properties of Biodegradable PHB/PLA blends

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Biodegradable polymer blends and composites are used extensively in different industries like packaging, medical devices, and consumable products. Polyhydroxybutyrate (PHB) is one of the biodegradable plastics which has interesting characteristics in terms of biodegradation time, non-toxicity, and good tensile strength that is relatively the same as commonly used polyolefins. Polylactic acid (PLA) is widely used in different industries, especially in additive manufacturing. However, these biodegradable plastics suffer from relative brittleness which limits their applications. Therefore, different modification methods like compatibilization are used to achieve desirable properties. Based on the weakest mechanical properties over PHB/PLA blend compositions, a PHB/PLA blend with 75/25 blend ratio was selected and melt blended with 5 different reactive and non-reactive compatibilization systems including Poly (ethylene-co-glycidyl methacrylate) (E-GMA), Polybutylene adipate terephthalate (PBAT), malleated PBAT (MAPBAT), in order to achieve the best mechanical properties. Differential scanning calorimetry (DSC) analysis showed a decrease in crystallinity and crystallization temperatures in all compatibilized blends compared to PHB and non-compatibilized PHB/PLA blend. E-GMA is a copolymer with a highly reactive glycidyl group which could react with ester groups in PHB, PLA, PBAT and Maleic Anhydride (MA). Through the reactive mixing, the miscibility coupled with interfacial tension between two components improved. Consequently, toughness and tensile strain at break of the reactively compatibilized blends increased significantly. Due to the synergistic effect of E-GMA as a reactive compatibilizer, the PHB/PLA/MAPBAT/E-GMA (75/25/2/2) wt.% and PHB/PLA/PBAT/E-GMA (75/25/2/2) wt.% blends showed the highest elongation-at-break, increasing from 6.2% for the neat PHB to 13.8% and 10.7%, respectively. However, other compatibilizers slightly increased the elongation at break of the neat PHB and PHB/PLA blend. Characterization tests like TGA, SEM, and rheometry were conducted on samples to understand deeply the relationship between mechanical performance and blends' structure.

Oral (S13-526, Time: Wednesday 14:30, Room: Mettler Toledo)

Reactive melt processing of heat-shrinking biocomposites

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Heat-shrinking wraps are widely applied as single-use packaging of, for example, magazines, beverage cans and bottles. The polymers conventionally used for these applications are fossil-based and non-biodegradable, such as polyvinyl chloride (PVC) and polyethylene (PE). To lower the environmental impact, it is of interest to improve the competitiveness of biopolyesters, targeting cost and performance comparable to the currently used polymers. In this work, we report a simple and scalable method to produce heat-shrinking biocomposites that are cost-competitive with PVC and crosslinked PE, with the advantages of biodegradability and bio-based content. Poly(butylene adipate-co-terephthalate) (PBAT) was chosen as matrix for the biocomposites, as it is the most growing biodegradable thermoplastic and the most applied in flexible packaging. To increase the bio-content in PBAT and improve its properties, the matrix was blended with wet pulp fibres via reactive melt processing. A water-assisted peroxide-initiated crosslinking led to around 50 wt.% of insoluble gel, enclosing all the pulp fibres in the 3D PBAT network. Creep tests in the melt state demonstrated that crosslinking induces heat-shrinking in the biocomposites and confirmed their potential as heat-shrinking films. Rheological measurements indicated that pulp fibres and crosslinking increase the melt elasticity and viscosity. In the solid state, the only addition of pulp fibres stiffens and embrittles the matrix, while the crosslinking strategy in presence of pulp retains the 60 % of deformability, doubling the PBAT Young's modulus. The circularity of the biocomposites was proven by the validation of both mechanical recycling and industrial composting as end-of-life options.

Oral (S13-573, Time: Wednesday 15:10, Room: Mettler Toledo)

Designing Hierarchically Porous Graphene Monoliths Using Polymer Blends As a Novel Templating Method

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The rapid development of rechargeable 3D batteries and supercapacitors has given birth to fabrication of high-performance electrode materials as a key part of the energy storage devices. Hierarchically porous, co-continues carbon structure, with bimodal porosity can provide the opportunity for tunning the properties of the electrode. During recent years, several efforts have been made to design hierarchical monoliths of graphene, however the large-scale production remained the important challenge. In this work, immiscible polymer blends with co-continuous morphologies are used as novel templates for isolating hierarchical graphene monoliths. For this purpose, polypropylene (PP) and poly (ethylene-co-vinyl acetate 12 wt%) (EVA12) were chosen as the immiscible polymers to form co-continuous morphology. Graphene oxide was functionalized with long alkyl chains (fGO) for selective localization and well dispersion in one polymer phase. To predict the localization of particles, wetting coefficient was measured and calculated for the system, and results showed that fGO particles have more affinity to the EVA12 phase. A two-step blending procedure was implemented; 10 wt% of fGO particles were blended in with PP phase in the first step for 5 min at 200 oC followed by addition of EVA12 in the second step. The total blending time for the derived polymer blend nanocomposites in a twin-screw microcompounder was 10 min. In the final step, a muffle furnace was used to remove the polymer template at high temperature (500 oC) and achieve the hierarchically porous graphene monoliths. This two-step process is a scalable and cost-effective method that can be easily retrofitted in the existing extruder facilities at large scale. Therefore, this novel templating method showed a promising solution for designing hierarchically structured materials for energy storage/conversion applications.

Oral (S13-650, Time: Wednesday 14:50, Room: Mettler Toledo)

Polymer Assisted Reduction of Metal Salts

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Nanoparticles can offer advantages over their micro-size particle counterparts, mainly due to their large surface area and their specific geometry. However, handling nanoparticles has always been problematic, particularly due to their potential harmfulness. In addition, producing well-dispersed nanoparticles in a plastic composite is challenging, an issue that can limit their application. A novel method to produce nanoparticles is in-situ reduction by reactive extrusion. Here, a metal salt is converted into nano-sized metal-plastic composites during extrusion. Silver-plastic nanocomposites offer several advantages, since silver is known for its excellent thermal and electrical conductivity, catalytic activity, and its antimicrobial ability. In order to create a polymer nanocomposite, silver oxide was reduced to elemental silver in an extrusion process. The use of silver oxide as a precursor is interesting, due to the high metal content compared to other silver salts, since silver oxide can easily be reduced, and because there are no toxic side products formed in the reduction process. Although micron-sized silver oxide powder was co-extruded with different polymers, the resulting polymer matrix contained nano-silver particles. The mechanism of reducing silver oxide to elemental silver in organic matters is still not well understood. This study attempts to explain the reduction mechanism and morphology development of silver nanoparticles. Simple alkanes, alkenes, and alcohol were tested with silver oxide to determine the actual mechanism of reduction. We found that the hydroxy group is responsible for reducing silver oxide, and that ethylene glycol and glycerol can reduce silver oxide at room temperature. When reducing silver oxide in alcohol, we observed a large amount of carbon dioxide, indicating total oxidation of the alcohol. This findings will be used to explain the reducing effect of polymers when converting silver salts to silver nanoparticles.

Poster (S13-333, Time: Thursday 17:00, Room: Foyer)

Effect of Nanoclay to Reduce Undesirable Odor Emission in Recycled Polypropylene

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Although recycled materials are desired to participate in the circular economy to reduce waste, unpleasant odor emissions during processing or use limit the utilization of these materials, especially in indoor applications. Although various commercial additives are used to solve this problem, different solutions are needed. This study investigated the performance of reducing VOCs causing undesirable odor emissions in Recycled Polypropylene(rPP) by using Nanoclay. The result of the jar method, which is a subjective study, revealed that the addition of 5% Nanoclay was effective in reducing odor. Total Volatile Organic Compounds of undesirable odors are determined by a qualitative method called Headspace GC-MS. The results show that the number of high-abundance VOC peaks belonging to rPP was observed as 32. After the addition of 5% Nanoclay to the content of rPP, it reduced to 14 (approximately 50%). Also, it determined that the number of peaks of chemical structures that have an odorous specialty such as 1-Hexanol 3,5,5-trimethyl/Nonilol, 2-Propanol, 1-(2 methoxy-1-methylethoxy), Dimethyl Sulfone decreased. The reason for this decrease is the Nanoclay with a lumen structure with a high surface area provides the function of binding to surface molecules with positive and negative charges on the inside. While we reduce the number of peaks, the new ones belong to chemical structures such as Benzaldehyde and 1-Propanol, 2,2'-oxybis-, Benzamine, 2,4dimethyl, Hydrazine, 1-(5hexenyl)-1-methyl, Triacetin appeared in Headspace GC-MS Chromatograms of 5% Nanoclay containing rPP. These peaks that appear are most probably caused by post-processing of the material and the related studies will be continued.

Poster (S13-465, Time: Thursday 17:00, Room: Foyer)

Flame retardation of poly(lactic acid) with a novel bio-based phosphorus additive

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Poly(lactic acid) (PLA) is a bio-based, bio-compatible compostable aliphatic polyester which is derived from lactic acid. Being derived from a renewable feed-stock, the interest for this bioplastic has increased in the last decade, as an alternative to oil-based plastics such as poly(ethylene) (PE) and poly(styrene) (PS). Ring-opening polymerization via reactive extrusion (REX) technique has been already established to obtain high molecular weight PLA with good mechanical properties1. REX is a "green" process, which allows for continuous production of polymer without the use of toxic organic solvents. However, like most thermoplastics PLA is highly flammable, preventing its widespread use in fire-safe applications such as in the automotive industry and the electrical sector. Herein, we report a novel bio-based, halogen-free phosphorus FR2 additive that when added during the polymerization process, grants flame resistant PLA in a one-step process (see Figure 1). Thermal stability was assessed through thermogravimetric analysis (TGA) and mechanistic insights into flame inhibition process of the additive was obtained through direct insertion probe mass spectrometry (DIP-MS). In addition, polymer stabilization studies were conducted on samples containing the FR to investigate the thermal stabilization effect of the additive.

Poster (S13-508, Time: Thursday 17:00, Room: Foyer)

Controlled Delivery of Carbon Dots for Improved Growth of Crops

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Carbon dots (C-dots) are the new generation fluorescent nanostructures that can serve in multiple fields due to their biocompatibility, tunable photoluminescence, and high water solubility. 1 Furthermore, they have been shown to promote plant growth by increasing light harvesting efficiency. 2 Currently, C-dots are deployed by seed priming, indiscriminately placed around roots or sprayed on leaves. 3–5 The ongoing shift in the agricultural industry towards controlled–release to ensure reliable delivery in a stable environment has yet to be explored for C-dots. So far, the controlled-release systems rely on the encapsulation of a chemical of interest —core— with a degradable polymer shell to allow the delivery of the core over a period of time and protect it from external conditions. However, the carbon footprint of these processes is considerably high due to the need for organic solvents and high temperatures. Accordingly, there is a need for improved and sustainable production methods, and co-axial direct-ink writing (DIW) holds promise for this application. Here, we used co-axial DIW to encapsulate C-dots in a degradable shell made of sodium alginate and polycaprolactone via room temperature, mechanical, green route to fabricate a core-shell system. The release profile of the C-dots through the polymer shell to an aqueous media was tracked by fluorescent spectrofluorometry and electrical conductivity measurements. We placed the designed meshes into the transparent soil to evaluate the effect of C-dots on crop (Triticum aestivum) productivity. The crop with C-dots displayed a faster growth at a degree of 50 % compared to the control sample in four weeks, which proved that C-dots are promising nano-fertilisers. We monitored the uptake of C-dots from the root to the plant body by confocal microscopy. Our results also reveal that the prototyping method presented here is scalable; it has the potential for sustainable, room-temperature fabrication of functional agricultural textiles for controlled delivery. 1 Yang, S. T. et al. (2009), 2 Zheng, Y. et al. (2017), 3 Hu, J. et al. (2022), 4 Li, H. et al. (2018), 5 Wang, H. et al. (2018)

Poster (S13-517, Time: Thursday 17:00, Room: Foyer)

Rheological and thermal properties of PP/EPDM based thermoplastic vulcanizates: "M-POSS as an alternative coagent."

Özkoç Güralp[1], Yazıcı Nazlı[2], Metin Merve[2], Inan Ozgenur[2], Kodal Mehmet[1]

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Thermoplastic vulcanizates (TPVs) are multipurpose materials with two or more phases. At room temperature, they are solid elastomers. They are produced using a method called dynamic vulcanization. They present both the high elasticity of traditional rubbers and the excellent processability and recyclability of thermoplastics. The PP/EPDM TPVs are the most extensively produced TPV type. Since peroxides make crosslinking through both their saturated and unsaturated bonds, they offer a low permanent set and exhibit excellent temperature tolerance. Therefore, they are frequently utilized in the crosslinking of TPV. On the other hand, they have significant drawbacks, such as unwanted disproportionation reactions and side reactions, as well as the beta chain scission of the PP phase. Coagents are used to overcome these drawbacks resulting from the peroxides. By reducing the side reactions, coagents improve the crosslinking effectiveness of peroxide and create bridges between the elastomer chains. The possible use of polyhedral oligomeric silsesquioxane (POSS) molecules with unsaturated bonds and Si-O cage-like skeleton as a coagent to manufacture PP/EPDM-based TPV via dynamic vulcanization was investigated in this study for the first time in the literature. A lab-scale Xplore model twin screw microcompounder was used to prepare PP/EPDM/POSS TPVs system. Methacryl-POSS (M-POSS) was used as a coagent. The rheological and thermal properties of samples were determined via differential scanning calorimeter (DSC) and rheometer, respectively. Moreover, the thermal stability of samples was evaluated by thermal gravimetric analyses (TGA). In a general conclusion, M-POSS was found to be an effective coagent for PP/EPDM TPVs. Acknowledgment This study was granted by The Scientific and Technological Research Council of Turkey (TUBITAK) (Project No: 119M378).

Keynote (S14-226, Time: Thursday 10:15, Room: Mettler Toledo)

Evaluating the molecular weight distribution of linear and branched ultrahigh molecular weight polyolefins by the linear viscoelastic rheology of their solutions

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Ultra-High Molecular Weight (UHMW) Polyolefins are a class of polymers with exceptional properties such as high wear resistance, toughness, and impact strength. For this reason, they are used in many industrial applications. However, the control of the final output of the synthetic process is challenging. For example, the very high molecular weight tail is not detectable by standard chromatographic methods, hindering an accurate determination of their Molecular Weight Distribution (MWD). The situation does not much improve when the evaluation of the MWD is attempted through rheology: extremely long relaxation times and high viscosities often make even the mere sample loading an impossible task. Furthermore, the determination of the longest relaxation times collides with thermal degradation issues deriving from the corresponding long measurement times at high temperatures. In this work, the MWD of ultra-high molecular weight of Polyethylenes (PE) and Polypropylenes (PP) is determined by the combination of linear rheology in solution and modeling. After measuring the rheology of solutions at different polymer concentrations, a time-concentration superposition principle is applied to extend the relaxation spectrum over the largest possible frequency range. Then, molecular models and scaling relationships are implemented to relate the concentration-dependent rheology to the polymer MWD. It is also proven that the same procedure can be applied to the MWD determination of more complex chain architectures, such as in the case of branched polymers. Acknowledgement This work forms part of the research programme of DPI, project #834.

Keynote (S14-345, Time: Thursday 13:45, Room: Mettler Toledo)

Interfacial shear and elongation Rheology towards Unveiling the Effects of In Situ Layer–Layer Interfacial Reaction in Multilayer Polymer Films via Multilayered Assembly: From Microlayers to Nanolayers

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The understanding of rheology and dynamics related to a reactive interphase is extremely important towards modifying interfaces for multilayer coextrusion process. In this talk, a state of the art on the recent advances on multilayer coextrusion and the governed interfacial phenomena will be addressed. the influence of interface modification through in situ layer–layer interfacial reaction during a multilayered assembly is revealed from micro- to nanolayer films, based on maleated poly(vinylidene fluoride) and polyamide-6. In the presence of interfacial reaction and confinement, layer architecture and microstructure are highly dependent on the number of layers. Specifically, for nanolayer films having smaller layer thicknesses and higher reaction extent, layer integrity is reduced with the occurrence of interfacial instabilities. Depending on the microstructural evolution from multilayer assembly, those films exhibit quantitatively different extensional rheological and dielectric properties from micro- to nanolayers. More importantly, dielectric spectroscopy reveals the contribution of copolymer-rich interphases to the dielectric performance of micro/nanolayered films. Additionally, charge transport dynamics in nanolayered films also differ significantly from their microlayered counterparts. They are attributed to the strong dependence of interfacial reaction extent and resulting microstructure on the number of layers and layer thicknesses. This work clearly illustrates how the control of layer–layer interfacial reaction in micro/nanolayer assembly can tune the interfacial, microstructure, and macroscopic properties of multilayered products. J. Rheol., 2016 and Ind. Eng. Chem. Res. 2018, Soft Matter, 2016, 2017, I&ECR 2020 and Macromolecules 2013; ACS Applied Materials & Interfaces, 2018, Polymers 2020, MME 2020, Polymers 2021, 2022.

Keynote (S14-467, Time: Friday 10:15, Room: Mettler Toledo)

The influence of profile extrusion process variables on the heat transfer coefficient at the polymer-calibrator interface

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In extrusion lines for the production of complex thermoplastics profiles the cooling stage takes place in one or more calibrators, where the profile contour geometry is also calibrated through vacuum. This cooling stage is crucial, since it determines not only the profile contour but also its level of thermal residual stresses and morphology, characteristics that clearly affect its behavior in service. Consequently, the performance of a calibration system, which is defined by its design, is a key issue in profile extrusion. One of the most influent parameters affecting the cooling performance of calibrators is the contact resistance at the polymer-calibrator interface, or its inverse: the heat transfer coefficient, h interface. The identification of the most appropriate value of this coefficient is still a challenge, since it depends on several factors, and is also difficult to characterize. Accordingly, values of h interface ranging from 10 to 10000 W/m2K can be found in the related technical literature. This framework hampers the task of obtaining realistic results for the extruded profile temperature distribution, when performing the numerical modelling runs. To overcome some of the above limitations and to obtain additional insights about the process, a prototype calibrator, conceived to allow obtaining realistic values of h interface, was previously designed and presented by this research team [1]. In this work, this prototype is used in a conventional profile extrusion line, and the value of h interface is determined by inverse engineering, by resorting to a 3D heat transfer modeling code available at the OpenFOAM library. Moreover, the influence of several processing variables on the value of this coefficient, namely the calibrator cooling fluid temperature, the applied calibration vacuum pressure, and the pulling velocity, will be investigated. [1] Carneiro, O. S., Nóbrega, J. M., Mota, A. R., & Silva, C., Polymer testing, 32(6), 1154-1161 (2013).

Keynote (S14-663, Time: Wednesday 17:00, Room: Mettler Toledo)

Small-scale extruders for in-process materials characterization

Covas Jose Antonio[1], Hilliou Loic Gilles[1]

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The efficient development of new or improved polymeric systems requires the availability of characterization techniques that are able to provide quickly relevant and accurate data. In-process measuring techniques are frequently adopted for this purpose, as they: i) minimize the time delay between the decision to make a measurement and obtaining the result; ii) avoid the need to subject the material to further thermal/flow cycles in order to prepare samples for the measurements, which may affect its initial characteristics. Using small-scale processing equipment for this purpose has the further advantage of requiring small material quantities, which may be especially pertinent at the early stages of material development. This work presents examples of prototype small-scale extruders coupled to in-process measuring devices that were used to characterize the rheological and morphological characteristics of various polymer systems.

Oral (S14-035, Time: Wednesday 16:40, Room: Mettler Toledo)

Revealing the long chain branching influence on the properties of polypropylene/carbon nanotube systems before and after shear flows in terms of both linear and nonlinear rheology

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Polymer composites with different fillers have been increasingly applied recently because of their lightweight, flexibility, and enhanced functional properties. In addition, the polymer chain structures (linear or branch) show profound influences on processing and filler dispersion. The present work tried to deeply detect the effect of long chain branching (LCB) on the rheological and electrical behaviors of the composites before and after steps shears via transient rheology, small amplitude oscillatory sweep (SAOS) and electrical conductivity measurements. Linear polypropylene (PPC) and branched polypropylene (PPH) were chosen as the polymer matrix and carbon nanotubes (CNTs) as the nanofillers. The SEC-MALLS and rheological tests all confirmed the existence of LCBs in PPH. The percolation threshold of LCB PPH is higher than Linear PPC, which is due to the high viscosity and elasticity of LCB PPH thus resulting in the contribution from the filler network to the apparent rheological properties becoming less critical. A transient shear with different shear rates was imposed on the composites and then SAOS and electrical conductivity measurements were conducted. The liquid-solid transitions of nanocomposites before and after percolation performed differently and were found to be dependent on the step shear rates. For Linear PPC, higher pre-shear rates would cause the breakdown of the fillers network while lower Pre-shear rates would help the agglomerating of nanofillers. Interestingly, for LCB PPH, both higher and lower pre-shear rates resulted in the breakup of the fillers network. These may be due to the high viscosity and elasticity of PPH. The confinement of polymer chains to the CNTs makes it difficult for fillers to move and align thus leading the formed network be destroyed easily under even slow and slight shears. By contrast, in terms of electrical conductivity, similar trends were found for those samples with fewer amounts of fillers. While for composites with fillers amount much higher than the percolation, the electrical conductivity displayed less correspondence with the shear rates, which also reflect a different percolation mechanism from rheology.

Oral (S14-061, Time: Thursday 10:40, Room: Mettler Toledo)

Inverse approach to Calibrate the Non-linear Viscoelastic Model by means of a Bubble Inflation Test on ABS using DIC

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This study addresses the mechanical characterization of ABS material under test conditions relevant to the vacuum-forming process. In order to determine the linear viscoelasticity of the material, a shear oscillatory rheometer was employed to extract the relaxation spectra which is directly used to satisfy the linear portion of the constitutive equation. To examine the nonlinear response of the material, a custom-made bubble inflation test setup was linked to a stereo Digital Image Correlation (DIC) system to measure the large deformation behavior during fast inflation in the range of forming temperature (140 to 180°C). The main goal here was to calculate the thickness of the material at the pole region by measuring the principal strains. A direct thickness measurement using an ultrasonic thickness gauge performed on the pole of the bubble in the final deformed state shows an excellent correspondence to the calculated thickness using DIC. To calibrate the nonlinear part of the viscoelastic model, an inverse approach was adopted based on Finite Element Model Updating (FEMU). The thickness variation at the pole was defined as an objective feed function to optimize the nonlinear parameters iteratively. To examine the robustness of the method, the simulated thickness values are compared with the experimental data across the arc length of the bubble at different times and temperatures. The calibrated constitutive model is afterward implemented into the simulation tool to predict the real industrial vacuum-forming process by knowing the temperature distribution using a thermal camera during forming step.

Oral (S14-109, Time: Thursday 11:00, Room: Mettler Toledo)

Inline quality measurement of preheated thermoplastic composite parts using passive thermography

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Thermoplastic composites play an increasing important role in lightweight design. The composite industry employs several production techniques for thermoplastic composite parts. A novel approach is the so-called thermoplastic composite production cell which consists of five processing steps: (a) tape laying, (b) consolidation, (c) preheating, (d) forming, and optionally, (e) overmolding. In the first step, unidirectional fiber-reinforced thermoplastic tapes are laid down and spot-welded in layers to form a tape stack. Next, the semi-finished product is consolidated using a heating and a cooling press. In the preheating step, the consolidated tape stack is placed inside an infrared oven, which preheats the part above the glass-transition or melting temperature of the matrix. Finally, the preheated material is placed in the mold of an injection molding machine, where the part is formed and optionally overmolded. To optimize process efficiency of the manufacturing chain and increase product quality it is important to gain real-time information of the quality of the intermediates between each processing step. This holds particularly for the preheated plate prior to injection molding. We developed an inline quality inspection method for the preheated part, including (a) a passive thermography camera for the measurement of thermal images and (b) a statistical data analysis approach to predicting critical quality attributes from the pictures. Several experiments with different set temperatures were performed using an industrial-scale infrared oven with consolidated plates made from polycarbonate and carbon fibers. To test our quality inspection approach, we used consolidated plates of different input qualities. The experiments confirmed the usefulness of the approach to assessing the process stability of the preheating step and the quality of the preheated plates.

Oral (S14-118, Time: Thursday 11:20, Room: Mettler Toledo)

Examining the rheological properties of highly concentrated deformable polymer particle suspensions in a polymer melt

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When polymer blends with different softening temperatures are processed in extruders, the order of the melting of the two components plays a crucial role in the transient morphology and rheology of the system. The polymer having the lower thermal transition tends to coat the remaining solid phase polymer pellets or particles, suspending them within the melted phase. When the solid phase comprises a high-volume fraction in the blend, the resulting highly concentrated suspensions can exert a high torque in the melting section of extruders. To model the rheology of these densely filled suspensions, we dispersed polystyrene beads (PS) in an ethylene-vinyl acetate (EVA) matrix at volume fractions ranging from 10 to 70% of PS (PS softens at 107°C and EVA melts at 57 °C). Interestingly, we observe that the viscosity of the suspension does not increase monotonically with PS content but exhibits a local maximum at 48% loading of PS followed by a recovery at 55-60 % PS volume fractions. Both the Mullins and Payne effect play a crucial role in our understanding of these concentrated suspensions. For this study, we used a rubber process analyzer (RPA) for our rheological tests fitted with a bi-conical die. We analyzed the morphology of the melt suspensions via reflective optical microscopy, both prior to the testing and after the testing. The melt-rheology of polymer-polymer suspensions can be correlated to the evolution of morphology in extruders, and this information will be useful to design and tune properties of polymer multiphase materials.

Oral (S14-122, Time: Thursday 11:40, Room: Mettler Toledo)

Investigation of Microstructures of Thermal Protection Systems Materials Using Synchrotron Hard X-Ray Micro-Tomography

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The microstructures of thermal protection systems (TPS) materials were characterized using the Lawrence Berkeley National Laboratory's Beamline 8.3.2 at the Advanced Light Source. The Synchrotron-based Hard X-ray Micro-Tomography instrument allowed for non-destructive 3-Dimensional (3D) imaging of 39 different samples of TPS materials. The tomography voxels were used to reconstruct images using the rendering software ORS Dragonfly. These 3D images are examined and compared to create new learnings about the microstructure differences between thermal protection material systems. Machine learning filters are applied to quantify microstructural quantities and to create visual separation between different materials in the samples. NASA's Porous Microstructure Analysis (PuMA) software was also used to calculate material properties from the samples' microstructure.

Oral (S14-159, Time: Thursday 12:00, Room: Mettler Toledo)

Determination of the Bulk Density of Differently Shaped Plastic Granules as a Function of Pressure and Temperature

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This work deals with the bulk density of differently shaped raw materials, considering shredded film which often occurs in recycling processes, spheroidal, and two different cylindrical shaped polypropylene pellets. The mass flow rate and the pressure buildup behavior of single screw extruders, which are often used for the recycling of polymers, are strongly influenced by the shape of the raw material. These differences arise in the melting zone and especially in the solids conveying zone where the external coefficient of friction and the bulk density in the screw channel influence the conveying behavior. In extrusion applications, higher bulk density usually leads to a higher mass flow rate and a better pressure build-up behavior. In this paper, the bulk density of differently shaped bulk materials is analyzed in detail at three pressures (0.4, 2.0 and 3.5 MPa) and at four different temperatures (20, 60, 80 and 120 °C). The bulk density is needed for the modeling of the extrusion process. The density of shredded film was not investigated in detail till now. It is shown that the density of virgin material and the two different cylindrical shaped pellets behave similarly, while the shredded film differs from the others. At low pressure and low temperature, the bulk density of shredded film is lower than that of the other raw material shapes. Nonetheless, at elevated temperatures and pressures above approximately 1 MPa, the density of the shredded film is higher than the density of spheroidal and cylindrical shaped raw material.

Oral (S14-162, Time: Wednesday 16:00, Room: Mettler Toledo)

Characterization of the Rheological Behavior of Mixed Miscible Polymers with a High-Pressure Capillary Rheometer

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In this work, the rheological behavior of two types of virgin polypropylene and blends of these materials were studied using a high pressure capillary rheometer. Furthermore, it was analyzed, if the viscosity of a mixture of two materials can be estimated from the viscosity of the pure materials by applying mixing rules. Therefore, existing mixing rules were adapted. Mechanical recycling of polymers often requires mixing polymer types with the same chemical structure but different properties, e.g., two different polypropylene grades with diverging melt flow rates. This leads to the question how the physical properties of the blended polymers change. In this paper, the rheological behavior of blends of two types of polypropylene with different weight fractions (0.1/0.9, 0.3/0.7, 0.5/0.5, 0.7/0.3 0.9/0.1) and the pure materials was studied experimentally and modeled by the Bird-Carreau-Yasuda model. Different mixing rules (Heitmiller, Bersted, Friedman and Porter, Tsenoglou) were adapted to consider the shear rate-dependent rheological behavior of polymers. The viscosity of the 0.1/0.9 and 0.9/0.1 mixtures can be described rather well by all models. In the other cases, the experimentally determined viscosity is in general higher than predicted by the mixing rules with deviations below 13 %. A comparison of the applied mixing models shows that the model of Heitmiller predicts the lowest values, while that of Friedman and Porter predicts the highest ones resulting in the lowest mean deviation of only 3.66 %.

Oral (S14-163, Time: Thursday 14:10, Room: Mettler Toledo)

Balancing mechanical properties for the synthesis of polyacrylic casts

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Methyl methacrylate (MMA) has been utilized widely as the main constituent in corrosion casting resins, a technique that seeks to create replicas of anatomical systems [1]. MMA-based resins offer reasonable pot-time and appropriate low viscosity for its application by injection. They also provide good mechanical resistance and acceptable durability after solidification. Nonetheless, there is need for further improvement. This work aims to tune up properties of new acrylic casts resins by modifications in the composition and the manufacturing methods [2]. Bulk free-radical polymerization (FRP) experiments were conducted and the rate of polymerization was inspected. The conversion profiles were compared, allowing to assess the changes and their dependence on the nature of the blend. Four formulations were further analyzed and their rheological data during polymerization was acquired in order to characterize the flow behavior during polymerization. A novel protocol was specifically developed to process rheological data for a polymerizing systems going from low to high viscosity; and similarly, an innovative approach to fabricate molds allowed creating representative specimens for mechanical testing from casting resins with initial low viscosity and undergoing subsequent shrinkage. The casts were fabricated to evaluate their mechanical properties, consequently resistance and durability. From the rheological analysis and mechanical testing it was possible to assess the pot-life and describe the way viscosity changes as polymerization transforms the resin. The mechanical analysis allowed to identify the blend MMA/EGDMA (2% crosslinker) as having the best performance.

Oral (S14-188, Time: Thursday 14:30, Room: Mettler Toledo)

Analysis of material degradation during serial hot gas welding

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Due to short cycle times, gas temperatures of 500 °C or higher are used for welding plastics by means of serial hot gas welding. These temperatures are often distinctly higher than the decomposition temperature of the plastic to be joined. The thermal stresses can lead to chemical degradation and physical aging. This can negatively affect the lifetime of the weld and lead to premature failure. In this paper, the fusion layers of polypropylene and polyamide are analysed in terms of their thermal and thermal oxidative degradation using viscometry and FTIR spectroscopy. The results show that a detection of chain degradation and degradation products in the individual melt layer depths is possible. This characterization represents the first step towards a holistic approach to the influence of thermal damage in the weld as a function of the process parameters and its effects on the long-term properties.

Oral (S14-219, Time: Thursday 14:50, Room: Mettler Toledo)

On finding the fundamental elastic response of polymer melts: polyethylene

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For processing, fabricators using PE need information concerning both melt viscosity and elasticity. As is well known, the zero-shear-rate viscosity is the fundamental material property of a polymer melt describing its viscous response. It depends only on the polymer's structure and thermodynamic variables (temperature and pressure) and is independent of dynamic variables such as shear rate. The corresponding elastic property is the steady-state recoverable creep compliance (SSRSC) measured at vanishing small stresses. It likewise depends only on thermodynamic variables and structure, especially molecular weight and its distribution. Being a linear viscoelastic property, it is not surprising to find that there are several identities relating the SSRSC to other viscoelastic properties, though there are few studies verifying these relationships for polymer melts. Using a 0.18-MI linear low-density polyethylene (LLDPE), we investigated these identities to find how practical they were in determining the SSRSC. For this resin, dynamic data can be used for finding the SSRSC, but data at low stresses are needed and often must be estimated using extrapolation procedures.

Oral (S14-261, Time: Wednesday 16:20, Room: Mettler Toledo)

Rheology in two dimensions - Observing structural changes during processing

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For rheological characterization of viscoelastic fluids, mainly two types of experiments are commonly used: steady shear and oscillation. Steady shear tests give valuable information on the change of the sample´s viscosity during processing conditions, but due to the large strains during such tests, the viscoelastic structure of the sample can be changed during the measurement. Oscillatory tests provide information on the viscoelastic response and the structure at or near equilibrium, which does not correspond to processing conditions. To obtain structural information on viscoelastic fluids under processing conditions, a simultaneous combination of both methods, steady shear and oscillatory experiments, is advantageous. When the oscillation deformation is perpendicular to the steady shear flow, both deformations are orthogonal to one another and can be analyzed independently. With this orthogonal superposition (OSP) technique, information on the viscoelastic material response under processing conditions can be gained for many complex materials but it requires rheometers that can deform the sample in two dimensions and a specially designed double wall concentric cylinder geometry. This technique can give insights into the breakdown of the gel-like, elastic structure of emulsions like cosmetic lotions at higher shear rates. Also, the structure recovery after applying a high shear rate can be observed with this method. Another possible experiment is the so called 2D-SAOS, i.e. the samples oscillate at small strain amplitudes in two orthogonal directions. This is a great tool to investigate anisotropic effects in one experiment.

Oral (S14-263, Time: Thursday 16:00, Room: Mettler Toledo)

Weld strength of thick-walled parts in heated tool welding

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The construction of containers and pipelines is one of the most important areas of application for polymers. Infrastructure projects in the water management sector are carried out with plastic pipes and panels of increasingly large dimensions. In some cases, wall thicknesses of more than 100 mm are already used. However, affected companies sometimes complain about broken weld seams on thick-walled plastic pipes that lead to considerable costs and a loss of confidence in the industry. Usually pipe sections are joined using heated tool welding. In the first investigations it was determined that the laws of similarity for heated tool welding cannot be applied to large dimensions between 30 and 100 mm. Furthermore, the edge areas of the weld test specimens only have a short service life in the long-term tensile creep test, which is the most important analysis method in the sector of semi-finished products. In addition, there are brittle fractures in the joining plane of the weld seams, which is not a typical behaviour for pipe and sheet materials made of polyethylene. Thus it is a sign of poor weld seam quality. The previous investigations concentrated on a variation of the DVS parameters, which did not lead to an improvement in the weld seam strength. However, further work on the subject has now led to fundamentally new findings. On this basis the research goal of developing a new process control for thick-walled semi-finished products is to be achieved in the present study. For this purpose, various hypotheses have been put forward with which it is possible to systematically examine different influencing factors. The focus is on the relationship between the temperature of the weld seam and the joining time under pressure. The overriding aim is to improve the long-term strength of the weld seam, which must not show any local weaknesses if it is to be used in practice.

Oral (S14-265, Time: Thursday 16:20, Room: Mettler Toledo)

PVdF/Organocarbonate Thermoreversible Gels: A Thermal, Rheological and Conformational study

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Polymer gels have a wide range of industrial applications and somewhat complex rheological behaviour, which can lead to significant challenges for processing. Sols and gels of Poly(vinylidene fluoride) (PVdF) and related copolymers, combined with organic solvents, have been characterised to better understand melt flow behaviour. Thermo-reversible gels were analysed by means of differential scanning calorimetry, parallel plate rheology, capillary rheometry, and vibrational spectroscopy; revealing an underpinning structural complexity and some peculiar visco-elastic properties. The rheological behaviour of the compounds was modelled with an 8 or 9 relaxation modes Giesekus differential visco-elastic constitutive model. The melting point depression and the chain configuration transitions of polymorphic PVdF homopolymer and copolymer structures were found to be composition dependent. The solvent mixture acted as a good plasticizer and gelling agent (χFlory ˆ 0.5). It promoted the formation of TTT (all trans, β-PVdF) and T3GT3G′ (Trans x 3 - Gauche, γ-PVdF) electroactive polymorphs during gelation. Oscillatory rheology tests performed over a wide temperature range (20 – 160 ^{0}C) revealed some instabilities occurring during the first gel-sol transition and a hysteretic thermal-mechanical behaviour. A pronounced die swell and, mostly, melt instability were found for mild to high shear rates (25 -2000 1/s) during capillary tests, which also showed a noticeable extent of wall slip for formulations and shear rates of interest for industrial production. These results, unitedly to the peculiar frequency response of the sols, give new inputs for modelling the visco-elastic properties of complex polymer fluids by means of differential constitutive models and therefore enabling CFD simulations with improved predictive power.

Oral (S14-341, Time: Friday 11:00, Room: Mettler Toledo)

Mechanical characterization methods for micro-injection molded specimens in the field of polymer material development

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The properties of plastic components show a particularly strong dependence on the processing history, compared to other material categories. Interactions with part dimensions and structure formation phenomena, such as crystallization, significantly determine the properties. The understanding of these process-structure-property relations is the basis for the development of tailored materials and the exploitation of new applications and processes. Additional challenges arise from political and economical transformations. New raw material sources and circular economy efforts lead to changing initial properties of the raw materials used. Micro-injection molding has proved to be an efficient method for processing and characterization in the material development of thermo-plastic polymer materials. In order to comprehensively describe the material behavior, a micro-tensile test and a micro-impact test were qualified for in-house developed specimens. The test results were compared to standardized tests for various polymer materials.

Oral (S14-380, Time: Friday 11:20, Room: Mettler Toledo)

Influence on rheological behavior of additives for rigid PVC processing with slit die extrusion using inverse method

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Research on the flow behavior of unplasticized PVC compounds remains rather limited even if rigid PVC is widely processed in the polymer processing industry. One major hurdle is that the shear viscosity of PVC is highly influenced by the thermomechanical history during processing. Within the conical double screw extruder, PVC's hierarchical grain structure is broken down and the flow profile is evolving dependent on its gelation degree. For standard thermoplastic polymer melts, shear viscosities are measured using standard rheometers assuming isothermal conditions. While for industrial processing of rigid PVC, viscous dissipation is high and can not be neglected at high shear rate regimes. Besides the remarkable viscous heating effect, rigid PVC also exhibits pronounced wall slippage as a result of the affluent presence of lubricants. A highly instrumented slit die on the extrusion line allows to measure pressure along the die during processing. Due to the non-isothermal manner of PVC processing, a model describing the pressure drop in the dies of different thicknesses requires 2D viscous flow simulations with slip boundary conditions. In this research, power-law and slip parameters (consistency index, power law coefficient, slip coefficients,...) on commercial rigid PVC compounds are estimated. An optimization is set up to minimize the pressure difference between slit die data and the 2D simulation. With the addition of lubricants, we noticed a reduction in extrusion load during slit die measurement, declined viscosity through the plate-plate rheometer test, and enhanced slip velocity from the capillary rheometer test. Such an inverse method, based on slit die experiments, provides a quantitative comparison of viscosity parameters before and after the addition of internal (fatty acid ester) or external lubricants (PE wax), which paves the path for future investigation on the prediction of rheological performance on PVC recyclates boosted with additives.

Oral (S14-469, Time: Friday 11:40, Room: Mettler Toledo)

Determination of polyethylene content in polypropylene pellet blends using near-infrared spectroscopy

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Next to polyethylene (PE), polypropylene (PP) is the most widely used plastic worldwide. Recycling of polyolefins is problematic, since it is challenging to separate polyethylene form polypropylene using sink float separation. Increasing PE content in PP leads to a poor interfacial bond strength, which could lower the mechanical properties of the final molded product. Here we present an efficient method to determine the polyolefin composition, based on near-infrared spectroscopy (NIRS) analysis. The main advantage is the short time to result of less than 1 minute. Mixed polyolefin pellet blends composed of polypropylene matrix and polyethylene between 2 wt % - 50 wt % were produced by compounding using melt extruders. All pellet blends were measured with near-infrared (NIR) spectroscopy in reflection. Chemometric models were created showing high correlation (R^2=0.998) to the weighted values of the polyolefin mixtures. The accuracy of the prediction model was assessed using cross-validation technique and by the prediction of external samples. Near- infrared spectroscopy enables fast determination of PE content in PP pellets, which helps to improve the quality control of recycled raw materials.

Oral (S14-511, Time: Friday 12:00, Room: Mettler Toledo)

Integrative material characterization of CNC (crystalline nanocellulose) reinforced filaments for 3D printing applications.

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Sustainability and green materials are getting more and more interest and significance within the past years. The wish to employ green materials in a wide range of applications and several different products leads to an increasing number of combinations of various bio and or recycled materials and the use of a wider range of production techniques coming into the focus of the scientific community. An interesting example for an interesting bio material is crystalline nanocellulose (CNC), that can act as a reinforcing fiber for polymers. The low density of CNC fibers in combination with its high stiffness leads to a remarkable specific modulus of approximately 90 MPa/(kg.m^3). The 3D printing process (in this case fused filament fabrication – FFF) subjects the processed material to relatively high stresses, both thermally and mechanically. This fact must be considered in material selection, product design as well as process design. For this study, a recycled 3D printing polypropylene grade was reinforced with different CNC fiber contents (up to 15 vol.-%). To increase the adhesion between fiber and matrix which is essential for the reinforcement effect, an aleic-acid-anhydride based compatibilizer was used for fiber coating. In addition to their shrinkage behavior, the bio-composite materials used were investigated in terms of their dynamic mechanical properties, as well as their shear rheological behavior. Against the expectations, the rheological measurements showed a decrease of viscosity of compounds with increasing filler content. This effect occurred together with a change in the sample color, and can be attributed to a degradation of the compatibilizer. This assumption could be confirmed by DSC measurements. Integrating a tailor-made aging process, that simulates the thermal material stress during the FFF process, into the DMA measurement procedure allows to perform a reliable material characterization that allows a prediction of the component properties.

Oral (S14-533, Time: Thursday 16:40, Room: Mettler Toledo)

Composition Processing Properties Relationship of Vitrimers Based on Polyethyleneimine

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Vitrimers are a new polymers' class with thermosets' structure and properties, but with reproducibility. The composition–properties relationship of imine-based vitrimer was studied using an innovative combination of characterization methods. Characterization led to classifying the chemical reactions during processing, affecting the final chemical structure and properties. Using different techniques, we concluded that the reactants and the processing conditions determine the chemical structure of the final material. While heating, three mechanisms affect the composition and properties – transimination, reduction (to amine groups) and oxidation (almost negligible). The reduction processes dominantly affect the mechanical properties: they changed from high-performance to insufficient. Under specific conditions, the imine cross-linking bonds are reduced, which causes the formation of more polar but lower energy cross-links, resulting in lower tensile strength and a change in the polymer's hydrophobicity, which may be used in future applications. Transimination imparts flowability and consists of two mechanisms: exchange and metathesis. Metathesis is activated by heat, whereas exchange is also activated by stress. Metathesis provides low viscosity at higher cross-link density and seems to promote flow at intermediate temperatures; around 150°C, the material behaves as a viscous fluid. Otherwise, the dynamic behaviour is an elastic solid at higher or lower temperatures.

Oral (S14-613, Time: Thursday 15:10, Room: Mettler Toledo)

Effect of molecular structure changes on melt rheological properties of polyamide (nylon 6)

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Tailoring the polymer melt rheology and the chain relaxation dynamics permits easy handling of polymer processing and enables broader range of applications. Novel strategy to control the polymer melt rheology and the chain relaxation dynamics was devised . A simple process for molecular structural change in a polyamide (nylon 6) to easily generate a long-chain branching in a controllable manner without forming a network structure led to unusually large enhancements in the relaxation dynamics. The zero shear viscosity of the polyamide has increased more than 200 folds of linear chains viscosity, whereas the molar mass change was ca. 1.6 times. Storage modulus and the loss modulus at low frequency increased more than 104 and 103 times to those of neat polyamide without forming a network structure. The rheological properties of the polymer (nylon 6) melts can be finely tailored by this simple process to cover a broad range of applications. We also investigated the effect of a change in the molecular structure and ensuing molar mass change of a matrix polymer (polyamide 6, Ny 6) on droplet deformation of a dispersed thermotropic liquid crystalline polymer (TLCP, a poly(ester amide)) in shear flow The total capillary number, including the elastic capillary number, was sufficiently greater than the critical capillary number for deformation of the dispersed TLCP droplets. Both the high viscosity and the first normal stress difference of m-Ny 6 promote the deformation and fibrillation of the dispersed TLCP droplets.

Oral (S14-634, Time: Friday 10:40, Room: Mettler Toledo)

Effect of processing condition on the thermophysical and rheological properties of polyhydroxyalkanoates (PHAs)

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Over the last few decades, packaging has contributed to over 60% of the generated plastic waste. PHAs are bio-based, biodegradable polymers that have the most potential to replace conventional polymers used in packaging. However, the processing of PHAs is challenging due to their thermal degradation and their thermophysical properties' dependency on processing conditions. As interest in the usage of PHAs grows, this study examines the challenges in their physical and rheological properties and their use in packaging applications. In this study, differential scanning calorimetry (DSC) and rheological anaylis were used to monitor the thermal behaviour of polyhydroxy butyrate (PHB) and poly(3-hydroxy butyrate-co-3-hydroxy valerate) (PHBV - featuring 2% valerate content) at different processing temperatures and residence times. Also, their crystallization behavior was studied at a wide range of cooling rates that exist in industrial polymer processes, from 5 °C/min to 200 °C/min. The experimental results showed that PHB and PHBV have different thermal responses as processing temperature and residence time vary. It was noticed that there is a higher chance of degradation and significant viscosity drop due to chain scission at higher temperatures and longer residence times. Also, their crystallization behavior strongly depends on cooling rates. When the cooling rate changed from 5 °C/min to 200 °C/min, the crystallization temperature of both PHB and PHBV shifted from around 125 °C to nearly 100 °C. Also, the degree of crystallinity of PHB and PHBV decreased from around 60% to the vicinity of 50% and below. The Avrami equation modeling was used to describe the isothermal and nonisothermal crystallization behavior of PHB and PHBV. The change in thermal behaviour and crystallization behavior of PHAs highlights the sensitivity of the thermophysical properties of PHAs to the processing conditions.

Poster (S14-086, Time: Thursday 17:00, Room: Foyer)

Impact of low-density polyethylene addition on the rheological properties and processability for bisphenol-A polycarbonate

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Effect of the low-density polyethylene (LDPE) addition on the steady-state shear viscosity was studied using bisphenol-A polycarbonate (PC). It was found that the addition of a small amount of LDPE having low viscosity considerably decreased the shear viscosity of PC. Moreover, the viscosity decrease was pronounced in the high shear rate region, which is different from a plasticized system. The viscosity drop was not originated from the wall slippage but presumably due to interfacial slippage between immiscible polymers. Since the viscosity of LDPE was much lower than that of PC, LDPE dispersion was deformed to the flow direction greatly, leading to a large interfacial area. Furthermore, we also performed injection molding to investigate the effect of the LDPE addition, and found that the warpage of molded products was remarkably reduced. In addition, it should be noted that the injection pressure was significantly reduced from 150 to 110 MPa with an addition of 5 wt. % LDPE. These results demonstrated that the processability at injection molding can be improved by the LDPE addition.

Poster (S14-186, Time: Thursday 17:00, Room: Foyer)

In line Rheometrical Slit Die for in Process Monitoring and Extrusion of Bio Nanocomposites

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Biomaterials must offer functional properties for high-volume applications and better performance than fossil-based materials to be accepted by the industry and end users. The transformation of biobased materials to the nanoscale answers this challenge, providing them with new properties required in many sectors. However, the ‘nano effect’ is intimately related with the extent of the dispersion of the nano additives in the polymer matrix. Thus, monitoring the level and homogeneity of the dispersion during the manufacture of bio nanocomposites is central to the production of materials with adequate performance. Coupling instrumented slit dies to extruders for in-line measurements seems particularly promising. Not only shear viscosity becomes accessible, but measurements are done at relatively higher shear rates. As with most similar attempts, the generation of a range of shear rates implies operating the extruder with varying screw speeds or feed rates. However, this changes the thermomechanical experience of the material inside the machine and, henceforth, may change the characteristics of the material, which in turn may jeopardize the validity of the measurements. To circumvent this problem, a design concept that has been seldom utilized, consisting of a die with two parallel identical slits, each fitted with an output control valve at its inlet, was adapted. The solution, developed under the Bionanopolys Project, that allows independent rheological measurements and extrusion while maintaining constant the extrusion conditions is validated and discussed. Bionanopolys intends to strengthen the circularity of nano-enabled bio-based materials in the economy launching and promoting an open innovation test bed to develop innovative bio nanocomposites from main feedstocks in Europe and bio-based nanoproducts in relevant sectors. This project has received funding from the European Union’s Horizon 2020 Research and Innovation Programme under grant agreement Nº 953206.

Poster (S14-335, Time: Thursday 17:00, Room: Foyer)

Rheological behavior of soft Thermoplastic Elastomers based on polyolefin blends: Process-oriented representation by rheometric data

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Repeated melt processability is one of the outstanding advantages of Thermoplastic Elastomers (TPEs), as this enables highly efficient manufacturing technologies that provide additional design freedom for the resulting product with elastomer-like application properties. However, the complex multiphase morphology of some TPE groups pose manufacturing challenges because it affects their flow behavior significantly. On the other side, the determination of process-relevant rheological properties needs at first a breakdown of the complex flow to simplified conditions, which comes with several assumptions about the materials flow behavior. Since the available rheometric data of TPE are limited and often exclude the details of the test setup and measurement method, there is little information on whether the assumed boundary conditions are actually met for manufacturing-relevant flow processes, such as capillary flow in a hot runner or extrusion die. In the present study therefore several commercially available TPE based on polyolefin blends, all with a hardness up to 60 Shore A, are investigated. They are characterized using the Small Amplitude Oscillatory Shear (SAOS) and the High-Pressure-Capillary Rheometric (HPCR) testing. The study provides an overview of the process-relevant rheological behavior of TPE and how this can be represented by rheometric data. It therefore indicates how important the experimental boundary conditions are for the significance of rheological data in process optimization and in the design of hot runners, gates, sprues and extrusion dies.

Poster (S14-400, Time: Thursday 17:00, Room: Foyer)

Measurement of the Rheological Properties of HBA/HNA Thermotropic Liquid Crystalline Polymer at High Flow Rate using In-line Twin Bore Capillary Rheometer

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We analyzed the rheological behavior of thermotropic liquid crystalline polymer (TLCP) composed of p-hydroxybenzoic acid (HBA, 73) and 2-hydroxy-6-naphthoic acid (HNA, 27) monomers using in-line twin bore capillary rheometer integrated by the single-screw type extruder. The TLCP resin dried at 130 ℃ melted by a extruder of Φ25 and extruded from two bores (two capillaries) by a gear pump at two different temperatures of 295 and 305 ℃. The pressure values were measured simultaneously from the two capillaries with different die lengths under at the high apparent shear rates of 3,700 ~ 18,500 s-1.. Ture shear stress and true shear rate could be calculated using Bagley's correction and Rabinowitch correction, respectively, and the corrected true shear rate increased to higher values of 4,900 ~ 24,900 s-1. It is interesting that the ture shear viscosity (true shear stress/true shear rate) in the capillary showed lower values of approximately 30 ~ 50% than the apparent shear viscosity (total viscosity), indicating that the elongational viscosity at the entry and exit of the capillary is considerably high and may have more influence on spinneret pressure. This characteristic elongational flow behavior was analyzed in detail using Cogswell's equation to understand the structural change of the molten TLCP at the spinneret in industrial melt spinning process.

Poster (S14-432, Time: Thursday 17:00, Room: Foyer)

A study of rheological properties of high molecular weight PET resin by solid state polymerization conditions

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Poly(ethylene terephthalate) (PET) is one of the most commonly encountered polymers, and it is applied to a wide variety of industrial fields such as injection-molded products, films, and textiles. PET products in these fields are applied with PET of various intrinsic viscosity (I.V.) and molecular weights (MW) depending on the manufacturing process. Typically, the I.V. of PET resin at the applications such as clothing fiber, drink bottles, and industrial tire cord are 0.65, 0.7-0.85, more than 0.85 dL/g, respectively. Due to the higher MW, the higher I.V., and the higher I.V., the stronger the fiber in general, in industrial fibers that require high strength, such as airbag fabric or tire cords, high I.V. PET resin is used. In this study, high MW PET was prepared through solid-state polymerization for application to industrial fibers, and its rheological properties were analyzed. The I.V. of basic PET resin was 1.05 dL/g, and it was solid-state polymerized by adjusting the polymerizing temperature, vacuum and purging gas. The rheological properties of the obtained PET resin were measured using a in-line twin-bore type capillaty rheometer systems. The rheological properties were obtained with various spinning conditions, such as controlling the temperature in the vicinity of spinneret, the spinning speed and shear rate. From the obtained data, the corelation among the rheological properties, polymerization conditions and the spinning conditions was analyzed. In conclusion, we could confirm the rheological properties of solid-state polymerized PET via different polymerization conditions by using the in-line twin-bore type capillary rheometer systems.

Poster (S14-501, Time: Thursday 17:00, Room: Foyer)

Effect of Plasticized UHMWPE on Temperature Dependence Dynamic Solid State Relaxation and Rheological aspects of HDPE/UHMWPE Blends

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The extent to which plasticization of ultrahigh molecular weight (UHMWPE) can modify the temperature dependence solid state relaxations under dynamic load at a frequency of 1 Hz and frequency dependence melt state time temperature superposition (TTS) under small amplitude oscillatory shear (SAOS) at a strain of 0.05% and different temperatures such as 145°C, 165°C and 185°C of HDPE/UHMWPE blends were investigated in this work by preparing blends using melt mixing. Fischer-Tropsch (H5) olefin wax is added to prepare plasticized UHMWPE (p-UHMWPE). Dynamic mechanical analysis (DMA) revealed the storage moduli around glass transition temperatures and energy dissipation above temperature of 30°C improved with plasticized UHMWPE contents of the respective blends. Further, the damping factor (Tanδ) reflected the enhancement of elasticity, and confirmed from Cole-Cole plots of the blends containing plasticized UHMWPE. Interestingly, the time temperature superposition principle was applicable only to neat HDPE and blends containing 5 wt. % of neat UHMWPE and 20 wt. % p-UHMWPE among all composition. The melt flow activation energy (EH, KJ/mole) and master curve of corresponding blends using horizontal shift factor were evaluated. The above results suggested that the plasticized UHMWPE improved the interfacial adhesion through chain entanglements and the improved viscoelastic properties was observed at 20 wt. % of plasticized UHMWPE content. Keywords:UHMWPE; HDPE; Wax; Plasticization; Viscoelasticity; Time Temperature Superposition.

Plenary (S15-803, Time: Thursday 08:30, Room: Sulzer Chemtech)

Fungal biorefinery: Upcycling waste to chitin-glucan nanomaterials with a promising future

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Greener alternatives to synthetic polymers are constantly being investigated and sought after. You will have heard about cellulose and maybe also nano(fibrillated)-cellulose, an exciting material strong and stiff extracted from cell walls of plants. Chitin has, however, been largely overshadowed by cellulose when discussing the materials aspects of its nanosized components. [1] Chitin is a natural polysaccharide that gives structural support to crustacean shells, insect exoskeletons, and fungal cell walls. Like cellulose, chitin resides in nanosized structural elements that can be isolated as nanofibers and nanocrystals by various top-down approaches, targeted at disintegrating the native construct. The structural element in fungal cell walls is chitin linked to glucan. Fungal chitin can effordlessly extracted from fungal biomass grown on agricultural, forestry side products or food waste. Fungal chitin forms a ready-made nanocomposite, that could be potentially useful for the development of a single-sourced renewable composite combining the strength of chitin and the toughness of glucan. The chitin-glucan material we will be talking about was extracted from common mushrooms (Agaricus bisporus) and tree bracket fungi (Daedaleopsis confragosa). The reference material is animal chitin extracted from crab shells (Cancer pagurus). We developed a mild extraction process to preserve the quality of the chitin-glucan present within the fungal source. Initially we will compare fungal- with animal chitin and than explore potential applications as films, membranes and leather alternatives for this type of material. We will show that in the case of fungal derived chitins the physical and mechanical properties do depend on its chemical composition.

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Keynote (S15-055, Time: Wednesday 10:15, Room: Bodensee)

Fabrication of electrically conductive PLA composite via secondary polymer-induced particle aggregation and variation of the electrical conductivity during 3D printing

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The performance of polymer composites is significantly depending on particle dispersion and percolation formation. In this study, particle dispersion was manipulated through the addition of secondary polymer with high-affinity to particles and analyzed corresponding changes in electrical conductivity and moduli. A ternary composite of PLA, poly(caprolactone) (PCL) and carbon black (CB) exhibited the PCL-induced CB aggregation when the amount of the PCL was comparable to that of particles. Particle percolation with a high-order structure improves the performance of composite like high storage modulus, high dielectric loss, and negative-positive switching of dielectric constant at high frequency (~ 100 Hz). Most of all, it induced a brittle-ductile transition to PLA different from the expectation of mechanical degradation by particle percolation formation. During 3D printing process of this ternary composite, CB aggregates were rearranged and percolation structure was disrupted under the given flow, resulting in a decrease in the modulus of the composite and lower electrical conductivity. Under high shear through nozzle, assemblies of CB aggregates could not remain close enough to maintain the percolation structure. The addition of secondary polymer makes the aggregates well dispersed, leading to a brittle-ductile transition and significant enhancement of ductility and conductivity of PLA. During additional process, the composite went through re-arrangement of particle structure and could not maintain electrical conductivity (This work was supported by the National Research Foundation of Korea(NRF) grant funded by the Korea government(MSIT) (No. 2020M3H7A1098305, No. 2021R1A2C1004746)).

Keynote (S15-224, Time: Wednesday 13:45, Room: Bodensee)

Biobased polyurethanes with different renewable macromolecular architectures. Towards green applications in the frame of circular bioeconomy.

Averous Luc[1]

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Nowadays, the use of renewable biobased carbon feedstock from different resources, such as vegetable oils, is highly taken into consideration because it offers the intrinsic value of a reduced carbon footprint with an improved life cycle analysis, in agreement with a sustainable development. Besides, compared to conventional fossil-based materials, innovative macromolecular architectures with improved or additional properties can be obtained. In this presentation, we report two decades of active research on the synthesis, characterization and processing of several innovative and renewable polyurethanes (PUR, PIR, TPU and NIPU) (1,2), with controlled macromolecular architectures to elaborate different designs and morphologies (membranes, foams,) (3), for a large range of green applications. These materials are synthesized from different biobased building blocks, which can be directly extracted from biomass or obtained from white biotech (fermentation,): (i) Aliphatic structures from different glycerides and derivate (dimer fatty acids,), sugar-based molecules, bacterial polyesters … (ii) Aromatic structures from lignins (4,5), tannins (6) and furans. A large range of materials with improved properties and durable applications have been developed/synthesized for a greener and durable future. The end of life of these materials is now also considered, by e.g. bio-recycling (7), from cradle to cradle. (1) Tremblay-Parrado, K.-K.; Avérous, L. ChemSusChem 2020 (2) Carré, C. at al. ChemSusChem 2019. (3) Peyrton, J.; Avérous, L. Mater. Sci. Eng. R Rep. 2021 (4) Laurichesse, S.; Avérous, L. Prog. Polym. Sci. 2014, 39, 1266–1290. (5) Duval, A. at al. Mater. Today Chem. 2022 (6) Arbenz A. ; Avérous, L. Green Chem. 2015 (7) Magnin A. et al. ChemSusChem 2021

Keynote (S15-251, Time: Thursday 10:15, Room: Bodensee)

Development of future-oriented biocomposite material from 3-component system in terms of sustainability based on natural/renewable raw materials

Leuteritz Andreas[1], Naseem Sajid[1], Heckel Sandra[2], Al-Ismail Abdullah[2], Zahel Martin[2]

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Conventional plastics are increasingly being replaced by biocomposites because of their environmental problems. Cellulosic fibers have gained attention in preparing biodegradable polymer composites because of its biodegradable nature, ecofriendly, low cost and low weight in comparison to synthetic fibers (glass and carbon). Inorganic fillers with strong polymer affinity could act as a compatibilizer with an enhanced synergistic effect and eventually reducing the need of compatibilizers. In this research work hybrid fillers of cellulose with inorganic fillers will be synthesized for use in biodegradable polymers such as PLA. Hybrid fillers (functional filler) of cellulose can be synthesized using mineral fillers (Talc, CaCo3, TiO2 etc) bound to pulp fibers. Enhanced chemical and physical interaction with such hybrid fillers can be anticipated and by that the failure mechanism of composites changes. The combination of inorganic filler with cellulose fiber generate the hybrid filler and its use in the biodegradable polymer (PLA) would generate the ternary biocomposites system. The aim of this research work is to generate partially or fully biodegradable ternary biocomposites with a hybrid filler system.

Keynote (S15-564, Time: Friday 10:15, Room: Bodensee)

High performance sustainable biopolymers and their blends based on PLA and their filled systems incorporating CNCs. Structure- rheology- forming and properties relationships.

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Through this work, we first present some promising routes to enhance processing ability and performances of which presents challenges mainly due to the poor shear and elongation properties of this biopolymer. To our knowledge, there is few papers dedicated to the investigation of foaming and/or blown extrusion and coextrusion of PLA based systems that involves structural, rheological and thermo-mechanical properties. Three main routes were selected: (i) the modification of its structure and rheological behaviour, (ii) blending it with another ductile thermoplastic biopolymer such as PBAT, PA11 or PHB-V, iii) PLA reinforced by CNCs. Various formulations of PLA with multifunctionalized epoxy, nucleants and plasticizer were prepared and characterized on the basis of their linear viscoelasticity and extensional properties. The balance of chain extension and branching has also been investigated using solution viscosimetry, steric exclusion chromatography (SEC) and rheology (Shear and elongation rheology). On one hand, a batch foaming assisted with supercritical CO2 was achieved. The influence of the foaming parameters, the extent of chain modification as well as the contribution of crystallization on cell morphology was evaluated. On the other hand, the stability maps of blown and coextrusion processing for neat and modified PLA were established at different die temperatures. We have achieved a great enhancement of the blown processing windows of PLA with high BUR (Blow Up Ratio) and TUR (Take Up Ratio) attained. We were able to demonstrate that a higher kinetic of crystallization can also be reached for chain-extended and branched PLA formulated with adequate amounts of nucleants and plasticizers. On another hand we have studied and optimized the processing of multi nanolayers based on PLA reinforced by CNCs. Hence, blown films with interesting thermo-mechanical, mechanical and barrier properties have been produced using an optimal formulation for PLA.

Keynote (S15-633, Time: Thursday 13:45, Room: Bodensee)

Injection moulding of stereocomplex PLA blends produced via melt processing for improved thermal stability

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One of the widely recognised limitations of PLA is its relatively low softening temperature which limits applications to those with low temperatures. One strategy to overcome this limitation is by stereocomplex formation between poly(D-lactide) (PDLA) and poly(L-lactide) (PLLA) enantiomers. The formed stereocomplex crystals have been shown to exhibit improved mechanical and thermal properties. A number of studies of PLA stereocomplex formation have been reported but these have predominantly focused on crystallisation kinetics using processing techniques unsuited to industrial scale production, such as solution casting. In this research, stereocomplex PLA was formed using twin screw extrusion, with process variables such as melt temperature and extruder screw speed examined to determine the optimum conditions. It was found that melt compounding an equimolar blend of PLLA/PDLA at 200°C with a relatively low screw rotation speed of 60 rpm achieved the highest level of stereocomplex formation, as determined by differential scanning calorimetry. Two polymorphs of stereocomplex crystals were observed, with melting temperatures differing by 10°C, influenced by extrusion conditions. The stability of stereocomplex crystals were examined at controlled conditions using capillary rheometry to determine the suitability of the material for further processing. Melt processing at temperatures significantly above the sterocomplex melting temperature were found to result in conversion to homocrystals, although melt processing at lower temperatures was found to maintain the stereocomplex crystal content. Injection moulding of the optimised blend was demonstrated, and samples exhibited mechanical properties similar to highly crystallised PLLA but with significantly increased heat deflection temperature of 140°C.

Keynote (S15-670, Time: Friday 13:45, Room: Bodensee)

Thermoforming process of biopolymer composites with coffee husk

Duarte Fernando Moura[1], Barbosa Sara Costa[1], Leite Rocha Filipa[1], Castro Maria Cidália R.[1], Martins Carla Isabel[1]

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Coffee husks (CH) are the primary waste product of the coffee-roasting process. Despite the growing attention in seeking potential reuse of this material, the majority of CH is commonly used as a fertilizer or then, it is discharged to landfills. Since CH is an organic residue, it can be used to prepare biopolymers composites, aiming to improve its circularity. Innovative applications with differentiated appearance, texture and smell can be made through thermoforming process for packaging industry such as fresh food transport containers. Therefore, the aim of this work is to develop CH biocomposites by extrusion and to study its processability for thermoforming applications. Composite sheets of 5%, 10% and 15wt% of CH and biopolymer Inzea F38 from Nurel, were produced using a Leistritz LSM 30.34 co-rotating twin-screw extruder. Then a thermoforming Formech 450DT machine with a prototype mold was used to study the thermoformability of the composite PLA/CH sheet with different draw ratios (length and area) in the temperature range between 135 ºC and 155ºC. Morphological and mechanical characterization were carried. The results showed a good compatibility and adhesion between the filler and the polymer matrix. F38/CH biocomposites retain a ductile behavior, however a maximum decrease of 40% in the tensile strength and 85% in the strain at break is observed when compared to neat PLA. Finally, an excellent thermoformability of the F38/CH was observed. A length draw ratio of 2 and a areal draw ratio of 4 could be applied for thermoforming temperatures in the range of 135ºC and 155ºC.

Oral (S15-017, Time: Wednesday 11:40, Room: Bodensee)

Rheological and Thermal Evaluation of Polydioxanone and Bioresorbable Polymers for the Optimization of Downstream Processing Parameters

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Bioresorbable polymers are of great interest to the medical sector as a means of invasive interventions, as they can be subjected to degradation in the human body. The increasing prevalence and incidence of chronic wounds is a considerable source of patient morbidity and financial burden to the healthcare system, a field where tunable biocomposites may facilitate a means of ameliorating persistent factors and warranting proximal tissue remodelling. In the development of a biopolymer medical device, there are several material considerations to note such as physicochemical, biological, and degradation profiles. Polylactic acid (PLA) and polycaprolactone (PCL) have been applied in a plethora of medical fields such as regenerative medicine, cardiovascular implants, pharmaceutical delivery, orthopaedic interventions and cancer therapy. Although biocompatible, PLA and PCL have degradation profiles that can extend from months to years. In the development of medical devices, the degradation time of a biomaterial complex should coincide with the regeneration process of proximal tissue to ensure proper remodelling of the tissue. Polydioxanone (PDO) is a synthetic bioresorbable polymer with a short term degradation profile and weak material strength that has been around since the 1980's, yet the majority of its medical application has been as suture material. The limited supply and expensive nature of PDO are considerable factors as to why minimal research has been conducted to date. To address these limitations, independent composite formulations of PDO/PLA and PDO/PCL were produced by solvent emulsion. The physicochemical and processing characteristics of all composites were determined by FT-IR, DSC, TGA, rheology and XRD. Polymers were observed to retain their crystalline nature in FTIR and XRD analysis; however, the rheological and thermal properties of PDO were noted as tunable depending on the percent concentration of PLA / PCL respectfully.

Oral (S15-024, Time: Tuesday 16:40, Room: Bodensee)

High Performance Biopolymer Fibers Derived by the HighPerCell® Process

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The processing of biopolymers, such as cellulose and chitin, using ionic liquids (ILs) as solvents is a promising research field with great potential for industrial applications.[1],[2] With the HighPerCell® process it is possible to produce cellulose and cellulose/chitin endless fibers by wet and air-gap spinning for various applications. Spinning cellulosic fibers based on the HighPerCell® technology is close to pilot production and ongoing pressure to develop environmentally friendly technology will rapidly increase the interest in this technology as there are no additives needed for spinning and a complete recycling of these materials is possible. Based on the flexibility of the IL-based processing of cellulose, there is more interest in developing cellulose-based materials, like all-cellulose composites (ACCs) [3] and super-microfibers. Likewise, ACCs can be used in lightweight construction and as a replacement for glass fiber reinforced plastics. In addition, the exploration of a new class of cellulose-based carbon fibers is made possible by IL-derived cellulosic fibers.[4] This way, poly(acrylonitrile) (PAN) based carbon fibers could be substituted by this ecologically and economically amended alternative.

Oral (S15-052, Time: Wednesday 10:40, Room: Bodensee)

Influence of Addition of PP-g-MA on the Mechanical Properties of Walnut Shell Powder Filled Plant-Derived Polyamide 1010 Biomass Composites

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To develop the all inedible plant-derived materials for new polymeric engineering materials, this study aimed to experimentally investigate the influence of addition of maleic anhydride grafted polypropylene (PP-g-MA) on the mechanical properties of walnut shell powder (WSP) filled plant-derived polyamide 1010 (PA1010) biomass composites. WSP was previously ground the dried walnut shell by crusher machine cryogenically in liquid nitrogen. The diameter of WSP used in this study is less than 53 micron. The volume fraction of WSP as fixed with 10 vol.%. PA1010 was a fully bio-based polymer made from sebacic acid and decamethylenediamine. Maleic anhydride grafted polypropylene (PP-g-MA) was used as a compatibilizer. The composition of the bilend of PA1010 and PP-g-MA was fixed as 98/2 by weight fraction. WSP/PA1010 biomass composites were extruded using a twin screw extruder, and injection-molded. Mechanical properties such as tensile and hardness were evaluated. The polished cross-sectional surfaces of these biomass composites were observed using a scanning electron microscope. We found that the mechanical properties of WSP/PA1010/PP-g-MA biomass composites are slightly higher than those of WSP/PA1010 biomass composites. These may be attributed to the change in the internal microstructure in the composites such as filler dispersion and interfacial interaction between filler and matrix polymer according to PP-g-MA uses or not.

Oral (S15-071, Time: Wednesday 11:00, Room: Bodensee)

Fast biodegradation, and high mechanical properties – a contradictio in terminis?

Krins Bas[1]

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Huge production of polymeric products and their disposal is a global concern currently. Especially for applications in which polymer inevitably ends up in nature, there is a growing awareness that the use of biodegradable materials is beneficial. In many cases this means that the existing application of conventional non-degradable polymer has to be replaced by a biodegradable polymer. For certain applications this means a combination of (relatively) fast biodegradation and high mechanical performance is required. This is very challenging, especially for high-end applications where high mechanical properties are required. In this lecture it will be shown that in general fast biodegradation and high mechanical properties are a contradiction. The results will be discussed using different trials with several biopolyesters and acquired properties. Subsequently, the focus will be put on different grades/copolymers of PHAs in order to understand the background of this contradiction in more detail. The only way to circumvent this challenge is to design smart compounds or new copolymers. Some examples of this approach will be presented.

Oral (S15-073, Time: Wednesday 11:20, Room: Bodensee)

Sugar-Based Polymer - from Reactive Natural Low Transition Temperature Mixtures: Towards sustainable organic binders for carbonaceous refractory materials

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NaLTTMs (Natural Low Transition Temperature Mixtures) are mixtures of natural components found for example in plants, which have a lower melting or glass transition temperature than its constituents taken separately. [1] Academic and industrial researchers have used of these liquids as neoteric solvents in green chemical domains such as extraction of natural actives for food, cosmetic, pharmaceuticals, or as solvents for analytical chemistry, organic synthesis, metal processing... [2] The advantages of NaLTTM are their simple preparation, innocuousness, possible biodegradability and biocompatibility among other. They are composed of H-bonded molecular species and may be obtained by mixing carbohydrates with organic polyacids. We have developed NaLTTM whose constituents have complementary reactive functions leading to biosourced polymers and polymer networks. [3, 4] Furthermore, this new approach allows using these polymers as carbonizable organic binders in carbon/carbon composites used as electrode in alumina electrolysis processes. We present herein results obtained with various [carbohydrate: malic acid] NaLTTM prepared by a mild process. The polycondensation was performed at 130°C as a function of time and the progress of the reaction was characterized by 13C-NMR, MALDI-ToF MS and SEC... Preparation of crosslinked-polymer was monitored by measuring the insoluble content and through dye release experiments. Further thermal treatments at high temperature (from 100 to 850°C) were scrutinized by Thermal Desorption Spectroscopy Mass Spectrometry, IRTF, Raman microscopy and X-ray diffraction showing that carbonization started from 500°C and that carbonaceous residues were produced. The promising properties of Carbon/Carbon composites obtained from these cured NaLTTM matrices allow to consider the replacement of coal tar pitch that are carcinogenic and covered by REACH. [1] Y.H. Choi, J. van Spronsen, Y. Dai et al., Plant Phys., 2011, 156, 1701 [2] C. González, N. Mustafa, E. Wilson, et al., Flavour & Frag. J., 2018, 33, 91 [3] J Clenet, G. Duaux, D. Portinha, E. Fleury Carbohydr. Chem., 2021, 45, 640 [4] G. Duaux, E. Fleury, D. Portinha Polym. Chem., 2021, 12, 548

Oral (S15-074, Time: Tuesday 17:00, Room: Bodensee)

Polyethylene Furanoate (PEF): the next-generation high performance bio-polyester

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The environmental impact caused by fossil resources through carbon-cycle addition and green-house gas emissions represents a matter of primary importance, nowadays. Therefore, the end-of-life cycles of fossil-based plastics are currently extensively addressed by the development of alternative polyesters from renewable resources. Towards a more sustainable future, polyethylene furanoate (PEF) represents a promising 100% renewables-based "green" alternative to its fossil-based counterparts polyethylene terephthalate (PET). PEF exhibits better material properties compared with PET. The non-linear conformation of its structure, and the electrical feature of permanent dipole, allow for a higher gas diffusion barrier for O2, CO2 and H2O, improved thermal stability at ambient temperature, higher glass transition temperature Tg and tensile strength, lower melting temperature Tm and degradation temperature Td. All the above-mentioned features highlight its wide range of applications, not only in bottle blowing for carbonated drinks and thinner bottle walls, but also in high-end applications.

Oral (S15-092, Time: Wednesday 12:00, Room: Bodensee)

Sustainable building blocks for a viable plastics economy - Novel technologies to produce bio-monomers from renewable feedstocks

Geven Mike Alexander[1], Distante Francesco[1], Luk Harris[1], Rima Simonetta[1], Farquet Patrick[1]

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As we are globally facing the challenge of reducing persistent plastic waste, synthetic biopolymers such as polyesters continue to receive major interests for use in the biomedical field, automotive industry and disposable consumer goods. Their degradability and the possibility to produce them from renewable resources makes them valuable candidates for tackling the transition from fossil fuel-based plastics to sustainable alternatives. The large-scale production of monomers for biopolymer production in a sustainable manner is still challenging and novel technologies are required. Meeting this demand, Sulzer has developed scalable technologies to produce bio-monomers from renewable feedstocks. This presentation will outline the scope of these technologies and will show how they integrate in a viable plastics economy.

Oral (S15-095, Time: Friday 14:50, Room: Bodensee)

Moving a step forward from poly(lactic acid) to bio-copolymers

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Subheading: Next generation polymerization process for biopolymers of versatile applications Main text: Polylactic acid (PLA) is a bio-based, compostable, and 100% recyclable polymer produced from renewable feedstocks and is one of the most commercially viable bioplastics nowadays. Beyond the strong and continuous growth of the PLA market, its domain of application can be further widespread by extending the mechanical and thermal properties of the virgin PLA form. Being the global market leader in PLA technology, Sulzer continues to strengthen its core business by offering tailored solutions to produce not only PLA but also other bio-(co)polymers of versatile applications. Herein, the successful PLA story in Sulzer will be illustrated as a base case and the next generation polymerization process for other bio-(co)polymers will be uncovered. The key breakthroughs as well as tools developed in R&D shall pave the road for commercialization and will be outlined in this presentation.

Oral (S15-096, Time: Wednesday 14:10, Room: Bodensee)

Preparation of cross-linked canola protein films by wet casting and compression method

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Canola protein isolate (CPI) films were prepared by wet casting followed by heat compression. It was found that high tensile strength could be obtained under low compression pressure of 1.5 MPa, attributed to the pre-denaturation of protein during wet casting. High temperature (150 °C) could lead to degradation of the CPI matrix, while low temperature ($\leq$ 90 °C) resulted in incomplete melting of the CPI matrix. To obtain CPI films with maximal tensile strength, the optimal curing conditions were 110 °C for 8 min or 130 °C for 5 min. Chemical modification of CPI using bisepoxide as a chain extender markedly increased the tensile strength of the modified CPI (MCP) film. FTIR and DSC results indicated cross-linking occurred, and the reaction kinetics showed that the cross-linking reaction could be finished within 6 min at temperatures from 110 to 150 °C. TGA results confirmed the degradation of the CPI matrix cured at 150 °C; furthermore, the thermostability of the modified CPI films was increased, attributed to the introduction of cross-linked structure in the CPI matrix. Therefore, the optimal curing temperature range to obtain maximum quality of CPI films was broadened by the chemical modification to 110 to 150 °C.

Oral (S15-134, Time: Wednesday 14:30, Room: Bodensee)

Monitoring the enzyme catalysed hydrolysation stage of poly(lactic acid) fibre surface by nanoscale thermal analysis

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Enzymatic modification of the bio-based polylactic acid (PLA) represents an environmentally-friendly route for a controlled surface modification of polymer fibres. In this work, the special combined nanoscale thermal analysis (nanoTA) with atomic force microscopy (AFM) was applied to investigate the topochemical hydrolysis reaction of cutinase from Humicola insolens (HiC) on PLA fibre surface. It can be visualised that the enzymatic hydrolysis preferentially occurs at the amorphous regions of the fibre surface leading to randomised hydrolysis, monomer release and ablation of the fibre surface during the initial phase of the hydrolysis. Since the accessibility of the enzyme to the fibre core is restricted, the change in crystallinity is predominantly on the fibre surface. As a consequence of the higher hydrolysis rate at amorphous regions, the surface crystallinity of the fibre increases leading to an enrichment in overall fibre crystallinity which could be proven by DSC and FTIR measurement on single fibres. Mapping the fibre surface with AFM-nanoTA reveals further detailed information on the hydrolysation stage of the fibre surface. It can be observed that with increasing treatment time, the surface softening temperature as determined by nanoTA increases from the glass transition temperature near to the melting on-set of crystalline PLA. The results support the hypothesis, that selective hydrolysis preferably occurs at the amorphous region at the fibre surface, resulting in modified PLA fibres with an unchanged fibre core and a very thin and highly crystalline surface layer. The study provides a new mechanistic insight in the topochemical hydrolysis of cutinases in heterogenous reactions, where reaction rate is modulated by the order of the polymer chains in the material.

Oral (S15-161, Time: Wednesday 14:50, Room: Bodensee)

Resistance of PLA starch blends to weathering effects

Goetjes Victoria[1], Heim Hans-Peter[1], Gemmeke Nicole[1], Zarges Jan-Christoph[1]

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The use of bioplastics in technical products is currently very limited. The reason for this is, among other things, the high price. By using native potato starch as a filler for poly(lactic acid) (PLA), both the price and the carbon footprint of the material can be reduced, as the extraction of the starch causes significantly less CO2 compared to PLA synthesis. For the application of a blend of PLA and starch, it is essential to be able to make statements about the durability of the material. For this purpose, blends of PLA with 40 and 50 wt.-% starch were produced using a twin-screw extruder. For comparison, a reference was made from pure PLA. Furthermore, test specimens of the materials were manufactured by injection molding. The specimens were exposed to various environmental influences such as in- and decreased temperature (70°C, 23°C, -18°C) or humidity (50%, 90%) for up to 504 h. They were also subjected to 504 h of accelerated weathering. After storage, the mechanical properties were characterized using the tensile test. In addition, the samples were examined by means of micro computertomography (µCT) and scanning electron microscope (SEM). It was shown that the mechanical properties, such as tensile strength, are reduced because of the previous exposure. It can be observed that both increased temperature and humidity cause considerable damages to the blends. The ageing effects, like decrease of tensile strength, as a result of higher humidity increases with increasing starch content. These observations do not apply to test specimens stored at 70°C and 50 % humidity. The damage decreases with increasing starch content. Investigations with the µCT show the formation of different crack structures as a result of storage. In addition, SEM can be used to show that there is insufficient adhesion between the PLA matrix and starch particles.

Oral (S15-178, Time: Wednesday 15:10, Room: Bodensee)

Activation of bio-based keratinous fibres as prerequisite for subsequent functionalisation: Monitoring the reduction state of wool fibre surface.

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Keratin is highly cross-linked protein with numerous disulphide bonds represented by the amino acid cystine and naturally occurs e.g. in wool fibres. As a biopolymer, keratin from protein fibres turned out to be an interesting source for alternative materials on non-fossil basis. Reductive treatment of keratinous polymers leads to the cleavage of the disulphide bonds and the formation of thiol groups. Due to the nucleophilic character of the latter one, these oxidised cystine moieties can work as anchor groups for incorporating additional functionalities by further modification, e.g. functional polymer grafting and coating. Furthermore, methods for protein fibre dissolution include cystine reduction to extract soluble keratin for further processing as biodegradable material in multiple scientific disciplines like tissue engineering, bio-based coatings or hair and cosmetics chemistry. To evaluate the quality of keratin extraction by protein fibre dissolution and the conditions for further processing, the change in thiol concentration therefore is the criterion of interest to be monitored. Analytical monitoring of broken disulphide bonds and thiols in keratin fibres therefore is of high interest as basis for the development of new materials and applications from bio-based protein polymers. An analytical method for thiol determination is presented here which can be applied for homogenous as well as for heterogeneous systems. This practical and reliable method allows to derive information about oxidation status of activated protein fibres for further processing as bio-based material. The results are valuable for a non-destructive assessment of reduction processes in solid keratin structures and other disulphide containing proteinaceous material.

Oral (S15-211, Time: Wednesday 16:00, Room: Bodensee)

Quantification of process-induced degradation: the effect of processing conditions on the degradation of PLA during single-screw extrusion

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Petroleum-based polymers play an essential role in today's society; as they are low cost, versatile and easy processable materials. The enormous accumulation of non-degradable waste in nature and the depletion of fossil raw materials, boosted the industrial efforts to develop bioplastics like poly(lactic acid) or PLA, which is an aliphatic, biobased and biodegradable polyester. The same ester groups that are responsible for the biodegradation are the vulnerable linkages during polymer processing techniques such as extrusion. PLA's sensitivity to high temperatures, moisture and shear causes a molecular weight reduction during processing that changes rheological, mechanical and thermal properties of the polymer. Therefore, understanding the importance of melt processing conditions is crucial to minimize process-induced degradation. The aim of this research is to understand and quantify the process-induced degradation of PLA during single-screw extrusion. The effect of five processing parameters (namely (1) screw rotation speed, (2) die geometry, (3) compression ratio of the screw, (4) thermal history and (5) moisture content) on the molecular weight of a low D-isomer content PLA is studied. Each parameter is varied at multiple levels to investigate the relation between the processing parameter and the molecular weight of PLA after single-screw extrusion. Additionally, the degradation throughout the extrusion process is also studied by measuring the molecular weight at different locations along the extrusion screw. The measured data is used for regression analysis and used to predict the molecular weight decrease resulting from specific processing conditions. The output of this research will help to understand and minimize degradation, which is essential to limit post-production waste and obtain high-quality and reproducible polymer products.

Oral (S15-229, Time: Wednesday 16:20, Room: Bodensee)

Morphology and mechanical properties of blends of poly(lactic acid) and proteinous bio-based thermoplastics.

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Biopolymers, such as proteins, starch and PLA, have been considered an attractive alternative for petroleum-based polymers. Proteins from decoloured blood meal (DBT) is a newly developed starting material for biopolymer production, produced from pre-treating blood meal with peracetic acid. Blood meal is rich in protein making it an ideal source of bio-based polymers, however, it requires some modification to improve processing and mechanical properties. Blends of poly(lactic) acid (PLA) and decoloured bloodmeal were prepared using itaconic anhydride functionalised PLA (PLA-g-IA) as a compatibiliser. Blends of various ratios of DBT to PLA, with and without compatibilizer were prepared. The compatibility between the material blends was investigated using mechanical testing, scanning electron microscopy (SEM), differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA) and wide-angle X-ray scattering (WAXS). Blending DBT with PLA increased the tensile strength and modulus of DBT, whereas the strain at break decreased. The glass transition temperature increased when compared to neat DBT. SEM revealed enhanced interfacial adhesion between the two phases in the blends with PLA-g-IA evident from the more homogenous microstructure obtained. WAXS revealed an insignificant decrease in the crystallinity of the blends compared to neat DBT, indicating that blending with PLA brought about no structural effects on DBT. The results presented in this study showed the feasibility of improving the properties of DBT with PLA-g-IA for use in agricultural and packaging applications.

Oral (S15-258, Time: Wednesday 16:40, Room: Bodensee)

Improvement of the thermo-oxidative process stability of biopolyesters by biogenic wine by-products as sustainable functional fillers

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Biopolyesters such as PBS and PLA are promising sustainable alternatives to petroleum-based polymers. Their sensitivity to thermo-oxidative degradation is one reason for their limited application. A problem that is usually solved by using conventional unsustainable stabilizers. In this research, biogenic by-products from regional waste streams are investigated as fully biobased stabilizers. Wine grape pomaces (WP) of two different varieties were studied. Consisting of grape seeds, stems and skins, these substantial unused low-value by-products of winemaking represent up to 25 wt.-% of the grape. This biomass provides high amounts of functional substances like polyphenols. WPs were prepared by drying and grinding, to be used as bio-additives or functional fillers at higher filling rates. The by-products were characterized in terms of composition, rel. moisture, and pH value, in addition to particle size distribution analysis, TGA, and assays to determine the total polyphenol content and the antioxidant activity. The biopolyesters were processed with a twin-screw compounder with WP contents up to 20 wt.-%. The thermal and mechanical properties of the compounds were investigated by DSC, TGA, and tensile tests on injection molded specimens. The thermo-oxidative stability was determined by dynamic OIT and oxidative TGA measurements. While the characteristic thermal properties of the materials remain almost unchanged, demonstrating the general applicability of WP as a functional filler, the mechanical properties were altered within expectable ranges. The analysis of the thermo-oxidative stability reveals WP as an efficient stabilizer for biopolyesters. This research shows that WP, as a low-cost and biobased stabilizer, improves the thermo-oxidative stability of biopolyesters while maintaining their key properties for processing and technical applications.

Oral (S15-267, Time: Thursday 10:40, Room: Bodensee)

Tuned processability and mechanical properties of PHBV copolymers by using Boron Nitride (BN) and medium-chain-length-PHA (mcl-PHA) as additives

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Polyhydroxyalkanoates (PHAs) are a class of fully biodegradable polyesters produced by microbial cultures. Because of their similarity to PE and PP and their high barrier properties, short-chain-length PHA (scl-PHA) such as poly-3-hydroxybutyrate homopolymer (P3HB) and poly-3-hydroxybutyrate-co-3-valerate copolymers (P3HBV) are the best candidates to replace fossil-based plastic in packaging systems. Despite these advantages, large-scale diffusion has been limited by their high crystallinity, brittleness, melting point, and sensitivity to thermal degradation that negatively affect processability. A possible solution for scl-PHAs processing issue could be PHBV copolymers production with high 3HV content to decrease melting point, glass transition temperature and crystallinity, leading to an increase of the ductility of materials. However, higher 3HV could induce difficulties in the processability of self-supported films with in particular a slow crystallization rate, not suitable for thermoplastic applications. In order to tackle these problems, the effect of three different factors on processability and final properties were investigated: 3HV units' content (3, 18 and 28 mol% of 3HV), the addition of 0.5, 1 and 2 wt% Boron Nitride (BN) as nucleating agent to increase crystallization rate and the addition of 5 wt% medium-chain-length-PHA (mcl-PHA) to improve mechanical properties and especially the ductility. All PHBV copolymers and their compounds with BN or mcl-PHA were prepared through a twin-screw extrusion and compression moulding processes to give 100 μm thick films. PHBV formulations were characterised on a macro- and microscopic scale, i.e. thermal (TGA, DSC), mechanical and barrier properties. The results from characterization showed an improvement in ductility and thermal stability when mcl-PHA is added, while the addition of BN leads to the obtention of a homogenous structure, a toughened material and, in both cases, no change in permeability.

Oral (S15-279, Time: Thursday 11:00, Room: Bodensee)

Polyesters for Flexible Film Applications based on Pentanediol

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Significant effort has been expended to develop a polyester-based film that can replace LDPE and LLDPE in flexible film applications. Two currently available commercial alternatives are based on butanediol reacted with a mixture of terephthalic (TPA) and adipic acid (AA) as well as butanediol reacted with succinic acid. In this talk, I will review the relevant literature in the field before discussing our recent efforts in this area, which are based on pentanediol and furandicarboxylic acid (FDCA) These two monomers have the significant advantage over the commercial materials because of their relatively inexpensive method of synthesis from biobased monomers. Copolymerization with other diacids can be used to tune the glass transition (Tg) and melting (Tm) temperatures, which is critical for this application. Our first polymer that has the necessary Tg and Tm is a copolymer with pentanediol instead of butanediol that is reacted with TPA and AA; more TPA is required vs. that required for butanediol to offset the increase in flexibility from the pentanediol. Our experiments with FDCA have been less successful; the quantity Tm-Tg is too small to create a polymer with the necessary thermal behavior. The other important processing parameter is the rheological behavior, which will also be discussed.

Oral (S15-281, Time: Wednesday 17:00, Room: Bodensee)

Highly oriented stereocomplex crystals in PLLA/PDLA bicomponent fibers prepared through laser-heated melt electrospinning and subsequent annealing processes

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Highly aligned sheath/core (S/C) and sea/islands (S/I) bicomponent fibers consisting of poly(L-lactide) (PLLA) as sheath or sea component and poly(D-lactide) (PDLA) as core or island component were fabricated by laser-heated melt electrospinning (LES) assisted with a take-up device. Take-up velocity was varied from 0 to 120 m/min, while constant laser power and applied voltage of 14 W and 20 kV, respectively, were adopted. After the LES, as-spun S/C and S/I ultrafine fibers were subsequently annealed at 120 or 190 °C for 1 hour. For both as-spun S/C and S/I fibers, fiber diameter decreased monotonously with an increase in take-up velocity. The S/C and S/I fibers exhibited amorphous structures except for the presence of homo-crystal in S/I fibers prepared at the take-up velocity of 120 m/min. A certain degree of molecular orientation along the fiber axis was confirmed through the birefringence and wide-angle X-ray diffraction measurements. After the annealing of as-spun fibers at 120 °C, the coexistence of highly oriented homo-crystal and a small amount of stereocomplex crystal was detected in the S/C and S/I fibers. In the case of the annealing temperature of 190 °C, the S/C fibers were fused to each other strongly to make a fiber bundle, however, the S/I fibers were separated well from each other. In addition, all homo-crystals were converted into highly oriented stereocomplex crystals. This result indicated the occurrence of sufficient mutual diffusion between PLLA and PDLA molecules to form stereocomplex crystal, in which a pair of PLLA and PDLA exists in the crystalline unit cell, during the annealing process at 190 °C for S/I fibers. For both S/C and S/I fibers obtained at higher take-up speed conditions, significant improvement of mechanical properties after annealing was also confirmed.

Oral (S15-293, Time: Thursday 11:40, Room: Bodensee)

Chitosan-modified dressings with improved antibacterial properties for hemostatic applications

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Hemostasis is a body’s normal reaction to severe wound injuries that causes bleeding. The main purpose of hemostatic products is to compensate for the compromised first step of wound closure. In the past decades, a wide range of hemostatic agents have been developed in order to prevent hemorrhage. Chitosan (CS) is a promising hemostatic agent because it can adhere to red blood cells and encourages platelets to adhere, activate, and aggregate at the site of vascular injury because of the biodegradable and biological properties. In addition, dextran (DEX) is a heterogeneous polysaccharide synthesized by the action of the bacterium Leuconostoc mesenteroides on sucrose with biocompatible and biodegradable properties. Some of the biofunctions of DEX are hemostasis, wound healing, and bacterial growth inhibition. In the present work, CS was modified with oxidized DEX (ODEX) to produce adhesive dressings with improved hemostatic properties. To enhance the antimicrobial ability of the produced hydrogel, inorganic additives (CaO-2CeO2) were integrated with the natural polymers.The modification process of CS developed though a Schiff base reaction and then freeze dried, fabricating a porous patch which confirmed by various characterizations. For example, the successful synthesis of material confirmed by FT-IR, its crystallinity was researched by XRD, the morphology of patch was analyzed with SEM images and water swelling was also investigated. Acknowledgement This research has been co-funded by the European Regional Development Fund (ERDF) and from national resources through the Operational Program "Competitiveness, Entrepreneurship and Innovation", NSFR 2014- 2020, under the call "Aquaculture"-"Industrial materials"-"Open innovation in culture" (project code: T2EΔK-00275)

Oral (S15-355, Time: Thursday 12:00, Room: Bodensee)

Multifunctional superhydrophobic and underwater oleophobic cellulose paper for separation of hazardous organic pollutants from water

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Due to the growing industrial oily wastes and frequent oil spill incidents, it is increasingly necessary to separate water/oil mixture. The present solution system is ineffective and generating secondary waste, which is inspiring the development of simple effective oil/water solution. In this study, we reported Janus multifunctional superhydrophobic and underwater oleophobic paper/fabric derived from biopolymer for separating oil/water mixtures and dye removal. Simple dip coating approach was used to create this multipurpose paper. First the paper was dipped in polyphenol extract, followed by FeCl3 coating to generate underwater oleophobic property. Subsequently this paper oxidized and then modified by myristic acid (MA), to create a superhydrophobicity. The results shows that the multifunctional paper has excellent superhydrophobicity having water contact angle 165° and self-cleaning property. The developed paper has been used to clean series of oil water mixture in acid and alkali environment with high separation efficiency >95% and flux > 150 L/m2/h without external pressure. The developed paper has reused for at least 10 cycles with high efficiency. In both mode water blocking and under water oil blocking mode the developed paper shows the efficiency >95%, with gravity driving force only. The multipurpose paper also used to treat wastewater for the elimination of methylene blue dye. All of these features combine to make the paper an outstanding choice for on-demand separation of various oil/water mixtures. Keywords: Biopolymer, cellulose, superhydrophobic, underwater oleophobic, self-cleaning and oil/water separation

Oral (S15-374, Time: Thursday 14:10, Room: Bodensee)

Processability study of modified thermoplastic starch/poly(butylene succinate) adipate blends with couple agents

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Increasing concerns about the global environmental issues caused by fossil-based materials generated a great need for the fabrication of biopolymer-based materials in the packaging industry. Thermoplastic starch (TPS) is a sustainable and inexpensive biopolymer. This could be a great approach to produce new biomaterials. However, TPS has low mechanical properties and high hydrophilicity, which makes it unstable in different environments. The strategy to overcome these limitations has focused on the modification of starches, the use of coupling agents and blending it with other biodegradable polymers. This article evaluates different formulations of blends of modified corn starch and poly (butylene succinate) adipate (PBSA), using glycerol as plasticizer and esterification catalysts as coupling agent. The blends processability was evaluated by films obtained in an internal mixer and compression molding. The results demonstrated that the addition of esterification catalysts improves its processability, evaluated by the torque measured during the blending process. Also, the mechanical properties, morphology of the film and hygroscopic properties were study and related with the processability capacity of the blends.

Oral (S15-389, Time: Thursday 14:30, Room: Bodensee)

Controlling the surface functionality of graphene-based quantum dots for multifunctional poly(L-lactide) nanocomposites

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Poly(L-lactide) (PLLA) is a sustainably sourced and compostable polymer offering a promising replacement for petroleum-derived polymers. However, slow crystallization kinetics, low melt elasticity, as well as insufficient stiffness/strength limits both PLLA's processability and use to consumers. Although these shortcomings can be overcome with the incorporation of stiff/strong nanoparticle fillers like graphene nanosheets or nanoplatelets, these typically render the composite black, with a loss of transparency. Despite this, these nanoparticles possess a high degree of thermal and electrical conductivity, which can also be imparted to their nanocomposites. Recently, graphene-based quantum dots (GQDs) possessing diameters less than 15 nm have arisen as a promising material imparting the benefits of larger graphene particles, but without significant loss of transparency. However, the increased surface-to-volume ratio of these particles creates significant edge effects, with surface functionality and defects on the basal plane dramatically altering particle and composite properties. Herein, this work aims to fabricate GQDs from citric acid, and identify how surface functionality impacts the molecular interactions with the matrix. By controlling surface functionality through reduction and/or nitrogen-doping, the intrinsic properties of the GQDs, including colour, electrical/thermal conductivity, intrinsic stiffness/strength, as well as matrix-filler interactions can be controlled, translating to changes in composite bulk properties.

Oral (S15-412, Time: Thursday 14:50, Room: Bodensee)

Increasing the Bio-content of Superabsorbent Polymers: Assessment of Long-Term Water Absorption Capability and Biodegradability

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Superabsorbent polymers (SAPs) are already an essential part of our lives and they are on their way to becoming an essential tool for farmers to cope with climate change. Our food production is becoming increasingly vulnerable to water scarcity, as rainfall regimes become less predictable and more sporadic. Globally, 80% of farmland relies solely on rainwater, so irrigation alone is not an economically viable option to combat water scarcity. Farmers need technologies that would deliver the available water to the plants as efficiently as possible. SAPs are hydrophilic, cross-linked polymers that absorb water several hundred times their own weight. When placed in soil, SAPs absorb the available moisture from the soil and retain their surroundings wet for extended durations compared to untreated soil. Hence, plants grown in SAP-treated soils can continue to grow even under abnormally long dry periods. This practice prevents potential yield losses that would normally occur due to insufficient rainfall. Currently, most commercial SAPs are fully synthetic, which are generally acrylic acid and/or acrylamide based and have very limited biodegradability. In literature, there are studies on SAPs containing biomaterials, such as carboxymethyl cellulose (CMC), albeit at very low weight fractions (e.g., 5 wt.%). Most such studies report only one SAP formulation. In this study, our aim was to understand how further increasing the CMC content affects the material properties, performance, and biodegradability of SAPs. Therefore, we successfully synthesized and tested 8 CMC-SAP formulations, incorporating 1,4 to 51,5 wt.% CMC.

Oral (S15-419, Time: Thursday 15:10, Room: Bodensee)

Influence of injection moulding process parameters on industrial Polyhydroxyalkanoates properties for medical device applications

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This work was initiated with the motivation of using bio-based polymers in high-end medical applications to achieve circularity and to reduce environmental impact. In this work, injection and micro injection moulding of bio-based polymers are studied with industrial grade of Polyhydroxyalkanoates (PHAs). PHAs can be processed with injection moulding but some material properties and processing conditions provide bottlenecks for high-end applications such as in medical devices. The aim of this research is to systematically reveal the bottlenecks for the implementation of such materials in high volume and highly demanding industrial applications. Initial thermal characterization of the materials was done using Differential Scanning Calorimetry and Dynamic Mechanical and Thermal Analysis to understand the behaviour of PHAs in a molten condition. Five moulding process parameters – Injection speed, Injection temperature, Injection pressure, Packing pressure, and Mould temperature - were identified as critical parameters to produce test parts. The moulded samples were subjected to physical and mechanical testing to reveal the the effect of the material degradation on their properties. Attention was paid to the evolution of the properties with the post-moulding crystallization of the bio-polyesters. The results of these analyses are presented along with the reflection on the identified limitations for industrial applications of PHAs in medical devices.

Oral (S15-474, Time: Thursday 16:00, Room: Bodensee)

Fogging behavior of selected biopolymers

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The use of plastics in automobiles has increased sharply in recent years; the interior in particular is made almost entirely of them. To improve the sustainability of plastics, increasing research is into alternatives. To date, however, the application focus of bio-based plastics has often been on short-lived products such as packaging. Bioplastics have not yet been used in the interior sector itself. In order to prove the suitability of bioplastics to vehicle manufacturers, it is necessary to be able to predict the material behavior over the application period and to meet industry-typical standards. This includes the emission behavior, which is of particular importance in the interior. Some of the emissions are low-volatile organic compounds (SVOCs). These can be deposited on the windows in the vehicle due to condensation and impair visibility (fogging).

DIN 75201 is a test specification for investigating this material behavior, which is often used by automobile manufacturers. This method was used to investigate the fogging behavior of almost 30 different types of plastics. The materials investigated correspond to typical automotive plastics and a selection of relevant bioplastics (like Bio-PA, PLA, PBS, Bio-PE). Prior to this, investigations were carried out to determine an optimum sample quantity for the measurement of granules. To determine the influence of moisture in the material on the emission behavior, moisture measurements were performed concomitantly. Subsequently, some generated condensates were analyzed for their constituents by Fourier transformation infrared spectrometry (FTIR).

The results of the fogging characterization show that bioplastics in particular are certainly suitable for use in automotive interiors. The influence of moisture in the investigated material on the fogging characteristic value can also be demonstrated, so for some of the materials an increased fogging behavior is shown as a result of an increased moisture content.

Oral (S15-542, Time: Thursday 16:20, Room: Bodensee)

Scale-up studies of bio-based monomers and polymers production from biomass derived xylose

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A key solution to fossil-based plastics replacement is the development of bio-based polymers from renewable and sustainable feedstocks. However, many challenges in achieving comparable material properties are slowing down the transition towards more sustainable polymers. Biomass is one alternative source of monomers that will enable the replacement of these fossil-based materials. Recently, an underutilised sugar molecule abundant in nature, xylose, was shown to be a valuable resource for monomers and polymers. The monomer derived from this non-edible sugar is dimethylglyoxylate xylose (DMGX), a heterocyclic difunctional diester that can be converted into poly(alkylene xylosediglyoxylates) (PAX) through classical polycondensation with diols (Manker et al. Nature Chemistry, 2022, 14, 976–984). This family of new polyesters are bio-based, degradable and chemically recyclable, with a high glass transition, tough mechanical properties and strong gas barrier properties compared to commercial biopolymers. The challenge we faced during this project was the up-scaling of the monomer and polymer synthesis from a lab protocol to a more industrial suitable process. We optimised a solvent free synthesis of DMGX and its purification allowing to develop a process which is feasible on the 100 kg scale. Batch and continuous reactors were investigated in order to characterise advantages and disadvantages in terms of viscosity, outputs, yields, and reaction times. The monomer was also successfully polymerised in batches of more than 1 kg. The development of this industrial process of both the monomer and the polymer allowed to produce quantities for market testing and prototyping, bringing the bioplastic closer to industrial applications.

Oral (S15-545, Time: Thursday 16:40, Room: Bodensee)

Degradation study of PHB/PLA/PCL blends under a seawater environment

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Single-use plastics are responsible for several tons of waste annually, accounting for nearly 50% of the total waste produced. To improve packaging waste management, producers expect to increase their recovery responsibility from 25% to 55% of all plastics produced by 2030. To approach this target and improve the circular economy of these materials in the environment, improvements in recycling strategies and the introduction of polymers from natural resources, known as biopolymers, have been attractive alternatives. A vast range of biopolymers has been investigated for use in single-use packaging and medical applications. Some biopolymers are biodegradable, making them interesting alternatives in terms of biological circularity. Despite this, many biopolymers lack mechanical properties and perform poorly compared to the high-performance fossil-based polymers available in the market. To overcome this problem and improve the mechanical properties of biopolymers, the blending process through extrusion combines the performance of different biopolymers, tailoring their flexibility for applications such as packaging, single-use utensils, and medical devices. This project aimed to study different formulations of PHB, PLA, and PCL, to evaluate their main mechanical and chemical properties, as well as moulding tensile specimens, to assess their biodegradability in a simulated seawater environment for up to 8 weeks. The blending process initially promoted the flexibility of the material, and after 8 weeks of seawater degradation, it exhibited a significant increase in the brittleness of all samples. Infrared analysis showed the expected chain scissions due to polymeric degradation, which was corroborated by the sample weight variance after 8 weeks. All samples presented a degraded appearance, as confirmed by SEM analysis. Promising blends with increased flexibility are going to be used in 3D printing of biodegradable ureteral stents (BUS) with Arburg plastic freeforming.

Oral (S15-546, Time: Friday 10:40, Room: Bodensee)

A Novel Sustainable Biobased Polyester: Processing, Characterization and Applications

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Nowadays, bio-based polymers have and are still studied to address environmental issues of the current, mainly fossil fuel-based polymers. One of the challenges in their development is to use feedstock that is not in competition with the food chain. This work focuses on a new family of biobased polyesters: poly(alkylenexyloseglyoxylates) (PAX). PAX is based on xylose, a non-nutritional pentose derived from hemicellulose, which is the second most abundant polysaccharide in plants. These novels polyesters have little been studied as to their processing and application performance. This study investigates these processing aspects in relation to other bio-based polymers. Specifically, hydrolysis of polyesters during processing from shear forces and heat is a big bottleneck. The PAX behaviour with respect to degradation versus water content was closely investigated. In addition, a large plasticizing effect of water was noted, shifting the glass transition temperature by up to 20 °C, which may have a major influence on the polyester's final application. Therefore, finding an equilibrium between the plasticizing effect and the hydrolysis is needed to ensure a molecular PAX stability while maintaining processability and performance. Rheological measurements have been used for quantifying these aspects. Accordingly, PAX has the potential to be processed for the production of products that address the needs of various markets segments.

Oral (S15-611, Time: Friday 11:00, Room: Bodensee)

Properties of cowpea lignocellulosic sidestream reinforced Poly (butylene succinate-co-adipate)/ Poly (hydroxy butyrate-co-valerate) bio-composites for packaging materials.

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Packaging accounts for about 42 % of plastic consumption, most of which are made from petroleum-based non-biodegradable plastics. The lack of plastic waste management systems, especially in sub-Saharan Africa, has led to their improper disposal leading to their accumulation in natural environments. Biocomposite packaging materials are attractive as they can be made up of biodegradable polymers and lignocellulosic fibers (natural fibers, residues from agriculture, forestry, and food industries). Agricultural sidestreams, including climate-smart African crops, such as cowpea (Vigna unguiculata (L.) Walp) are generally considered as waste and as such, have no economic value. In this study, biocomposites were prepared using Poly (butylene succinate-co-adipate)/ Poly (hydroxy butyrate-co-valerate) as a matrix reinforced with up to 30 % cowpea lignocellulosic sidestreams. The biocomposites were further processed into films by film extrusion for flexible packaging and injection molded specimens for rigid packaging. The addition of cowpea sidestream led to an increase in tensile stress at maximum force and an increase in Young's modulus while decreasing the strain. The stereo-microscope images showed that the fibers were well dispersed within the matrix and aligned in the processing direction in the films. The rheology results revealed that the PBSA/PHBV blend, and the resulting bio-composites had a shear thinning behavior with complex viscosity decreasing with an increase in angular frequency. The complex viscosity increased with an increase in fiber loading. The DMA results revealed that the storage modulus increased with fiber loading in the temperature range of -80 to 70 °C. In this study, we have demonstrated that agricultural sidestream of locally available climate-smart African crops can be used to produce bio-composite films and injection molded specimens with balanced material properties for packaging.

Oral (S15-612, Time: Friday 11:20, Room: Bodensee)

Effect of lignin modification on the interaction at the interface with biodegradable polyesters

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Plastics are the key materials that have allowed the new technologies development enabling nowadays lifestyle. However, the mismanagement of plastics at the end of their use has led to current environmental impact of plastic wastes, particularly the one coming from single use plastic. Despite the growing awareness on the risks of plastic pollution, more than 95 % of global plastics still relies on the extraction of fossil fuels to produce non-biodegradable products. Around 60 % of all plastic ever produced has been dispersed in the nature or accumulated in landfill, harming lives and ecosystems. One of the approaches to overcome this issue is the development of bio-based and biodegradable bioplastics. In this regard, the development of new and high value materials from biomass as well as from industrial side streams is of interest to decrease the use of fossil raw materials. Biomass has already shown to be an excellent source for materials providing large scale applications. Lignin is the second largest biopolymer on the earth, and it is also the largest non-commercial waste product, with an annual production around 50 million tonnes. Recently, development of lignin to other than fuel purposes (low-value products), has received much attention, one example being carbon fibers developed from lignin. However, how to exploit lignin as biopolymer in thermoplastics is still challenging. In this communication, the use of modified lignin by reactive extrusion in thermoplastic blends has been explored focusing the attention on its interactions with aliphatic and aliphatic/aromatic polyesters such as polylactic acid and poly-butyl adipate terephthalate. The influence of these different interactions on the mechanical, thermal, and morphological properties as well as on the degradation behavior in composting conditions of the melt-processed blends has been studied.

Oral (S15-615, Time: Friday 11:40, Room: Bodensee)

Research 4.0: A Modular Approach to Boost the Development of Innovative Biobased Fibres

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An accelerated growing demand for textile fibres demands a more proactive approach which includes implementing frameworks and technologies for recycling and a broad use of biobased materials as an alternative to polymers like polyester and nylon. Biobased fibre products pose a big technological challenge as growing demands on the varying quality of recycled feedstock and the achievement of a consistent fibre performance require a continuous development and optimisation of both technology and production parameters. Research work in the field of biobased fibres is characterised by a high degree of adaptation needs. Thus, facilities must be able to be adapted after initial experiments and the knowledge gained thereby. Research 4.0 is an approach enabling researchers to work optimally supported by the system. Necessary adjustments are made under the conditions of a highly complex manufacturing process, which is determined by many parameters. This results in the following requirements for the flexibility of modular research facilities:

INTEGRATION: Easy integration of new production modules
SCALABILITY: Easy replacement of production modules with modified specifications
FLEXIBILITY: Easy modification of production steps' positioning within the production line
HIGH PERFORMACE: Intelligent production modules synchronized by a master process control level
ANALYTICS: Continuous monitoring and evaluation of process parameters

A reliable development of new innovative biobased fibres demands an efficient, systematic and, in part, self-optimising experimental working system, which must be intelligent in gathering data from the process and flexible in enabling the rearrangement of the process. Applying the principles of Research 4.0, each module of a plant represents a production step and is equipped with a PLC to control itself and to organise in association with other modules within the line. The control hierarchy has an intelligent modular structure that configures itself according to the arrangement of each single module given by the hardware and the interfaces within the system. Moreover, all production parameters can be permanently visualised and recorded, enabling a complete traceability of the process.

This paper will present the implementation of Research 4.0 as a tool for the development of a product from the idea to its practical implementation based on a modular conceptual approach offering the required flexibility for the complete validation process: principle > process > product.

Oral (S15-619, Time: Friday 12:00, Room: Bodensee)

3D Printed Composite Scaffolds of Nanoparticle-Doped GelMA for Bone Tissue Engineering

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While bone disorders and defects are continuously increasing, the advancement of bone regeneration methods is perpetually evolving. The key objective of this study was to acquire and understand the biomaterials that can be used in bone tissue engineering purposes. Gelatin, fibrinogen, hyaluronic acid, chitosan, alginate, and cellulose are non-cytotoxic and biocompatible natural polymers and universally used biomaterials for tissue engineering. Nevertheless, these natural biomaterials were found to have low bioactivity and inadequate mechanical strength that restrict their applications. To address these disadvantages, these biomaterials can be paired with nanoparticles and construct composites. The integration of nanoparticles enhances the mechanical properties of the final structure along with its bioactivity and regenerative capacity. This article presents the development of a nanocomposite hydrogel from gelatin methacryloyl (GelMA) incorporated with magnesium oxide nanoparticles and chitosan for tissue engineering applications by investigating its mechanical, morphological, and bio-compatibility properties. In this research, fourteen different combinations were studied on GelMA to manipulate its density and shape fidelity as an aim to enhance the mechanical properties and cell regenerative capacity of GelMA. Rheological tests were used to compare the mechanical properties of composite scaffolds. It can be seen that, MgO and chitosan combination enhances the mechanical properties of pure GelMA, while improving the cell viability immensely. The subject reported in this study will be an introduction for GelMA-MgO-Chitosan composite hydrogels for further research studies in tissue engineering.

Oral (S15-652, Time: Friday 14:10, Room: Bodensee)

Investigation of Barrier and Mechanical Properties of Biopolymer Based Flexible Laminated Packaging Films

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In packaging industry, the most promising biopolymers such as thermoplastic starch (TPS), polybutylene succinate (PBS), polylactic acid (PLA), and polybutylene adipate-co-terephthalate (PBAT), polycaprolactone (PCL), and polyhydroxyalkanoate (PHA), are used due to their potential biodegradable, sustainability and zero carbon footprint emissions instead of nonbiodegradable/fossil-derived polymers. For example, thermoplastic starch has oxygen and carbon dioxide permeability are better than moisture permeability, but in terms of mechanical and barrier properties, it is below the limit values compare to traditional industrial packaging applications. That's why TPS is tried to usable in the packaging industry by compounded with other biopolymers, and additives, coating or laminated to improve its weak properties. Recently, the coating technology of new generation packaging films with bio-based materials has attracted a lot of attention. Cellulose nanocrystals (CNCs), which is one of these providers, has an important place in packaging preserving thanks to its oxygen and moisture transmission performance, durability features and ecologically friendly features.

In this study, the biopolymer-based flexible laminated packaging films were produced by using different commercial biopolymers such as TPS, PBS, PLA PBAT, PCL, and PHA. Firstly, biopolymers were compounded within the maximum rate 1,25 phr of adipic acid and a polymeric epoxy-based chain extender in the twin screw extruder and then turned into a film helping by the blown film extruder. On the other hand, the paper was coated by 3-5 gsm of two different CNCs-based solution which has barrier properties against oxygen and moisture. According to optimum CNCs solution coating gsm range on paper were defined. Our biopolymer films were laminated with CNCs solution coated paper using a water-based compostable adhesive. The effect of CNCs solution coating on barrier properties of packaging films and mechanical & barrier comparison of different laminated/non-laminated film structures were investigated.

Oral (S15-665, Time: Friday 14:30, Room: Bodensee)

Biopolymer-based systems with bioactive agents as a future solution for food packaging of perishable products

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Food industry is one of the major importers of synthetic polymers, however, due to ecological and social concerns, there is a huge interest in packaging that performs better and pollute less. The food packaging films available in the market are difficult to recycle, usually, due to their multi-layer structure made by different polymers. Owing to this, the development of packaging films using a single polymer with similar or improved properties became essential; and biopolymers have been vital in the development of novel and sustainable systems. In fact, an active food packaging that preserves food quality, safety and shelf-life of fresh products have been requested from consumers. Despite biodegradable benefits, some limitations on mechanical and barrier properties have been notice. Yet, several approaches can be made to improve its performance. One of the most attractive residues used in these approaches are proteins, either vegetable or animal-sourced, due to their propensity to change the molecular structure when exposed to specific values of pH and temperature. As a result of this variation, it is possible the influence the film properties. Therefore, the main objective of this work was to develop polylactic acid (PLA) and fish gelatine (FG) films with good mechanical, barrier, and antioxidant properties, as a green alternative to conventional food packaging, evolving a single layer packaging, fully biodegradable. The films were produced in the melt with fish gelatine (pure and chemically modified) and green tea extract (GTE) addition, in different amounts. The results revealed an improvement of the oxygen barrier properties of PLA with the addition of FG. Additionally, the homogeneity of the FG dispersion is enhanced when combined with GTE. The addition of GTE also increases antioxidant capacities due to its phenolic and flavonoid content.

Oral (S15-693, Time: Thursday 11:20, Room: Bodensee)

Influence of polyethylene glycol on the morphological and mechanical properties of polylactic acid/melt flowable lignin biocomposites

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Polylactic acid (PLA)–lignin biocomposites are considered promising renewable green materials towards a sustainable future. However, the complex and heterogeneous structure of lignin, together with its poor flowability and rheological characteristics limit lignin incorporation at high contents (e.g., >20wt%). At such loadings, the melt processing is usually challenging, or the resultant composite exhibits high brittleness and low strength. Recently, a new melt-flowable biopolymeric lignin with improved structural homogeneity, named Bioleum (BL), has been developed. Due to its appealing thermo-mechanical characteristics, Bioleum may offer great promise toward manufacture of thermoplastic biocomposites. In this work, PLA was blended with BL using a melt extrusion method to test its suitability for the preparation of PLA based composites. polyethylene glycol plasticizer (PEG2000) was introduced to PLA-BL composites in varying amounts (up to 5wt%) in order to increase the biocompatibility of PLA and enhance the dispersibility of BL. Scanning electron microscopy (SEM) and tensile testing were performed. Biocomposites with lignin content up to 40wt% were successfully extruded, and compared to the literature, improved mechanical properties were obtained. Moreover, the incorporation of PEG greatly affected the morphology and mechanical properties of the composites.

Poster (S15-042, Time: Thursday 17:00, Room: Foyer)

Poly(ethylene furanoate) (PEF) – From Crop Waste to Product

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2,5-Furandicarboxylic acid (FDCA) has the potential to become one of the most important platform chemicals of a bio-based chemical industry due to its similarity to the widely used and well established, but petrochemically-derived terephthalic acid (TPA). Our target is to combine methods of synthetic biology and chemo-enzymatic synthesis to develop new and sustainable production chains for FDCA and its precursor 5-hydroxymethylfurfural (5-HMF) from waste biomass and to use this molecule as basis component for a wide range of tailor-made fiber polymers. The bio-based synthesis route will enable drop-in solutions for PEF. Synthesis of PEF via different polycondensation routes: Polycondensation of FDCA and furan-2,5-dicarboxylic methyl ester (FDME) with ethylene glycol are the best routes for the synthesis of PEF. The monomer quality as well as the chosen catalyst of both FDCA and FDME has a great influence of the polycondensation. Optimal polycondensation parameters have been defined for the further production of tailer-made fibers. The final goal of the presented project is the understanding of the physical properties of PEF in terms of a fiber polymer for the different applications in reinforcement, technical textiles and cushion materials. The polymer properties were related to the processing properties for high strength, filament and staple fibers respectively. Spinning of PEF fibers succeeded for intrinsic viscosity range (0.50 – 0.65 dL/g) for textile fibers in technical scale. Filaments were drawn exceeding the targeted strength > 40 cN/tex at 25 % elongation. Textile fibers were drawn already to strength for technical fibers > 50 cN/tex as well as draw-texturized. Fibers were processed to textile materials. Higher intrinsic viscosity range polymer (0.65 – 0.80 dl/g) was spun and drawn to high tenacity fibers for tire cord.

Poster (S15-175, Time: Thursday 17:00, Room: Foyer)

The Processing Characteristics of Poly(3-hydroxybutyrate-co-4-hydroxybutyrate)

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Poly(3-hydroxybutyrate-co-4-hydroxybutyrate) [P(3HB-co-4HB)] is a biodegradable aliphatic polyester developed to improve the low crystallization rate, high crystallinity, and low thermal stability of poly(3-hydroxybutyrate) [P(3HB)]. Due to the low crystallization rate of P(3HB), big spherulites are formed which impinge to form an interface and makes P(3HB) brittle. Thermal degradation of P(3HB) occurs near the melting point to make products by melt processing very difficult. To overcome these issues, 4-hydroxybutyrate (4HB) is biosynthetically copolymerized with 3-hydroxybutyrate (3HB) to prepare P(3HB-co-4HB). In this study, the effect of the melt processing temperature of P(3HB-co-4HB) on rheological and crystallization behavior is investigated. Specifically, this research is focused on the effect of thermal degradation of P(3HB-co-4HB) of different 4HB content exhibiting different thermal stability on the rheological and crystallization behavior, It was found that thermal degradation occurred at 180oC or higher and increased with increasing 4HB content. A small amount of thermal degradation reduces melt viscosity and increases the tanδ. As a result, it may cause a problem with melt processing ability. P(3HB-co-4HB) copolymers undergoes thermal degradation near melt processing temperatures and the degree of degradation increases with increase in the 4HB content. The formation of low molecular weight P(3HB-co-4HB) from thermal degradation reduces the crystallization temperature. Thus, the isothermal crystallization rate and crystallinity decrease when processed at 190℃ compared with 180℃ where less thermal degradation occurs. The effect of thermal degradation of P(3HB-co-4HB) on the isothermal crystallization behavior is more evident in the case of P(3HB-co-4HB) of high 4HB content.

Poster (S15-294, Time: Thursday 17:00, Room: Foyer)

Synthesis and characterization of modified chitosan dressing with enhanced antimicrobial properties.

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The treatment of skin lesions is a big issue in healthcare that costs billions of dollars worldwide. Such treatment is often performed with wound dressings. Wound healing dressings should have some essential characteristics such as biodegradability, biocompatibility, and non-toxicity with antibacterial properties. Adhesion of bacteria on a surface, subsequent colonization (biofilm formation) and contamination/infection is mediated by different types of specific and non-specific interactions. In this context, zwitterions, i.e., overall neutral molecules bearing positive and negative charges, and zwitterionic polymers have been successfully investigated for antibacterial applications. Additionally, chitosan (CS) is a natural biopolymer widely used in wound healing applications due to its low toxicity, high biocompatibility and good biodegradability. In addition, CS provides bacteriostatic and fungistatic activities. In the present work, CS was modified with 2-(methacryloxy)ethyl]dimethyl-(3 sulfopropyl)ammoniumhydroxide (MEDSAH)via free radical polymerization to enhance the antibacterial properties of CS. MEDSAH was selected as zwitterion molecule showing promising antibacterial properties. The obtained MEDSAH-grafted chitosan derivatives (CS-MEDSAH) were fully characterized for their successful synthesis by NMR and FT-IR, for their crystallinity by XRD showing reduced crystallinity compared to CS alone. Furthermore, swelling studies were conducted with the grafted derivatives showing extensive swelling capacity (maximum degree of swelling up to 1800%).
Acknowledgement This research has been co-funded by the European Regional Development Fund (ERDF) and from national resources through the Operational Program "Competitiveness, Entrepreneurship and Innovation", NSFR 2014- 2020, under the call "Aquaculture"-"Industrial materials"-"Open innovation in culture" (project code: Τ6ΥΒΠ-00288)

Poster (S15-422, Time: Thursday 17:00, Room: Foyer)

High Performance Polyimides Derived from Biomass: Design, Synthesis and Properties

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The development of sustainable polyimides (PIs) from abundant renewable biogenic feedstocks has been limited by the bio-based content and the lack of mechanical or thermal properties. This study synthesized a series of daidzein-based PIs with high bio-based content up to 53%, which was significantly higher than the reported bio-based content (approximately 40%). The mechanical and thermal stability of the resulting polymer is comparable with commercial PIs, with the tensile strength of 148.6 MPa and glass transition temperature (Tg) of 318.2 °C. Additionally, the daidzein-based PIs showed excellent fluorescent characteristics with maximum fluorescence efficiency of 45.18 ns and absolute fluorescence quantum yield of 14.1%. Such a performance combination has not been reported in bio-based PIs even petrochemical PIs. Interestingly, the integration of experiments and density functional theoretical (DFT) calculations revealed an effective intermolecular/intramolecular conjugation and the inhibition effect of intramolecular charge transfer by "enone and ether bonds" in daidzein-derived PIs. This work provides a possible route for the design and synthesis of multifunctional polyimides with high bio-based content.

Poster (S15-424, Time: Thursday 17:00, Room: Foyer)

Bio-based formulations for technical textile to prevent plastic contamination

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Every year, millions and millions of tonnes of litter end up in the ocean worldwide, posing environmental, economic, health and aesthetic problems. Poor practices of solid waste management, waste water (including storm water) collection and treatment, lack of infrastructure and awareness of the public at large about the consequences of their actions aggravate substantially the situation. This can have devastating effects on marine ecosystems. Fishing nets have been identified as an item of particular concern as part of the European Directive on Single-use Plastics. However, the collection of fishing nets that are lost accidentally at sea remains a challenge. SEALIVE is working to address these issues by developing bio-based fishing nets made from green alternative materials such as vegetal-base polymers. Nowadays, there is vast knowledge on additive-based chemistry developed to improve the performance and processing of fossil fuel-based polymers, and this knowledge can be used to develop new additive chemistry to improve the performance and properties of bio-based polymers. Production of the nets using bio-based formulation is more sustainable compared to those based on traditional fossil fuel plastics. SEALIVE's goal is to enable a transition to biodegradable and compostable nets to boost the usage of bio-materials in the framework of the circular economy.

Poster (S15-482, Time: Thursday 17:00, Room: Foyer)

Microcontact Printing of BSA Hydrogels for Biosensing Applications

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Bovine serum albumin (BSA) hydrogels are 3D microstructures made from a biopolymer composed of cross-linked BSA subunits. Microcontact printing (μCP) is a cost effective, non-invasive, and customizable printing technique used in the creation of biosensor devices for detecting cytokines and proteins of diseases, like cancer and inflammatory-mediated diseases. However, a longstanding limitation of μCP is the ability to achieve high signal-to-noise levels that are reproducible and repeatable in immunofluorescent assays for diagnostic applications. As such, the objective of this project is to introduce the application of BSA biopolymer hydrogels in the μCP protocol to enhance signal intensity and reduce limit of detection (LOD) of diagnostic biosensor devices. The formation of BSA hydrogels requires highly concentrated BSA printed on a substate and functionalized with 1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide (EDC) to form a BSA biopolymer. Capture antibodies (CAb) are embedded in the BSA hydrogel biopolymer and become the basis for enzyme-linked immunosorbent assays (ELISAs) to detect target biomolecules of disease. The μCP protocol uses a predesigned polydimethylsiloxane (PDMS) stamp to transfer and immobilize BSA hydrogels onto a substrate in a specialized pattern. Recently, we have established an optimized protocol for μCP that has achieved industry-comparable levels of repeatability and reproducibility. As well, optimized features of the BSA hydrogels have been determined, such as concentration of BSA and EDC, using industry standard non-contact dispensing techniques. As such there is potential to combine both approaches in a printed biosensing device platform using biopolymer hydrogels that are fabricated through an optimized μCP technique. This approach shows promise for the multiplex detection of biomolecules of disease with one device, making it significant for diagnostic capabilities.

Poster (S15-656, Time: Thursday 17:00, Room: Foyer)

Effects of Different Biopolymers and Additives on Mechanical and Barrier Properties of Poly(3-Hydroxybutyrate-Co-3-Hydroxyvalerate) Blend Films

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Biopolymers are very popular today, as they contribute to the natural cycle and have a positive effect on reducing environmental pollution. The laws on sustainability in flexible packaging are structured to require the use of biological packaging structures that can replace fossil-based packaging.

In this study, poly(3-hydroxybutyrate-co-3-hydroxyvalerate), commonly known as PHBV, was blended for four ratios with both polyhydroxyalkanoate (PHA) and poly(butylene adipate-co-terephthalate) (PBAT) by using solvent casting method. Chloroform was chosen as the suitable solvent. Four bio-blends were aimed the maximum ratio of 20 % of PHBV was incorporated into PHA and PBAT which are amorphous structures because PHBV is brittle. The neat bio-blends were prepared such as PHA/PHBV and PBAT/PHBV without any additives to determine PHBV behavior into PHA and PBAT. The ratio 1 phr of PEG 400 were used as plasticizers to make good compatibilization between PHA and PHBV, PBAT and PHBV. After the optimum main biopolymer blend ratios have been determined according to mechanical, oxygen permeation, and optical test results, hydroxypropyl methyl cellulose (HMPC) was added at 3 phr as reinforcing agent into PHA/PHBV and PBAT/PHBV main biopolymer blends. Two different biopolymers structure within maximum 3 phr of HMPC were investigated for flexible packaging.

The reason for using HPMC in bioblends is the expectation that it could increase the oxygen and moisture barrier, improve the mechanical and the optical properties in the direction of transparency by giving elasticity to the structure. The antimicrobial effect on the films were be examined by adding 1 phr titanium oxide (TiO2) to the option, which provides the most flexible packaging quality for both PHA/PHBV and PBAT/PHBV blends. In the light of this study, optimum biopolymer ratios and additive amounts for semi-industrial production have been determined.

Poster (S15-662, Time: Thursday 17:00, Room: Foyer)

Mechanical and Barrier Properties of Thermoplastic Starch Based Antimicrobial Packaging Films Prepared with Solvent Casting Method

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The legal directives of the European Union have given importance to the concept of sustainability in every aspect. Especially in packaging industry, plastic waste has become one of the most important problems in the world. Therefore, biodegradable polymers prefer instead of non-biodegradable synthetic polymers made from fossil fuels cause environmental pollution. Thermoplastic starch is one of the most remarkable biopolymers due to its potential low-cost, biodegradable/compostable, and abundant availability in nature. Biodegradable/compostable polymers because of its nature have weak properties compare with synthetic polymers.

The aim of this study, cassava/tapioca and rice starch based antimicrobial packaging films are produced in presence of sorbitol as a plasticizer, milk protein and carrageenan as co-biopolymers by using solvent casting method in according to experimental parameters which are consisting of film forming temperature, time, plasticizer ratio by using central composite design program. The native cassava/tapioca and rice starch are plasticized in presence of the ratio 25-40 phr of sorbitol to determine best experimental condition for packaging film. The maximum 5 phr ratios of milk protein and carrageenan are incorporated into thermoplastic cassava/tapioca and rice starch-based film to improve barrier and mechanical properties of the films, respectively. After obtaining best option for films, the ratio 1-3 phr of lignin grades are incorporated into film to achieve improvement of strength. At final stage, e-polylysine and nisin are added into cassava/tapioca and rice starch-based films as antimicrobial agents. In this study, comparison of different starch source, the performance of co-biopolymers, and different ratio of lignin and it's the effect on formation of antimicrobial packaging films will be investigated.

Poster (S15-715, Time: Thursday 17:00, Room: Foyer)

Biogenic antioxidant stabilizers for biopolymers

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To avoid oxidative degradation of plastics, they are usually stabilized after polymerization. Due to their polarity, biopolymers are particularly susceptible to oxygen-induced aging processes during extrusion and application, usually resulting in a reduction of the molecular weight and associated polymer properties. To date, almost exclusively conservative additives have been also used for biopolymers. Some of them contain particularly critical substances in addition to their petrochemical origin. To produce fully bio-based polymer applications and protect them against premature aging, biogenic by-products from agriculture, food and forestry offer great antioxidant potential. Since the primarily polyphenol and vitamin-based antioxidants are also sensitive to thermal-oxidative interactions, gentle drying, milling, and extrusion of the byproducts are essential to achieve a reliable and homogeneous stabilization in the melt and final product. The antioxidant properties of different varieties and process histories of by-products from coffee and wine production, cocoa, orange peel, green tea and various Upper Franconian tree barks were analyzed and, after compounding, led to an increase in the stability against thermo-oxidative aging of Poly (Butylene Succinate), Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) and Poly (Lactic Acid). Although differences in the level and saturation limit of the antioxidant properties of the investigated by-products and biopolymers were found, small amounts of most biogenic by-products are sufficient to replace conservative antioxidant additive concentrations. In addition, biogenic by-products can achieve economic and other ecological advantages due to their location-independent and inexpensive availability.

Poster (S15-720, Time: Thursday 17:00, Room: Foyer)

Synthesis and characterization of zwitterionic chitosan derivative as drug delivery system for antibiotic levofloxacin.

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A wide variety of medical applications for chitosan (CS) and its derivatives has been reported over the last decades. Modified chitosan has been considered a promising material for pharmaceutical formulations and drug delivery applications. Attention has been focused on its absorption enhancement, controlled release and bio-adhesive properties. Indeed, CS is known for its biocompatibility, allowing it to be used in various medical applications such as topical and ocular applications, implantation or injection. CS has three types of reactive functional groups, an amino group, a primary and a secondary free hydroxyl group. The cationic –NH2 groups react with a number of multivalent anions to form hydrogels. In the present study, new CS derivative containing 2-Methacryloyloxyethyl phosphorylcholine (MPC) has been prepared and studied.

The modified CS was synthesized via a free radical polymerization technique at 60oC under a nitrogen atmosphere, similar to previous reports [1]. The hydrogel was fully characterized with several techniques like FTIR, DSC and XRD and then loaded with 10%, 20% and 30% of levofloxacin to study the drug release profile.

References

1.Filippousi M., et al. J. Mater. Chem. B. 2015, 3, 5991.

Plenary (S16-197, Time: Friday 08:30, Room: Sulzer Chemtech)

3D Printing for Energy Storage: Customized Processing towards High Energy/Power Density

Zhang Chuhong[1], Li Xiaolong[1], He Hanna[1]

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Modern electronics place stringent requirements on power supplies, calling for high energy and power density within restricted footprints. The idea of architecting three-dimensional (3D) electrodes exploring the benefits along the neglected height direction to increase loading density has seen great success and been well-received. The advent of 3D printing, an additive rapid prototyping technique, able to tailor arbitrary 3D structures on demand in a facile, consistent and rapid manner has attracted considerable attention for the design of 3D energy storage devices. However, the sluggish mass transport within printed bulky matrixes and intrinsic porosity associated puffy structure present serious issues to charge storage kinetics as well as volumetric energy density. Herein, we devise various 3D printing processing approaches assisted by self-healing, interfacial engineering, capillary densification etc. to successfully address the above challenges. Meanwhile, the printed device is equipped with superb mechanical compliance, a rarely achieved yet gravely desired attribute for 3D printed energy storage devices. This work puts in perspective that flexible energy storage devices with unimpaired kinetics at extremely large loading densities securing large energy output could be realized, highlighting 3D printing a powerful processing tool towards outstanding energy storage performance for a broad range of applications.

Keynote (S16-005, Time: Tuesday 10:15, Room: Angst+Pfister)

Additive Manufacturing of Functional Materials

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The design of functional polymeric materials has become much easier with the use of tools from additive manufacturing. There are myriads of problems that may benefit from functional polymeric structures, such as in removal of waterborne small molecules and oil droplets, airborne pathogen and noxious particles, and even removal of exudates from chronic wounds. However, conventional fused deposition modeling or stereolithographic techniques cannot directly produce structures that must provide high specific surface area from a set of functional polymers supported on load-bearing constructs. This talk will elaborate on a handful of examples on manufacturing porous, hairy polymer aerogel coatings on 3D-printed polymer constructs or active porous gel materials for separation of suspended liquid droplets via size-exclusion or strong adsorption of surfactants. A "scientific marriage" of the concepts of additive manufacturing and polymer science to manufacture such multi-functional materials will be elaborated in this talk.

Keynote (S16-053, Time: Tuesday 13:45, Room: Angst+Pfister)

Experimental and Numerical Investigations of the dimensions of the deposited strand in the FFF Additive Manufacturing Process

Agassant Jean-François[1], Xu D.[1], Pigeonneau Franck[1]

[1]PSL Research University, Paris, France

Predicting the dimensions of the deposited strand in the material extrusion (FFF) additive manufacturing process is a prerequisite for the design of the building path. The shape of the strand, its height H and width W, depends on the polymer rheology, the geometry and the temperature of the printing head (especially the nozzle diameter D), the extrusion velocity U, the printing head displacement velocity V, and the gap g between the printing head and the substrate. Extensive experiments have been done with an ABS formulation, varying printing parameters. The results showed that the non-dimensional strand height H/g and strand width W/D can be fitted as a function of a non-dimensional parameter, alpha=D/g U/V. This is consistent with previously developed analytical models presented at the PPS 34 International conference in Taipei and published in Additive Manufacturing. A thermomechanical multiphase numerical model is proposed. It is based on a finite element method coupled with a level-set approach to capture the interface between the molten polymer and the surrounding air. The polymer is considered as a Carreau-Yasuda purely viscous fluid. Heat transfer with the nozzle, the substrate and the surrounding air is accounted for. The numerical strand shape geometry agrees with experimental observations which justifies the relevance of the non-dimensional parameter, alpha, to define the width and the height of the deposited strand as a function of the printing parameters. Moreover, the numerical model provides complementary results as the applied pressure on the substrate and the temperature field between the printing head and the substrate which govern the welding capacity with the previously deposited strands.

Keynote (S16-143, Time: Wednesday 10:15, Room: Angst+Pfister)

Optimization of polypropylene-based materials for 3D printing processes: a detailed rheological study

Frache Alberto[1], Bernagozzi Giulia[2], Battegazzore Daniele[2], Arrigo Rossella[1]

[1]Politecnico di Torino, Alessandria, Italy
[2]Politecnico di Torino, Torino, Italy

Among all the 3D printing techniques for polymers processing, Fused Filament Fabrication (FFF) is the most used and the most developed in the global production system, being simple and economic in terms of both materials and tools. However, the limiting factor for additive manufacturing processes for polymer-based systems is the poor variety of available material. The aim of this work is the development of a deep knowledge about polypropylene (PP) focusing on its fundamental characteristics in order to be suitable for FFF process. PP-based compounds containing different amounts of talc, calcium carbonate or an organomodified montmorillonite have been formulated using a twin-screw extruder and characterized, aiming at optimizing the thermal and rheological properties of the materials. In particular, the introduction of the different types of fillers allowed reducing the melting entalphy of the PP-based systems, hence minimizing the typical high volumetric shrinkage of PP and, consequently, the severe warpage usually observed in 3D-printed manufacts based on semi-crystalline polymers. As far as the rheological behavior of the investigated materials is concerned, the results of small amplitude oscillatory shear measurements pointed out that the presence of all used fillers caused the disappearance of the Newtonian behavior exhibited by unfilled PP at low frequencies, with the occurrence of a yield-stress behavior which is progressively more pronounced as the amount of fillers increases. Furthermore, all investigated composites shown a marked shear thinning behavior at high frequencies. Therefore, the introduction of the different fillers allowed properly modifying the rheological behavior of PP, helping in ensuring an effective printability of the materials. Finally, a step-by-step optimisation of the process parameters has been conducted to fulfil the FFF filament requirements and to improve the details resolution of a 3D printed little model.

Keynote (S16-271, Time: Thursday 10:15, Room: Angst+Pfister)

Biofabrication using spider silk proteins

Scheibel Thomas[1]

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Proteins reflect one fascinating class of natural polymers with huge potential for technical as well as biomedical applications. One well-known example is spider silk, a protein fiber with excellent mechanical properties such as strength and toughness. We have developed biotechnological methods using bacteria as production hosts which produce structural proteins mimicking the natural ones. Besides the recombinant protein fabrication, we analyzed the natural assembly processes and we have developed spinning techniques to produce protein threads closely resembling natural silk fibers. In addition to fibers, we employ silk proteins in other application forms such as hydrogels, particles or films with tailored properties, which can be employed especially for biomaterials applications. We could e.g. design spider silk-based sheets and scaffolds that prevent adherence of microbes. However, the spider silk sheets and scaffolds do not kill any cells. Unlike current treatments they prevent infestation to begin with. The designed spider silk scaffolds are even bio selective, meaning that this designer silk repels microbes while allowing human cell attachment and proliferation. Spider silk hydrogels can be even employed as bioinks for biofabrication (3D bioprinting together with cells), but also non-aqueous solvents can be used to 3D-fabricate spider silk scaffolds. Their elastic behavior dominates over the viscous behavior over the whole angular frequency range with a low viscosity flow behavior and good form stability. No structural changes occur during the printing process, and the hydrogels solidify immediately after dispense plotting. Due to the form stability it was possible to directly print multiple layers on top of each other without structural collapse. Cell-loaded spider silk constructs can be easily printed without the need of additional cross-linkers or thickeners for mechanical stabilization. Encapsulated cells show good viability in such spider silk hydrogels.

Keynote (S16-340, Time: Wednesday 13:45, Room: Angst+Pfister)

Drop-on-Demand Electroprinting of Complex Fluids

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Inkjet printing is a versatile additive manufacturing tool, which has been widely used in applications ranging from graphic products to manufacturing of ceramics, even for cell engineering. To fulfill the requirements in different fields, especially in polymer, biomaterials, and ceramic printing, many methods for inkjet printing emerge in recent years. However, none of them can generate droplets on demand from highly viscous materials: the existing printing technologies fall short to eject jets from thick fluids and break them into droplets. Viscosity of inks that may potentially cause the lack of performance of materials is typically limited to the water level. To address this challenge, a new wire-in-a-tube technology for drop generation has been developed replacing the nozzle generator with a wire-in-a-tube drop generator. Electric field is used to detach and guide the drop to a target. With this wire-in-a-tube technology, we successfully formed droplets on demand from highly viscous (~10,000 cp) inks and printed them. The mechanisms of drop formation in the wire-in-a-tube drop generators are studied in detail. These mechanisms couple unique fluid mechanics, capillarity and wetting phenomena providing a new platform that can be used in different polymer processing applications.

Keynote (S16-413, Time: Wednesday 17:00, Room: Angst+Pfister)

Camphene as a Mild, Bio-Derived Porogen for Near-Ambient Processing and 3D Printing of Porous Polymers

Ellison Chris[1], Self Jeffrey[1], Xiao Han[1], Hausladen Matt[1], Bramanto Rafael[1], Usgaonkar Saurabh[1]

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Porous structures are ubiquitous in nature and have inspired various synthetic porous polymer technologies due to their advantageous mechanical and transport properties. While many manufacturing processes have been developed to generate porous thermoplastics, these usually include hazardous processes, such as high pressures and temperatures, or chemical components, and few are compatible with additive manufacturing methods such as 3D printing. In this work [1], we introduce the bio-derived terpene camphene as a solvent and porogen for freeze-casting of thermoplastic parts under mild conditions. Enabled by a low melting point (50 °C), camphene is used for melt processing camphene-polymer solutions at moderate temperatures then later undergoes room-temperature crystallization to template polymer-rich domains. Due to its high vapor pressure, camphene can be sublimed directly from these biphasic structures producing an interconnected microporous polymer. Various thermoplastic polymers are demonstrated to compatible, including polystyrene, an olefinic elastomer, a polystyrene-block-poly(ethylene-ran-butylene)-block-polystyrene elastomer, a cyclic olefin copolymer, and poly(ethyl methacrylate). The porosity and pore structures were dependent on solution composition, including both the polymer type and loading. In addition to compression molding various samples, rheological measurements were conducted on a series of polystyrene–camphene solutions to understand and optimize the formulation and conditions for direct ink write 3D printing. Porous parts with complex structures were successfully printed under mild conditions. These results underscore the advantages of camphene as a sustainable, nontoxic porogen and will inform future development of porous polymer systems derived from these methods.

[1] Self, Xiao, Hausladen, Bramanto, Usgaonkar, and Ellison, ACS Appl. Mater. Interfaces 2022, 14, 49244–49253.

Keynote (S16-523, Time: Thursday 13:45, Room: Angst+Pfister)

Molecular Engineering and Additive Manufacturing of Polyisobutylene-Based Functional Elastomers

Xu Weinan[1]

[1]University of Akron, Ohio, USA

Additive manufacturing or 3D printing of elastomers with extrusion-based methods such as fused filament fabrication (FFF) and direct ink writing (DIW) remains challenging due to the difficulty in balancing the rheological, mechanical, and thermal properties of the printing inks or filaments. The types of elastomers that have been explored for 3D printing are still limited; most of them are based on silicone or polyurethane elastomers. To overcome those limitations and bring new functionalities to 3D printing of elastomers, it is critical to explore new types of elastomers with tunable molecular structures and physical properties that are compatible with extrusion-based 3D printing. I will present our recent work on the molecular engineering and extrusion-based 3D printing of polyisobutylene (PIB) elastomers and their hybrid materials. PIB-based thermoplastic elastomers (TPEs) such as poly(styrene-b-isobutylene-b-styrene) (SIBS) copolymers and PIB-based polyurethanes have been successfully used for FFF 3D printing. The microphase-separated structure of TPEs can be precisely tuned by adjusting the hard to soft block ratio or blending with rigid homopolymers. Butyl rubber and its nanocomposites have been successfully 3D printed with solvent-assisted DIW method by adjusting the rheological, mechanical, and electrical properties with nanoclay and graphene. Soft and stretchable strain sensors based on the 3D printed structures were also demonstrated. Refs: Xu et al. ACS Appl. Polym. Mater. 2021, 3, 4554; Macromol. Rapid Commun. 2022, 2200109; MRS Commun. 2022, 12, 597–602.

Oral (S16-014, Time: Tuesday 10:40, Room: Angst+Pfister)

3D Printing-based Advanced Composites in 1D, 2D, and 3D Systems

Song Kenan[1]

[1]Arizona State University, Tempe, USA

Advanced composite materials (ACMs), especially nanoparticle-reinforced polymer composites (NpRPCs), exhibit desirable physical and chemical properties that include low density coupled with advantages of super mechanical, thermal, and functional properties. However, current manufacturing methods cannot always produce high-performance composites with the precise control of processing factors, including (i) uniform particle dispersions, (ii) effective interfacial interactions, and (iii) ordered molecular or particle structures. Therefore, new manufacturing of 3D printing involves creative design and processing mechanisms and is needed for the structural and functional versatility in complex systems (e.g., 1D, 2D, and 3D objects). Discussions of new processing-structure-property relationships in advanced composites either for or via 3D printing, especially our in-house developed manufacturing platforms, to manipulate polymer or nanoparticle orders, in addition to their applications in defense, energy, and health, will be the focus of this talk. These manufacturing methods include conventional textile engineering, coating technology, and novel additive manufacturing (3D and 4D printing).

Oral (S16-032, Time: Tuesday 11:00, Room: Angst+Pfister)

Heat Transfer and Rheological Effects on Maximum Feeding Rates in Fused Filament Fabrication

Wolff Rebecca[1], Damanik Hogenrich[2], Fatima Arooj[2], Malatyali Hatice[1], Rudloff Johannes[1], Baudrit Benjamin[1], Hochrein Thomas[1], Bastian Martin[1], Turek Stefan[2]

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[2]Institute of Applied Mathematics, TU Dortmund University, Dortmund, Germany

In the Fused Filament Fabrication (FFF) process the main limiting factor is the printing speed, as it is the case with many other Additive Manufacturing (AM) processes. To enhance the printing speed, systematic experimental investigations are necessary to understand the material dependent processes in the nozzle. In FFF, the filament partially melts inside of a heated hotend and flow is induced by the feeding force of the solid filament part, acting as a stamp on the molten polymer. In various studies, it was shown that at low feeding rates the feeding force increases linearly, while above a critical feeding rate the force increases rapidly and fluctuates until reaching a maximum feeding rate. Therefore, the polymer must reach a viscous state before extrusion. In a previous work of Wolff et al., Computational Fluid Dynamics (CFD) simulations of the FDM hotend based on experimental studies were performed using the experimental maximum feeding rate as upper boundary condition. It was shown that the numerical model can predict the feeding force with good accuracy and represent the change between process windows at increasing speeds. To calculate the maximum feeding rates, the heat transfer conditions in the hotend must be considered. In this paper, different methods for estimating the maximum feeding rates are compared for two acrylonitrile-butadiene styrene (ABS) and polypropylene (PP) materials and different nozzle geometries. If the solid filament cone reaches the contraction zone with glass transition/melting temperature at maximum feeding rates, the results match well with the experimental data. Ultimately it is possible to quantify the heat transfer in the nozzle through numerical modeling of the filament feeding force.

Oral (S16-072, Time: Tuesday 11:20, Room: Angst+Pfister)

3D Printing thermoplastic vulcanizates based on PP/EPDM

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3D Printing by Fused Filament Fabrication (FFF) allows for rapid prototyping of products for a range of thermoplastic materials without the need for additional tooling. Previously it was suggested that thermoplastic vulcanizates (TPVs), an increasingly industrially popular type of elastomer, were not printable. This work demonstrates how thermoplastic vulcanizates (TPVs) can be printed by modifying their formulation and the FFF process. For the first time, an FFF filament based on a polypropylene (PP)/ethylene propylene diene monomer (EPDM) blend was printed and characterized. The development of these TPV filaments allows a broad range of properties by varying the grades of the thermoplastic/rubber phases. Commercial elastomeric filament is typically thermoplastic polyurethane (TPU), having elongation at break of approximately 600% and tensile strength of ~30 MPa. The newly prepared TPVs possessed tensile strength of ~20-30 MPa and elongation at break ~700-1100 % when measured in the direction of printing. Tensile properties (at 0° and 90° to the printing direction), interlayer adhesion, and Micro-CT were measured and compared with PP and PLA. Given the increasing prominence of TPVs, as replacement for thermoset rubbers, it is industrially beneficial to be able to prototype these materials by FFF.

Oral (S16-076, Time: Tuesday 11:40, Room: Angst+Pfister)

High-temperature thermoplastic composites reinforced with recycled carbon fibers and thermal black for Fused Filament Fabrication

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This study investigates the development of novel formulations for high temperature thermoplastic polymer composites by mixing either amorphous polyetherimide (PEI) or semicrystalline polyphenylene sulfide (PPS) matrices with incorporation of recycled carbon fibers (rCFs) and thermal black (TB) particles followed by processing them into filaments for Fused Filament Fabrication (FFF) 3D printing. Different formulations of composites were prepared using combination of rCF and TB in which rCF content is gradually replaced by TB to investigate the possible synergistic effect on the performance of the composites. The effect of rCF and TB contents on mechanical, rheological and thermal properties of composites were investigated. With the incorporation of 20 wt.% rCF, tensile modulus and strength of the composites enhanced up to 7 and 2 folds respectively. The incorporation of TB provides flexibility for the composite filaments while maintaining thermal and mechanical performance of the composites. The rheological characterization showed that melt viscosity of thermoplastic matrices are preserved even at high loadings of TB (up to 20 wt.%). The differential scanning calorimetry (DSC) characterization results showed that incorporation of TB shifted the crystallization temperature to lower degrees and hindered the crystal formation of semicrystalline PPS matrix which could possibly provide better layer adhesion of the printed parts. Moreover,the use of TB and rCF provides significant cost saving compared to virgin CF. The obtained results will allow us to understand the developed composites behavior and optimize the FFF printing parameters for better mechanical performance of the printed parts for aerospace applications.

Oral (S16-089, Time: Tuesday 12:00, Room: Angst+Pfister)

Irreversible and Repeatable 4D Printing of FFF-printed Thermoplastic Parts

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4D printing is an advanced application of 3D printing with additional shape transformation over time. For appropriate shape transformations, various 4D printing methods have been developed using shape memory materials or multi-material structures. To be used in practical applications, however, 4D printing has to solve two challenges: (i) the manufacturing costs should be competitive; and (ii) the shape transformations must have high dimensional accuracy and repeatability. To ensure competitive manufacturing costs, this study proposes an efficient 4D printing method using a fused filament fabrication (FFF) type 3D printer and a single thermoplastic polymer without a shape memory function (ABS). To realize a shape transformation in a single thermoplastic material, we designed printing paths to have an intentional anisotropy that generates a bending deformation in response to a thermal stimulus. To obtain high dimensional accuracy and repeatability, the 4D printing method was applied while using a geometric constraint. The relevant dimensional accuracy and repeatability of the constrained and unconstrained thermal deformations were compared. The proposed 4D printing method was then applied to realize various shape transformations: (i) localized bending for a self-assembly function; (ii). in situ assemblies of a composite frame–membrane structure; and (iii) a molded interconnect device (MID) with a curved shape.

Oral (S16-097, Time: Tuesday 14:10, Room: Angst+Pfister)

Fused Filament Fabrication (FFF) with PP-based composites: effect of fillers on thermal conductivity and flammability

Bernagozzi Giulia[1], Arrigo Rossella[2], Battegazzore Daniele[1], Frache Alberto[2]

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[2]Politecnico di Torino, Alessandria, Italy

3D printing of polymeric materials is growing and increasingly used and among the most developed techniques there is Fused filament fabrication (FFF). In recent years, researches have focused on adding polypropylene (PP), one of the most widely used polymers in the world, to the portfolio of materials that are currently FFF 3D printable. Furthermore, the applications of objects produced with FFF technology must be functional and no longer limited to the simple aesthetic function. Therefore, the goal of this work is to 3D print with FFF technique PP-based composites containing both additives with flame retardant and thermal conductivity properties. Initially, samples for fire resistance tests and for thermal conductivity tests were 3D printed using FFF technique and compression moulded using commercial filaments based on ABS, for flammability, and PLA, for conductivity. In this way the printing parameters were optimized to obtain comparable results between the two techniques. Subsequently, PP-based composites were then produced by melt compounding in a corotating twin-screw extruder adding boron nitride and nanoclay. Rheological characterizations were carried out on the composites in order to verify their 3D printability. The suitable filaments (constant diameter, circularity and low roughness) to feed the 3D printer were made with PP-based composites and then samples for fire resistance tests and for thermal conductivity tests were 3D printed using FFF. For comparison, tests were also carried out on the compression moulded counterparts. The objective is therefore to verify the functionality of the fillers in the composites compared to PP, both in the compression moulded and in the FFF 3D printed samples, to assess whether the different internal structure of the samples affects the flammability and thermal conductivity.

Oral (S16-113, Time: Tuesday 15:10, Room: Angst+Pfister)

A microstructural study of the parameters affecting the interlayer bonding of semicrystalline high temperature polymers in material extrusion

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In general, the mechanical properties of parts manufactured by material extrusion (MEX) process depend upon the interlayer bonding strength. In the case of high temperature polymers such as polyaryletherketones (PAEKs), the printing parameters, in particular the temperatures applied within the process, become a critical factor. The printing-structure-property relationship for these semicrystalline high temperature polymers is complex and not fully understood, often relying on statistical analysis to identify the trends between the processing parameters and the mechanical properties, while missing the microstructural interpretation in between. By controlling the temperature settings and the printing speeds, PAEK parts can be printed into amorphous state or semicrystalline state. In both cases, molecular chain diffusion at the interface is crucial for achieving good mechanical properties. Using temperature profiles directly determined by the printing parameters, the current work investigates the formation of mobile amorphous phase (MAF) during printing and its correlation with the correspondent Z tensile strengths. MAF is defined as the fraction of amorphous phase with a higher mobility, which is beneficial to chain diffusion. The conditions required to generate MAF is explored by fast scanning calorimetry (FSC). The MAF-Z strength correlation is aiming to provide a deeper understanding of the complete processing-structure-property relationships of the MEX process for semicrystalline polymers. It may also provide a microstructural explanation on why slow crystallising PAEK grades are desirable in the MEX process. This study is concentrated on PAEKs been printed in an amorphous state to avoid the effect of crystallisation kinetics.

Oral (S16-158, Time: Wednesday 12:00, Room: Angst+Pfister)

Laser-Based Additive Manufacturing of Polypropylene-Agarose Composites: Processing Properties and Compressive Mechanical Properties

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Gelling polymers, used for the fabrication of hydrogels, are applied for various applications in medical engineering. The present paper explores a novel approach for the additive manufacturing of polypropylene-hydrogel composites by applying laser-based powder bed fusion of powder blends at ambient temperatures, hence allowing for the integration of thermo-sensitive additives. Based on applying phase-shifted, superposed fractal exposure strategies for the temporally and spatially discretized melting of prepared powder blends, the processing of composites containing a proportion of up to 75% of agarose can be demonstrated. Prepared specimens depict a water absorption exceeding 100 wt-% while exhibiting non-linear, progressive mechanical properties under compressive stress and stress relaxation properties similar to human cartilage. The results illustrate the suitability of novel discretized exposure strategies for the processing of thermo-sensitive materials with divergent thermal properties. Furthermore, the proposed approach provides a novel process route for the fabrication of functionalized polymer components, representing a foundation for the facilitated integration of hydrogels for medical applications, such as drug delivery applications and functionalized, load-bearing scaffolds.

Oral (S16-164, Time: Tuesday 14:30, Room: Angst+Pfister)

Comparing thermal degradation for fused filament fabrication (FFF) with chain or step-growth polymers

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Fused filament fabrication is an important additive manufacturing technique that can be used for part production from both chain-growth and step-growth polymers. Limited focus has however been on the impact of degradation reactions in a comparative context. In the present work, such comparison is provided, selecting polystyrene (PS) (and the related acrylonitrile butadiene styrene polymers [1]) as well as polyethylene terephthalate glycol (PETG) as reference materials [2]. It should be noted that step-growth polymers can be prone to extensive thermal degradation due to the presence of a heteroatom in the main polymer chain and chain-growth polymers can possess various structural defects for degradation initiation. Also, thermal and thermo-oxidative degradation should be minimized during processing in order to maintain the high quality of the polymer parts. We perform extensive characterization of the molecular properties of the polymer by measuring the average molar mass and molar mass distribution via SEC, melting characteristics via DSC, and melt flow behavior via steady-state and small angle oscillatory rheological tests. Based on these data, we selected the most optimal FFF printing parameters. We also vary printing parameters and study the influence of the layer adhesion during FFF on the mechanical properties of the printed parts. Additionally, the recycling possibilities of the polyester were studied. It is shown that for the PS case the filament extrusion step has an impact on the balance of thermal versus mechanical degradation contributions. For the polyester case, we observed that the influence of printing temperature and printing velocity on the mechanical properties is rather limited despite the variation of the temperature by 25 oC. At higher temperatures, we observed better layer adhesion which ensured enhanced mechanical properties of the printed parts. We demonstrate that the addition of up to 40% of the recycled fraction has a small impact on the tensile and impact properties of the material, highlighting an excellent reprocessability.

[1] Ceretti et al. Sustainability 2022 14, 15488 [2] Trossaert et al. Polymers 2022 14, 196

Oral (S16-171, Time: Tuesday 14:50, Room: Angst+Pfister)

Enhanced structural stability and interfacial strength of thermoplastic elastomers using dual material bi-layered filaments by material extrusion additive manufacturing

Patil Nikhil Avinash[1], Joshi Kartik[1], Strawhecker Kenneth[2], Lawton Timothy[3], Wetzel Eric[2], Park Jay[1]

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While FFF technique for soft materials is attracting a lot of researchers due to its advantage as a solvent free processing technique, critical processing challenges need to be addressed for fabricating soft material specimens. Present technologies fail to provide solutions for good print resolution, repeatability of the printed parts, and buckling of filament as critical processing problems. This investigation focuses on developing structural integrity and mechanical performance of thermoplastic elastomer (TPE) parts by FFF technique. The proposed approach improves 3D printing characteristics of TPE (shore hardness 75A) by assembling smaller volume fractions of ABS material, to develop circular core-shell structure of ABS and TPE respectively. Rheological characterizations of feed material using high and low shear viscometry provided an insight about optimizing extrusion parameters for filament production as well as wetting characteristics at the interface of print structures. The structural integrity and surface uniformity of produced filaments was verified by fabricating high precision 3D printed complex benchmark geometries. Izod and 3-point bending tests verify that filament structure was significant for enhancing impact resistance and flexibility of the printed part, owing to its superior interlayer adhesion. Incorporation of higher volume fractions of TPE exhibited better interlayer adhesion among the print structures, withstanding mechanical strain of 25% owing to its better interlayer cohesion. Microstructures of printed geometries under microscope verified good print uniformity of 3D-printed structures with high packing density within adjacent layers. Further, AFM modulus mapping of print structures was crucial for micro-analyzing interlayer mechanical strength among soft segments of print structure. Approved for Public Release PAO: PR2022_58311

Oral (S16-191, Time: Thursday 16:00, Room: Angst+Pfister)

Bridging experimental data and flow modelling to maximize the lifetime of additive manufactured moulds

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Additive manufacturing (AM) can be applied to make injection moulds for short product series. This method, previously referred to as hybrid injection moulding, offers a lower energy consumption and material waste during mould production compared to conventional mould making methods [1]. In this research, a multi jet fusion PA11 mould is used, as shown to be most suitable for this purpose compared to other broadly available AM materials [1]. Since the lifetime of a polymeric AM mould is much shorter than that of a conventional steel mould, this research is dedicated to identify acceptable tool wear and part deformation resulting from different injection moulding parameters. When injection moulding a high viscosity ABS grade (Trinseo, Magnum M3404) at 250°C in the PA11 mould, detrimental wear was observed after the first few injected parts [2]. Injection moulding with Lanxess Durethan B30S PA6 at the same melt temperature however, appeared to be perfectly feasible. Therefore, aspects beyond processing temperature should be taken in mind to assess processability and mould wear. Moldex3D simulation software is applied to distinguish the effect of process parameters on shear and mould temperature for low and high viscous injection material. It is proven that the polymer melt shear stress transferred onto the cavity walls is the most important factor for mould wear. This can be stated since the areas indicating high shear stress in the simulations correspond to the locations where mould failures first occur. Consequently, the higher shear stresses cause a locally increased insert temperature, decreasing the resilience of the mould material against stress. It can be stated that the most important guideline to extend the lifetime of a hybrid mould with numerous parts is to select low viscous polymers. [1] Fernandez, E. et al Sustainability 2022, 14, doi:10.3390/su14020877 [2] Fernandez, E. et al. PPI conference proceeding 2022

Oral (S16-210, Time: Tuesday 16:00, Room: Angst+Pfister)

Multi-material implant structures with medical-grade polyurethanes via Arburg Plastic Freeforming

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Additive Manufacturing offers the possibility to create personalized medicine and implants. Patient-specific structures can be fabricated quickly and reliably. The production of orthopedic implants by means of additive manufacturing could greatly benefit from multi-materials. The skeletal system is composed of hard bones and softer cartilage performing different tasks in the body. Multi-material implant structures require biocompatible materials that can withstand in-body conditions for extended periods of time. Among possible material candidates, polyurethanes have been selected for further investigation. In this study, two medical-grade polyurethanes (with established clinical history for long-term implants) produced by DSM Biomedical with a Shore hardness of 75D and 80A, respectably were used to fabricate three-dimensional structures using the thermoplastic material jetting technique (MJT) known as the Arburg Plastic Freeforming (APF). Tensile and bending specimens were produced using optimized processing parameters for the individual polyurethanes. Also, multi-material specimens were produced to analyze fracture failure at the interface between the two polyurethanes in tension. Based on the information collected in this investigation, recommendations on preparing implantable structures such as rib replacement systems, are given.

Oral (S16-217, Time: Tuesday 16:20, Room: Angst+Pfister)

Optimization of the 3D printing process of continuous carbon fiber prepreg filament

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Additive Manufacturing (AM) is a material processing technique that employs layer-by-layer production for the part. It has been successfully applied for polymer and polymer composite part production for the fabrication of high-performance lightweight components with enhanced tensile strength, flexural properties, and impact toughness. In the current research, continuous carbon-fiber-reinforced thermoplastic composites were manufactured by AM. We applied the pre-impregnation technique to produce filaments from a continuous carbon fiber bundle with polyamide 12 as a matrix. We have systematically varied the pre-impregnation parameters to study the influence of filament production on the material properties. The produced filaments were used for printing test specimens via Fused Filament Fabrication (FFF). In FFF, the polymer filament is melted in the extrusion orifice or nozzle on a moving bed to create the final product layer by layer. We also compared the influence of the printing parameters, e.g. the nozzle temperature, layer height, and inter-path distance, on the material properties of the composites. We have found that the printed composites' flexural strength and flexural modulus significantly improved by decreasing the printing temperature and the layer thickness and increasing the inter-path distance. Eventually, by optimizing the printing parameters cCF-impregnated composites could be obtained with superior flexural properties. Keywords: 3D printing, Continuos Carbon Fiber, Polyamide 12, lightweight composite materials.

Oral (S16-225, Time: Tuesday 16:40, Room: Angst+Pfister)

Liquid Additive Manufacturing - Investigation of the influence of additives and process parameters on mechanical and optical properties of liquid silicone rubber.

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Additive manufacturing of components enables individual, tool-free production of prototypes and special designs. "Liquid Additive Manufacturing" (LAM) describes the process of processing Liquid Silicone Rubber (LSR) in 3D-printing. It is among the latest manufacturing processes that can be used to 3D print parts made from LSR. The material, which consists of two components, is fed into a mixer and extruded through a nozzle onto a build platform. Then the silicone, which is thermally crosslinked layer by layer, becomes a finished part. In contrast to other silicone additive manufacturing processes, the generated part can be used directly because it is already almost completely vulcanized during production ("in-situ vulcanization"). The aim of this work is to investigate the influence of the manufacturing parameters (welche?) on the optical and mechanical properties on parts printed with LSR. In addition, the standard material is to be additivated in order to improve the rheological and the vulcanization properties to get isotropic material properties in the parts. Within the scope of this work, both proven and alternative materials are investigated. Two different LSR materials are used, one is the proven Dow Silastic 3335D for 3D printing and the other is an alternative material which is usually used for injection molding. The alternative material Shin-Etsu KEG-2003H-50 has shown promising results in preliminary tests In addition to the LSR materials, the following different additives were tested. An inhibitor, a platinum catalyst and a viscoregulator from Wacker Chemie.. In addition to the rheological properties and the vulcanization properties, the additives also influence the mechanical properties of the 3D printed part. From an initial visual inspection to transmission and surface measurements to hardness and tensile testing, the products are tested and their properties are compared.

Oral (S16-239, Time: Wednesday 10:40, Room: Angst+Pfister)

Influence of printing direction on the thermal conductivity of 3D printed polymer heat sinks

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It is no longer possible to imagine prototype development without 3D printing. In the very fast-moving electrical industry, 3D printing is also a good way to react quickliy to changing developments, for example when it comes to heat sinks made of thermally conductive material. Everything should become smaller and lighter, but also perform better than the previously used product. Here, 3D printing is ideal for the production of prototypes of heat sinks made of thermally conductive plastics. As 3D printing is a layer-building process, the thermal conductivity of the printed heat sinks is a function of the printing direction. The aim of this work was to investigate the influence of the orientation of the fillers on the thermal conductivity of filled polylactic acid (PLA). In this work, PLA was filled with 30 wt% boron nitride and printed into heat sinks in two different ways to investigate the influence of the orientation of the filler on the thermal conductivity. The printed heat sinks were then placed on a heated aluminium block and examined with an infrared camera. It could be shown that especially with platelet-shaped fillers such as boron nitride, the orientation of these in the test specimen has a very large influence on the thermal conductivity.

Oral (S16-253, Time: Wednesday 11:00, Room: Angst+Pfister)

Influence of process parameters on the porosity of 3D printed components by fused filament fabrication

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There are many 3D printing methods on the market till now which all are defined by the additively building of parts. The Fused Filament Fabrication (FFF) represents a versatile, cheap and fast method to produce volumetric parts. Nevertheless, to this date this type of production can only be used in industry to a limited extent, due to the low mechanical properties that can be achieved compared to their conventionally manufactured counterparts. Porosity, which is inherent in FFF printing owing to its geometry has hardly been considered so far although it is already considered a high influencing factor regarding mechanical properties in comparable disciplines such as composites processing. In this study, a partial factorial approach of varying process parameters like the chamber temperature, extrusion multiplier, printing temperature and printing fan has been taken. Therefore, thirteen test specimens were printed in one shot with ±45 ° infill on a Prusa i3 Mk3s to test the bending-, impact- and tensile- properties of the parts. To reach the defined environmental temperature and get rid of air draught a housing for the 3D printer was built. After the mechanical tests, samples were taken from the cross-section of the parallel part of the specimens and examined under the microscope for their void content. The mechanical and microscopy investigations show that the use of a cooling fan and the chamber temperature are the most influencing parameters on the porosity. The void content decreases with increasing chamber temperature from 3,46 to 0,35 % at 35 and 45 °C chamber temperature. Due to the faster cooling of the extruded material at the lower chamber temperature, a decrease in interface diffusion can be seen. Further, because of the higher contact temperature between the extruded paths at the higher chamber temperature a higher stiffness from 7 % respectively can be found in case of the standing specimens. The mechanical properties showed a correlation to the porosity, which leads to further investigation to control this and thus predict mechanical properties more accurately in the future.

Oral (S16-266, Time: Wednesday 11:20, Room: Angst+Pfister)

Applying bi-component melt-spinning technology on filaments for 3D-printing

Dul Sithiprumnea[1], Hufenus Rudolf[1]

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Fused filament fabrication (FFF) is a widely used 3D printing method due to its simplicity and flexibility. In this process, a thermoplastic filament is melt-extruded through a nozzle and deposited layer-by-layer by CAD filing control onto a platform. The layer structure in 3D printing inevitably leads to limited adhesion and void formation between the layers, negatively affecting the mechanical properties of the printed part. Rough and porous surfaces stemming from the collateral deposition also limit the application possibilities. To overcome these issues, in this work, core-sheath filaments were co-extruded from two different polymers. The resulting bi-component filaments featured a highly viscous polymer in the core, which can act as a rigid structure, and a low viscous polymer in the sheath, which can ideally compensate irregularities of the layer structure. Various polylactides (PLA) and polyamides (PA) were considered, provided that the sheath polymer was compatible with the core polymer (e.g. PA12 with PA6). The filaments were utilized to produce 3D components (i.e. tensile test samples) with two distinct infill patterns of 0° and 45° regarding tensile testing direction. The results showed a notable enhancement of 3D-printed parts produced from bi-component filaments. Low shrinkage of bi-component PA filaments, compared to pure PA, led to lower warpage and improved dimensional accuracy. Reduced surface roughness of 3D parts printed from bi-component PA filaments was also observed. Most interestingly, those parts also showed significantly higher tensile strength and elongation at break. On the other hand, no significant differences could be found for the PLA specimens, possibly due to the similarity of the two selected PLA polymers. It can be concluded that both printability and mechanical properties can significantly be improved by using bi-component technology.

Oral (S16-300, Time: Wednesday 11:40, Room: Angst+Pfister)

Investigation of the Volume Flow in the FFF Process as an Influencing Factor on the Resulting Orientation of Thermally Conductive Filler Particles

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In the Fused Filament Fabrication (FFF) process, a molten thermoplastic is discharged as a strand through a nozzle. This results in an orientation of non-spherical fillers contained in the polymer matrix in the direction of deposition. The consequence is an anisotropy of the material properties. In terms of the thermal conductivity of such polymer composites, the conductivity within the layers is significantly higher than between the layers. The aim of this study is to investigate the volume flow as a possible influencing factor on the orientation of thermally conductive flakes in the deposited polymer strands. The focus is on the travel velocity of the nozzle during strand deposition and on the strand geometry. By adjusting these parameters, the volumetric polymer discharge and, thus, the material behavior within and shortly after discharge through the nozzle is changed. As an evaluation criterion for the particle orientation, the thermal conductivity of the manufactured test specimens is analyzed, which shows a direction-dependent change because of the reorientation of the fillers. For this purpose, a method is used which allows a comparative evaluation of test specimens manufactured in the FFF process with Laser Flash Analysis (LFA). In addition, the process temperatures during manufacturing are considered as factors influencing the flow behavior of the polymer matrix. After manufacturing, also the resulting porosity of the test specimens is determined and compared due to the significant influence on material properties. The measurement results are validated by various methods, such as micrographs, Computer Tomography (CT) scans or Scanning Electron Microscopy (SEM) images, which document the influence of the parameters on the particle orientation.

Oral (S16-304, Time: Wednesday 14:10, Room: Angst+Pfister)

Two-photon polymerization printing of micro-dimpled surfaces: Optimization of Tribological Properties against Rubber

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Surface texturing has been recognized as an effective means to reduce friction between sliding surfaces. Optimization of textured surfaces sliding against metals and rigid polymers has been widely studied, but to our knowledge, the friction of structured surfaces against rubber has not been experimentally explored. Moreover, polymer micro-structured samples are usually fabricated using a two-step process: master mold fabrication and molding or imprinting, which is time-consuming and sometimes more costly. Therefore, this study aimed to directly fabricate millimeter-sized samples with micro-dimples on the surface using two-photon polymerization. In addition, it aimed to optimize the microstructure for friction reduction during contact with rubber. A series of printing parameter adjustments were done to ensure that the printed samples' surface and stitching quality did not affect friction measurements. With the best printing parameters, micro-structured surfaces were fabricated. Tribological tests were done using a tribometer with a rubber pin as the opposite surface. It was concluded that a two-photon polymerization method is a convenient and useful tool for precisely fabricating millimeter-sized samples for tribology studies on micro-structured surfaces. The results of this experiment are useful for applications involving sliding contact with rubber, such as low-friction syringes to replace silicone oil.

Oral (S16-313, Time: Wednesday 14:30, Room: Angst+Pfister)

First steps of the melting of an amorphous polymer through a hot-end of a material extrusion additive manufacturing

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Nowadays, 3D printing by fusing filament is a widespread technology. Nevertheless, there are questions about how it works. In particular, the understanding of the melting of the filament in the printhead is not yet agreed upon. Bellini et al. [1] assume that the polymer is completely melted as soon as it enters the printhead. Osswald et al. [2] challenged this assumption by considering that melting only occurs when the polymer and the printhead walls are in contact. In the two previous works, the polymer melting remains practically axial. However, the printhead is heated along its entire length, which is ten times longer than the filament diameter. Therefore, the heat transfer is expected to be radial. To allow the filament to be inserted, the diameter of the print head inlet is larger than the filament diameter. Then, the following question arises: does the air gap between the polymer and the walls of the printhead persist during operation? To answer this question, numerical simulations were carried out. The polymer and the air are taken into account using the level-set method. The fluid mechanics equations coupled with heat transfer and implicit interface transport are solved using a finite element method. A transient regime is highlighted. For very short periods, the filament melting occurs when the polymer contacts the nozzle wall in the presence of the air gap. Subsequently, the air is expelled by the rise of the molten polymer along the walls of the printhead. For longer times a perfect contact between the nozzle walls and the polymer is established over the entire printhead. This transitional regime lasts only a few seconds. [1] A. Bellini, S. Güçeri, and M. Bertoldi. Liquefier dynamics in fused deposition. J. Manuf. Sci. Eng., 126(2) :237-246, 2004. [2] T. A. Osswald, J. Puentes, and J. Kattinger. Fused filament fabrication melting model. Addit. Manuf., 22 :51-59, 2018.

Oral (S16-347, Time: Wednesday 16:00, Room: Angst+Pfister)

Viscous Heating Fused Filament Fabrication: Accelerating Print Speeds

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Fused Filament Fabrication (FFF) is perhaps the most prevalent additive manufacturing (AM) process in the market and has been on the forefront of AM due to the relatively low cost and broad availability of machines. However, the traditional FFF process is limited by the low melting rates due to the small force exerted on the filament. Such physical limitations to this technology results in lengthy production times which has hindered its adoption within high-volume production even though demanding engineering fields could greatly benefit from its compressed development cycle. A novel FFF viscous heating 3D printer has been patented by the Polymer Engineering Center (PEC) and has the ability of providing industry with an energy efficient method for 3D printing plastic products at higher speeds by introducing a relative motion between the nozzle and the filament. The modification to the traditional FFF system introduces viscous dissipation by rotating the nozzle surrounding the filament, accelerating melting, and ultimately increasing extrusion speeds. Results from trials with PEEK and PA66 indicate extrusion speeds increase by a factor of 3 at RPMs above 10000 and is expected to achieve faster extrusion speeds for higher RPMs. Viscous heating caused by the interaction between the filament and the nozzle surface improves the melting efficiency given that the generated heat is localized and not lost to the exterior of the system, an issue seen in conventional FFF heating mechanisms. It is crucial to have fast printing systems but to also have a process in charge of assessing print quality. In addition, the printer's image-based machine learning quality assessment system provides an effective strategy to assess the quality of the print in real-time by alerting the user when under-extrusion, over-extrusion or layer shift occurs.

Oral (S16-384, Time: Thursday 16:40, Room: Angst+Pfister)

Accuracy of a 3D-Printed Polymer Model for producing Casting mold

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This paper evaluates the possibility of manufacturing casting tools used on green-sand and cold box molding in partnership with "Hübner Fundição" utilizing Fused Filament Fabrication (FFF) extrusion-based additive manufacturing, using as-built printed part fabricated using PLA (Polylactic acid). This way obtain a cheaper alternative to the traditional methods of fabricating casting tools.Taguchi Orthogonal matrix was used to evaluate the influence of extrusion temperature, bed temperature, layer height, and infill. Nine samples were designed and printed using the configurations of the Orthogonal matrix. A laser-based 3D scanner was used for dimensional evaluation of the printed parts within the limit of ±0,2 mm with repeatability between the measured samples, making use of ANOVA and the Tukey Test to validate the data obtained. The statistical evaluation shows that there was no difference in the means of the dimensions between the samples, also extruder temperature and bed temperature were not significant to the interactions between the tool and the mold, which was affected by layer height and infill. Fewer infill results in a poor top surface finish and layer height was critical due to sand being anchored in the tool due to the higher layer surface resulting in the roughness of the outer layers. With these two variables and creating polynomials, obtained using Response Surface Method (RSM), it was possible to formulate an optimization problem for the two variables to be minimized, using sand granulometry as the restriction and the infill greater than 10%. Sequential Least Squares Programming (SLSQP) was used to obtain the optimized results with a layer height of 0.15mm and 20% of infill. With little changes to the original concept of the samples, they could be used as tools in the casting process, without the need for post-processing, seeing that the dimensional accuracy of the printed parts and the casted ones were within the constraints established.

Oral (S16-423, Time: Wednesday 16:40, Room: Angst+Pfister)

Material qualification and multi material combinations in additive manufacturing

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Extrusion-based Additive Manufacturing (EAM) is rapidly growing, since it is providing highly customizable parts with a great freedom of design while being able to process a big range of materials. Besides that, due to the low waste production, EAM offers the chance for a more sustainable process regarding material usage. An in-depth knowledge of the process-structure-properties relation in EAM is important to further qualify this process for industrial applications. Especially the great number of interfaces generated during printing as well as the internal stresses developing through inhomogeneous cooling rates between the layers, which can cause warpage or delamination, is something to put the focus on. In the M-ERA.NET project MultiMat3, materials are being qualified for EAM using Fused Filament Fabrication (FFF) as the benchmark technology and the novel Arburg Plastic Freeforming (APF). The aim is to study the influence of printing parameters on the mechanical properties and morphology of printed parts and link it to rheological and thermodynamic processes, which occur during printing and heavily affect the print result. Further, material combinations, mainly hard-soft combinations, are investigated and tried to improve using FFF and APF. Therefore, peel test specimen where the soft component is printed on top of the hard component are used and compared to specimens produced by injection molding.

Oral (S16-464, Time: Thursday 10:40, Room: Angst+Pfister)

Fused Filament Fabrication (FFF) of Aluminium Feedstock by Using Polylactic Acid (PLA) as the Backbone: Thermal Properties, Rheological Behavior, and Printability

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Material extrusion-based additive manufacturing (MEAM) processes have been extensively utilized recently because of their low-cost equipment and process simplicity. Fused filament fabrication (FFF) of metals, as a MEAM technique, has gained interest due to its similarity with metal injection molding (MIM) as a well-known process. One of the most important factors is the binder system formulation, which significantly impacts the feedstock and the final parts. In addition, Aluminium (Al) and its alloys, used in many applications, are interesting in producing lightweight components in a cost-effective process like FFF. Because of the low sintering temperature, which is near the degradation point of most polymers, and requirements for the feedstock, such as flexibility, the FFF of Al is very challenging. It is well accepted that before sintering (during debinding), the binder constituents must be entirely removed; otherwise, several mechanical defects, such as cracks, could emerge in the final product. The other problem with incomplete debinding is that high residual oxygen and carbon decrease the mechanical performance of the Al alloys. Then, binder system characteristics such as thermal and rheological behaviors play a critical role in each step and must be investigated. Therefore, using a binder system with high flexibility, which could be degraded completely at lower temperatures, could be an option to overcome the obstacles for the FFF of Al. For this reason, in this study, two thermoplastic elastomers were chosen to be used as the soluble part of the binder system due to their high flexibility and polylactic acid (PLA) as the backbone because of its low degradation temperature. The debinding results of PLA showed the high stability during solvent debinding, which is vital to help shape retention before sintering. Also, the TGA findings revealed that the degradation temperature of the samples is in the acceptable range for the Al. The rheology, TGA, and printing trials were done to analyze the printability and thermal behavior.

Oral (S16-477, Time: Thursday 11:00, Room: Angst+Pfister)

Direct ink writing of high-permittivity dielectric elastomer transducers

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3D printing of dielectric elastomer transducers would significantly accelerate their ap-plication in soft robotics. Currently, dielectric elastomer transducers are manufactured in a tedious layer-by-layer process, limiting the availability of DET for engineers. Printing these devices would ease the fabrication process and allow for various geometries and rapid prototyping. Even though facile printable DETs are highly desirable, the number of printed DET is very limited. Printability is hindered by multiple factors, such as the need for a multi-material printing of dielectric and compliant electrodes and the relatively large thickness, high stiffness, and poor mechanical properties of elastomers printed by direct ink writing (DIW). The rather large thicknesses in DIW printed structure lead to even higher actuation voltages and low sensing signals compared to traditionally produced DETs. Increasing the permittivity of elastomers is the only tunable material parameter, which can reduce the actuation voltage, or increase the sensor signal, as the minimum thickness is fixed by the printing resolution. In this work, we present DIW printable high-permittivity polysiloxanes to increase the capacitance of the printed structures. Besides the high permittivity, further material parameters and inter-dependencies between ink requirements and final material performance are explored. Here, the significant impact of ink formulation on electro-mechanical properties is highlighted. The facile printing of these high-permittivity dielectrics with standard 3D printers is demonstrated. Lastly, the performance of various DIW-printed DETs is analyzed in sensor and actuator prototypes.

Oral (S16-486, Time: Thursday 16:20, Room: Angst+Pfister)

Aerosol Jet Printing of 3D Pillar Arrays from Photopolymer Ink

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Aerosol jet printing (AJP) built on top of precise motion system can provide positioning deviation down to 3 µm, printing areas as large as 20 cm × 20 cm × 30 cm, and five-axis freedom of movement. Typical uses of AJP are 2D printing on complex or flexible substrates, primarily for applications in printed electronics. Nearly all commercially available AJP inks for 2D printing are designed and optimized to reach desired electronic properties. In this work, we explore AJP for the 3D printing of free-standing pillar arrays. We utilize aryl epoxy photopolymer as ink coupled with a cross-linking "on the fly" technique. Pillar structures 550 µm in height and with a diameter of 50 µm were 3D printed. Pillar structures were characterized via scanning electron microscopy, where the morphology, number of printed layers and side effects of the AJP technique were investigated. Satellite droplets and over-spray seem to be unavoidable for structures smaller than 70 µm. Nevertheless, reactive ion etching (RIE) as a post-processing step can mitigate AJP side effects. AJP-RIE together with photopolymer-based ink can be promising for the 3D printing of microstructures, offering fast and maskless manufacturing without wet chemistry development and heat treatment post-processing.

Oral (S16-499, Time: Thursday 11:40, Room: Angst+Pfister)

In situ characterisation of flow-induced phenomena during FDM of polymer composites using 3D X-ray microtomography

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Fused Depositon Modelling (FDM) is based on the heating of a polymer filament which is deposited by means of an extrusion nozzle of a very fine diameter (▪ 0.1 to 1.2 mm) on a substrate to build a geometric shape from a CAD file. The parts manufactured by FDM from the commercially available thermoplastic polymers are extremely varied. Polymer filaments reinforced with discontinuous fibres can also be used to obtain semi-structural aeronautic components with increased mechanical and/or physical properties. However, using FDM for the reliable mass production of components with high-added value requires a better understanding and control of the production tools and the process-induced properties. This can only be done if the flow and thermal phenomena (polymer melting, flow and cooling) occuring during the deposition are well understood and controlled. Thus, this study aims at characterizing the effect of the filament diameter and convergent angle of the nozzle on the deposition of fibre-reinforced polymer filaments (PA-6 plus glass fibres and PLA plus wood fibres) using in situ 3D imaging by fast X-ray microtomography on the BM5 beamline of the ESRF (Grenoble, France). The evolution of microstructural descriptors (porosity shape and size, fibre length and orientation, pore and fibre local volume fractions) were measured in various locations in the tested nozzles. A comparison with numerical predictions of the evolution of the fibre phase was also conducted. The knowledge acquired during these experiments will lead to the manufacturing of optimized nozzles.

Oral (S16-506, Time: Thursday 12:00, Room: Angst+Pfister)

Influence of the fiber content of different Fiber types on the mechanical properties and the printing accuracy for FFF-3D printing

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The Fused Filament Fabrication (FFF) is a useful tool for prototyping due to the high freedom of design. The dimensional accuracy was examined in different works and shows that the accuracy of FFF printing has a relative high tolerance class. The deviations of the dimensions are due to the tensions in the part due the processing, the material shrinkage and the accuracy of the drive. Therefore, the aim of this work was to determine mechanical and geometrical properties of short fiber reinforced and unreinforced PLA as a function of fiber type, fiber content and infill orientation after the FFF process. Carbon, glass and cellulose fibers were pre-compounded with 5 to 30 wt% as fillers. The materials were processed to 1,75 mm Filaments and the melt flow rate was determined. The FFF printing process was performed at 0° or 90° orientation to the longitudinal axis of the test specimen. For mechanical properties, 3-point bending test and unnotched Charpy impact test were used. Furthermore, the fracture surfaces were, which were obtained during the Charpy tests, were investigated by SEM in regard to fiber orientation and porosity. In addition to the dimensional determination of test specimens, the process accuracy of the FFF system was also determined with increasing load. Our results show that a higher extrusion rate reduces the porosity of the 3D printed specimens.

Oral (S16-509, Time: Thursday 14:30, Room: Angst+Pfister)

How to 3D-print personalized vaginal inserts from highly elastic polymers

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Pessaries enable a straightforward option to treat stress urinary incontinence (SUI) – a disease affecting more than 200 million people worldwide. Such polymer-based vaginal inserts change the position of the urethra, uterus, and/or bladder, thereby yielding immediate relief of symptoms. Since pessaries are available only in a few shapes and sizes, treatment success rates hardly exceed 50%. To circumvent these limitations this study presents the development of personalized pessaries via material extrusion-based additive manufacturing, also known as fused filament fabrication (FFF). To flawlessly process the necessary biocompatible, highly elastic thermoplastics – a requirement for inserts to avoid local tissue irritation and allow self-administration – the entire FFF process was adapted for ethylene vinyl acetate (EVA) copolymers with a VA content of 28 wt%. This material class is the golden standard for vaginal inserts, but has previously been described as non-printable by multiple sources. Here, EVA28-based 3D-printed pessaries were produced for the first time. To treat SUI efficiently, the pessary shape/size as well as its mechanics, resembling the force a pessary exerts within the vaginal cavity, were customized via the infill density and the cross-sectional diameter. Moreover, pessaries were loaded with progesterone – a relevant drug to treat SUI – yielding an innovative combination product with drug release rates tailored to the patient's needs via proper selection of 3D-printing parameters and/or via the design of multi-material inserts. The elaborated personalization concepts rely on the same filament and associated FFF process for each pessary, allowing a realistic production setting for mass customized drug products for the first time.

Oral (S16-515, Time: Thursday 14:50, Room: Angst+Pfister)

Prototyping of Degradable Meshes through Direct Ink Writing for Fertilizer Release

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The annual use of fertilizers, pesticides, and herbicides is over 200 million tons.1 The implementation of controlled release systems is of great importance to limit the excessive usage of these agrochemicals, thus protect the ecosystem compared to traditional methods in which these chemicals are deployed in bulk. So far, these systems are mostly confined to core-shell configurations—fibers and particles—which rely on complex synthesis procedures and use of solvents. Moreover, when more than one chemical is used, implementing multiple functionalization routes becomes a necessity. 2,3 Overall, there is a need to realize environmentally-conscious methods for the fabrication of controlled release systems. Here, we used direct ink writing to incorporate a commercial-grade fertilizer in a biodegradable shell made of sodium alginate and methylcellulose via room temperature, solvent-free, mechanical, green route to fabricate a core-shell system. The release of the fertilizer through the biodegradable shell to an aqueous media was tracked by FTIR, ICP-OES, and electrical conductivity measurements. Furthermore, core-shell meshes exhibited superior encapsulation efficiency of 100 % and a loading capacity of 86.8 % compared to other controlled release systems made out of particles and electrospun fibers. We evaluated the application performance of designed meshes on Triticum aestivum, the system increased shoot growth by ~2-fold and ~1.8-fold compared to the control and traditional application, respectively, in 4 weeks. Our findings have also demonstrated that even with a 30 % reduction in fertilizer load achieved ~1.5-fold growth compared to traditional application. We believe such green, facile, scalable fabrication of encapsulated fertilizers offers a lightweight, foldable, sustainable solution for controlled release and overcomes the limitations of current systems. 1 FAO. (2019) 2 Qiao, D. et al. Carbohydr. Polym. (2016) 3 Tan, H. et al. Int. J. Biol. Macromol. (2021)

Oral (S16-538, Time: Thursday 15:10, Room: Angst+Pfister)

The Role of Interlayer Adhesion on the Mechanical Performances in Fused Filament Fabrication of PLA

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Additive Manufacturing (AM) techniques have triggered industrial innovations thanks to their flexibility and the ability to realize objects with so complex shapes that are impossible to obtain by adopting conventional processes. AM constructs polymeric parts layer by layer. The AM parts generally exhibit poor and anisotropic mechanical performances, making them unsuitable for industrial applications and wherever structural properties are required. This is due to the layer-by-layer deposition strategy: the filament deposits on a previously deposited layer undergoing cooling, inducing the formation of a weld line. The interlayer bond strength depends on the molecular diffusion at the interface, namely on the possibility of the molecules recovering their entangled organization at the interface after deposition. This work investigates correlations between the morphology and mechanical properties of AM parts and correlates them to the thermomechanical history experienced by the polymer during deposition. The residual orientation is analyzed and compared to the molecular weight between entanglements (Me). The effect of deposition plate temperature and printing speed are analyzed. The decrease of the average temperatures at a longer distance from the deposition plate contributes to hiding the molecular relaxation, as demonstrated by the increase of Me with the distance from the deposition plate. Me also increases with the printing speed; this effect is more evident when conducting the test with low deposition plate temperature. A simulation code has been developed to reproduce the thermomechanical histories experienced by polymer chains during AM. Simulations consistently reproduce the orientation evolutions in the first deposited layer, and the residual orientation are consistent with the experimental one evaluated in terms of Me.

Oral (S16-559, Time: Wednesday 16:20, Room: Angst+Pfister)

Compression mode analysis of lightweight, patterned magnetoactive elastomeric structures developed with additive manufacturing

Mondal Somashree[1], Clemens Frank[1]

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Certain magnetoactive soft elastomers (MSEs) demonstrate a reversible stiffening effect under a magnetic field, also termed the magnetorheological (MR) effect. This effect strongly depends on the elastic modulus of the composite and typically increases by reducing the Shore hardness of the elastomer matrix. In this work, we expand this modulus dependency concept by investigating the additive manufacturing process of MSEs to create compliant mechanical structures with tunable geometrical stiffness, to modulate the MR effect. Material extrusion-based additive manufacturing (MEX AM) was used to fabricate lightweight, patterned structures with MSEs. The MSE comprised thermoplastic elastomer (TPE) as the matrix material and soft ferromagnetic particles as magnetic fillers. Honeycomb patterned discs with varying infill densities from 15 % to 50 % were fabricated using MEX AM. Furthermore, a step-by-step magneto-mechanical characterization under compression mode was performed to eradicate additional effects during the testing. Permanent magnet configurations, pre load, and the Mullins effect in the magnetoactive elastomer proved to have a significant effect on the MR behavior of the composite. Finally, we could demonstrate, how the varying infill density influences the MR effect. Structures with an infill density of 20% showed, at a 1% deformation range, the highest MR effect. MSE structures with an infill density between 30% and 50% showed a lower MR effect, however, samples with 100% infill demonstrated the lowest MR effect. Through FEMM simulations we could conclude that this is not solely an effect of reduced geometrical stiffness but also due to the magnetic flux distribution within the honeycomb-patterned samples which varies from the flux distribution in a solid sample. In our work, we could develop lightweight, honeycomb-patterned MSEs with improved MR effect, useful for applications that require high reversible stiffening while remaining comparatively lightweight.

Oral (S16-560, Time: Thursday 11:20, Room: Angst+Pfister)

Improved core-shell filament for accelerated annealing of fused filament fabrication (FFF) thermoplastic parts

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Approved for Public Release PAO: PR2022_37614 While great for prototyping, parts resulting from fused filament fabrication (FFF) have limited strength compared to other types of additive manufacturing. The strength is limited by interlayer adhesion, and therefore FFF is not suitable for most engineering applications. Post processing solutions, such as annealing, enable fusion of these interlayers, thus increasing adhesive strength and reducing the gap in strength between parts printed and injection molded. Initial core-shell PC-ABS filament termed 'alpha' were produced to provide thermo-structural stability during annealing but were still limited to relatively low temperatures and long annealing times. In the current work, a collection of polycarbonate grades with high heat deflection temperatures (HDT) are processed along with ABS into a PC-ABS core-shell filament. Parallel plate rheology and viscometry were performed to determine suitable processing and annealing conditions, mechanical strength determined using ASTM methods for Izod and tensile testing, and thermal stability through modified ASTM creep testing. All filament compositions were compared to select the best performing filament material pairing, noted as 'beta'. This beta filament was printed, annealed, and evaluated; ultimately displaying equivalent strengths at accelerated annealing times compared to the earlier alpha formulation of PC-ABS core-shell filament. This work highlights that careful material selection and design of a bicomponent filament geometry can lead to parts, printed with FFF, with increased strength compared to other post-processing techniques at reduced processing times.

Oral (S16-614, Time: Wednesday 14:50, Room: Angst+Pfister)

Fused Granular Fabrication (FGF) of Thermoplastic Polyurethane (TPU) Elastomers

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In this study, a fused granular fabrication (FGF) extruder for 3D printing was developed. Due to a wide range of polymers' properties especially the viscosity, the whole development process was carried out using a thermoplastic polyurethane (TPU) elastomer. Since the extruder was to be attached on the kinematic system of any standard desktop FFF 3D printers, it should be short and slim. Thus, the proposed length and diameter of the screw was 63 mm and 16 mm respectively resulting in a length to diameter (L/D) ratio of only 3.9:1. The calculations were carried out using the material properties of the selected TPU elastomer grade; and the first step was to set the shear rate normally used for extrusion, followed by calculating the best screw helix angle for 3D printing purpose, and then selecting the motor. Due to the significantly lower L/D ratio compared to standard industrial extruders (L/D ratio of 20:1 or higher) as well as this study's aim to be able to process high temperature polymers, an optimum thermal balance between the feeding zone to feed the pellets (cold zone) and the melting/extrusion zone (hot zone) should be achieved. This is to avoid the premature melting of the pellets in the feeding zone so that the pellets feeding is not interrupted as well as to ensure sufficient melt pool in the metering zone for a continuous melt extrusion. Thus, the heating and cooling blocks needed to sized accordingly. The assembled extruder was attached on a Creality3D CR-10S 3D printer to assess its performance. This includes printing tensile specimen to investigate the dimensional accuracy and mechanical properties of the printed specimen. The tensile specimens were successfully printed with a relatively high dimension accuracy. However, the printed TPU elastomer became foamy so that the printed tensile specimens had a lower stress at any strain. This could result from air trapped in the melt which can be caused by a low melt compression in the screw extruder.

Oral (S16-627, Time: Tuesday 17:00, Room: Angst+Pfister)

Development of Lower Crystalline Reactor Made 3D Printable Polypropylene: A Unique Synthetic Pathways

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Additive manufacturing enables to produce geometrically complex 3D objects, shapes, and textures in another way it's known as 3D printing. The process comprises building up a three-dimensional (3D) object from computer-aided design (CAD) models through a layer-by-layer fabrication process. This unique methodology requires less material than traditional manufacturing methods and allows the production of items that were simply not possible to produce economically with traditional manufacturing. More commonly, low-crystallinity polymers such as acrylonitrile butadiene styrene copolymer (ABS) or polylactic acid (PLA) are well known for 3D printing and easily available in the market. Whereas 3D printing of semicrystalline polymers e.g., polyethylene or polypropylene remains challenging due to large thermochemical stresses generated when the polymer solidifies on cooling. These stresses result in the warpage of the printed part. Therefore, polyolefins are still not much explored due to their highly crystalline nature and non-suitable thermal properties. Herein, we have intended to develop high-performance polyolefin-based printable material having a balance of thermal properties, which is suitable for 3D printing. We have synthesized polypropylene using Zr based metallocene catalyst through high-pressure slurry polymerization. Synthesized product characterized through MFI, GPC, NMR, XRD, and polarized optical microscopy. The thermal characteristics of the synthesized polymer measured on DSC indicated lower melting, crystallization, and % crystallinity. The microstructure pentad distribution measured on NMR indicates that metallocene PP has a mixture of isotactic, syndiotactic, and atactic also tiny Sphrurlite size of mPP measured on polarization optical microscope reflected as a highly transparent polymer. The synthesized polymer used for the preparation of 1.7 mm filament for the FDM technique, Prepared 3D article from mPP filament has no sagging and warping.

Oral (S16-661, Time: Thursday 14:10, Room: Angst+Pfister)

Fused Filament Fabrication of Functionally Graded Microcellular Foams with Variable Density

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Functionally graded foams (FGF) are known for superior and efficient energy absorption with larger failure strain characteristics. However, manufacturing FGFs using traditional methods is a challenge. To that end, we have developed an additive manufacturing method through which the density of printed polymer foam can be varied layer-wise. Foamable polylactic acid (PLA) filaments loaded with thermally expandable microspheres (TEM) were first prepared using single screw extrusion process. During the printing process, density gradient of foams was enabled in the print build-up direction by controlling the print conditions, i.e., the nozzle temperature and volumetric flow rate. Employing only a single filament, density gradients as large as 0.8 g.cm-3 (ranging from about 1 to 0.2 g/cm3) were achieved with the concurrent change in the nozzle temperature from 175 to 225°C and flow rate from 100% to 30%. FGFs having several density gradient profiles were printed and characterized with quasi-static compression and low-velocity impact testing and compared to the baseline samples having a uniform density. Stress-strain curves of quasi-static compression as well as the peak forces and energy values of the impact tests were used to assess whether the mechanical performance is dependent on the the density gradient and density orientation of the printed foams.

Oral (S16-671, Time: Wednesday 15:10, Room: Angst+Pfister)

Characterization of 3D Printed Polyether ether ketone (PEEK) samples by Fused Filament Fabrication (FFF) with a direct annealing system

Giubilini Alberto[1], Lannunziata Erika[1], Saboori Abdollah[1], Minetola Paolo[1], Iuliano Luca[1]

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Metal replacement is a significant approach for sustainable and modern manufacturing of polymer products in various application fields, such as aerospace and automotive, with the key advantage of reducing the weight of the components. Metal replacement is aimed to substitute components previously fabricated in metal with redesigned lightweight geometries, that can guarantee equal or higher performance. Technopolymers are a class of polymeric materials with increased properties, i.e., thermal and chemical stability as well as mechanical resistance, compared to traditional plastics, thus resulting as an attractive alternative for metal parts. Nowadays, Additive Manufacturing (AM) is a game-changer production technology due to its high flexibility, geometrical accuracy, reduced time and costs, and minimal waste. Therefore, an attractive research topic for technopolymers is their use in AM, also considering the final applications they are referred to. One important aspect to be considered for semi-crystalline polymers is their crystallization behavior during the extrusion in the AM process of Fused Filament Fabrication (FFF). In this work, a study was conducted on the effect that the infill pattern and density have on the FFF process for Polyether ether ketone (PEEK). Different PEEK samples were fabricated varying the infill conditions, and their mechanical and thermal properties were evaluated together with the printing accuracy and energy consumption. Furthermore, an innovative direct annealing system was tested in line with the fabrication of the specimens. The direct annealed samples were compared with the unannealed ones and with the ones that were annealed after 3d printing. The comparison highlights the advantages and disadvantages of the direct annealing approach in terms of production time, cost and energy, as well as its influence on the final thermal and mechanical properties of PEEK material.

Poster (S16-148, Time: Thursday 17:00, Room: Foyer)

The relevance of (hybrid) nanocomposites for polystyrene-based additive manufacturing

D'hooge Dagmar R.[1], Amaral Ceretti Daniel Victor[1], Van Waeleghem Tom[1], Fiorio Rudinei[2], Cardon Ludwig[1], Edeleva Mariya[1]

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Fused filament fabrication (FFF) is the most used additive manufacturing (AM) technique to 3D print thermoplastics. One of the main challenges of FFF still remains the development of new materials, compatible with the process requirements and that exhibit an improved performance when 3D printed. To this end, the fabrication of nanocomposites may be valuable, including novel hybrid nanocomposites, which contain two or more nano-sized fillers dispersed in the polymer matrix. These materials may push even further the window of applications for parts produced via FFF. In this contribution, the development of ABS-based mono and hybrid nanocomposites materials for FFF was assessed and filaments of the most promising compositions were manufactured, and 3D printed. The influence of composition, layer thickness and processing temperature on the mechanical properties of 3D printed parts was discussed. Several ABS-based mono and hybrid formulations containing graphene nanoplatelets (GNP), carbon nanotubes (CNT) and titanium dioxide (TiO2) were manufactured. The analysis of rheological properties shows that hybrid formulations containing GNP and CNT and GNP, CNT and TiO2 exhibit synergistic effects, indicating an improved dispersion state of the nanoparticles and an improved interaction between nanoparticle and matrix [1]. In addition, printability tests indicate that GNP and TiO2 improve the coalescence between deposited filaments. The performance of 3D printed parts confirms that the joint addition of GNP, CNT, and TiO2 gives rise to a remarkable synergistic effect, leading an increased tensile modulus and strength of 3D printed ABS by 16 and 20%, respectively. Decreasing the layer thickness increases the mechanical properties of the materials, while the printing temperature does not lead to major variations of the mechanical properties. [1] Ceretti, D. V. A. et al.; J. Appl. Polym. Sci. 139, 1–21 (2022).

Poster (S16-245, Time: Thursday 17:00, Room: Foyer)

Fabrication of Hierarchical Structures from Poly(Butylene Succinate-Butylene Dilinoleate)(PBS-DLS) Copolymers Containing Poly (Ethylene Glycol)(PEG) for Potential Multilayered Vascular Grafts

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Biological tissues, such as blood vessels have complex multilayered structures, and combination of different processing techniques is required to obtain such architectures. Biodegradable poly(butylene succinate-dilinoleic succinate-ethylene glycol succinate) (PBS-DLS-PEG) copolymers (synthesized via polycondemsatio) of tailored physico-chemical properties resulting in good processability. Therefore, combination of fused deposition modeling (FDM) and electrospinning has been tested to fabricate architectures mimicking native vascular walls. The chemical, physical and thermal properties of the synthesized PBS-DLS-PEG copolymers differing in segmental composition were evaluated to determine structure-properties relationship and translate it to their electrospinability and 3-D printability potential. The biological properties have also been assessed by enzymatic degradation and cytotoxicity test. The new PBS-DLS-PEG copolymers were successfully collected directly after the reaction as crystallized standard size filaments (1.75 mm) suitable for 3D printing. The chemical structure (analyzed by NMR and FT-IR) revealed formation of expected functional ester groups. The physical properties showed that changing of hard to soft segments ratio results in variable thermal and mechanical characteristics of materials, suitable for processing via additive manufacturing of FDM and electrospinning. The 3D printing of copolymers was strongly dependent on segmental composition, showing that higher hard segments facilitated good printability. Similar observation has been made during electrospinning, however the materials containing 60 wt% and 70 wt% of hard segments showed nanofibers formation. Finally, multilayered tubular porous (micro/nano) structures of 5mm in diameter were successfully fabricated through combining 3D printing and electrospinning techniques, showing potential of the new copolymers for further processing and fabrication of hierarchical and biomimetic vascular grafts.

Poster (S16-344, Time: Thursday 17:00, Room: Foyer)

Functionally Graded Materials: Innovative Multilayer composites based on Poly(D,L lactide)/Bioactive Fillers fabricated by 3D Direct Pellet Printing multi Extrusion process

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The aim of the present work was to investigate an innovative 3D manufacturing process dealing with 3D Direct Pellet multi-Extrusion (DPPmE). Throughout this study and in comparsion with the state of the art, original functionally graded materials (FGM) were obtained with tailored properties for bone regeneration. On the one hand, a deep rheological study of poly(D,L-lactic-acid) (PDLLA) without and with bioactive model fillers was firstly carried out towards a modelling of their healing properties during 3D DPPmE. The influence of DPPE processing conditions is investigated in terms of temperature, time and printing speed. For this, we propose a modelling of the process-induced interphase thickness between two deposited layers considering the non-isothermal polymer relaxation and accounting for the contribution of entanglement rate. Hence, considering the induced chains orientation and mobility coming from filament deposition, this model quantifies the Degree of Healing. Furthermore, the proposed model is validated by an experimental investigation using a tensile properties and lap shear results. PDLLA matrix and Bioglass S53P4 (BG) or hydroxyapatite (HA) bioactive fillers were chosen to obtain hybrid FGM in comparison to composite references. Different compositions and shape factors (from microspheres to fibres) of fillers were used to reach and tune the desired gradient of properties. PDLLA matrix degradation during DPPmE process was reduced. Interestingly, good interfaces and adhesion properties were obtained. Subsequently, SEM observation, SEC and tomography measurments corroborate the present findings. Besides, tensile as well as thermomechanical properties showed the potential of the DPPmE process to fabricate FGM composites with tailored architectures with this original 3D multi-extrusion printing for orthopedic applications. Rheologica Acta., 2022, Polymers 2021, and ACS Applied Polymer Materials, 2023.

Poster (S16-471, Time: Thursday 17:00, Room: Foyer)

Optimization of Porous 4D Precipitation Printing Toward the Manufacturing of Sensing Devices

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Precipitation printing (PP) is a novel additive manufacturing technique which involves printing an ink into a non-solvent bath to allow for a rapid solvent exchange and polymer solidification. Compared to traditional direct-ink-writing (DIW) techniques, this method allows for the formation of porous structures which can be useful in many applications such as sensing and energy storage. The application of this new method to 4D printing, which involves the use of shape memory materials, could allow for the fabrication of complex printed devices such as hybrid actuation/sensing structures. In this work, the potential of 4D PP is investigated to examine the effects of the porous structure on the shape memory behaviour and strain sensing potential. A polymer blend of thermoplastic polyurethane (TPU) and polylactic acid (PLA) is embedded with carbon nanotubes (CNT) to create a conductive 4D printable ink. Through controlling of the printing parameters, it is found that the porous microstructure and print resolution can be modified. Comparisons to traditional DIW also show better print precision allowing inks to be fabricated at lower viscosities. The shape memory effect is demonstrated and shows fixing and recovery ratios of 74% and 87%, respectively, and a recovery time of just over 15s. Finally, the strain sensing potential is investigated and shows a change in relative voltage of ~75% with pressing and releasing of the porous structure. The fabrication and control of porous microstructures in combination with 4D printing techniques demonstrates significant future potential for the printing of sensor devices.

Poster (S16-618, Time: Thursday 17:00, Room: Foyer)

Exploiting the potential of Soft Robotics and Additive Manufacturing utilizing PP and thermoplastic elastomers

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Additive Manufacturing (AM) is increasingly adopted in robotics and automation for its ability to provide cost-effective production of complex, lightweight and made-to-measure tools. Simultaneously, soft robotics is an emerging technology in automation where complex functionality is created using material compliance instead of rigid members and systems. A typical example of soft robotics are Fin Ray fingers, which when pressed against an object deform around it, creating a large contact surface and low contact pressure, providing the ability grab many types of objects including those of a soft or delicate nature. Many variables are used to adapt the functionality of Fin Ray fingers, including material hardness and the arrangement of internal ribbing. From a design perspective it makes sense to produce Fin Ray fingers using AM. Most AM methods are limited to manufacture of either hard or soft materials and control over hardness is achieved by means of the part geometry. An exception is Arburg Plastic Freeforming (APF), a technology utilising injection moulding screws and a droplet-based dosing system. APF is suitable for a wide variety of materials including thermoplastic elastomers and can build multi-material objects. Using APF, a gripper was designed featuring Fin Ray fingers in Shore 30A SEBS, complemented by a compliant actuation mechanism in PP. By design, the gripper functions solely on material compliance and consists of chemically compatible polymers, so that it may be constructed and recycled at end-of-life as a single component. The internal ribbing of the Fin Ray fingers was built using infill parameters of the APF build process instead of being included in the input geometry, which allows freedom to optimize gripping force through the layer jamming effect without changing the actual input geometry. Building the ribbing based on infill parameters also cut build time by over 50% compared to printing the ribbing as input geometry.

Poster (S16-712, Time: Thursday 17:00, Room: Foyer)

Advanced polymer processing technologies for mechanical energy harvesting

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To realize high-efficiency conversion and application of the large-scale discrete, unusable and wasted mechanical energy in the environment to electric energy is the key for the development of sustainable green energy, which is also the research frontier of materials science, environment, energy and other interdisciplinary. From the material preparation to structure design, a variety of flexible 3D multi-stage mechanical-to-electric conversion reinforced structure are constructed to achieve 3D piezoelectric devices with excellent performances based on the solid phase shear milling, 3D printing, controlled foaming and other advanced polymer processing methods, and the relationship between the structure and performance is deeply revealed: 1) Breaking through the limitations of conventional polymer-based piezoelectric composites such as poor interface performance, low filling and few piezoelectric crystal phase by taking the advantages of the solid-phase micro-nano dispersion and capacitive effect of solid-phase shear milling, and varieties of piezoelectric materials with high filling and high piezoelectric crystal phases suitable for 3D printing are prepared on a large scale. 2) Adopting 3D printing technology, combined with controlled foaming technologies such as SC-CO2 and chemical foaming, to achieve a series of energy harvesters with complex 3D enhancement structures, that enable "small stress to large strain" to amplify force-to-electric conversion, significantly promoted the piezoelectric outputs of polymer-based 3D piezoelectric devices. For example, the output voltage of piezoelectric PVDF device printed FDM technology is as high as 8.6 V, etc. These works provide new materials, devices and conceptions for solving the key problems of mechanical/electric conversion in the fields of energy, environment and resources in the world.

Poster (S16-714, Time: Thursday 17:00, Room: Foyer)

Advanced 3D Printing for High-performance Zinc-ion Batteries

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Aqueous Zn ion batteries (ZIBs), which hold appealing merits of high theoretical capacity, high ionic conductivity and high safety, have attracted wide attentions in energy storage systems. However, the limitation of energy density of cathode materials and the dendrite growth issues of zinc metal anode become the key to limit its industrial application. This report will focus on our research progress in the field of 3D printing customization for high specific energy density and high safety ZIBs: (1) A multi-stage pore structured three-dimensional lithium iron phosphate thick electrode (LFP-3DP) with fast electron/ion transmission channels was successfully fabricated via 3D printing technology. The 8-layer LFP-3DP electrode (30 mg cm−2) delivered an ultra-high areal energy density of 5.25 mWh cm−2, outperforming most reported 3D-printed and thick electrodes in aqueous Zinc-based hybrid-ion batteries, breaking through the energy density threshold of traditional 2D thin-film batteries. (2) Innovative design and 3D printing of "reservoir", "gradient" customized structures for dendrite-free zinc anode. The customized 3D printed structures equipped with regular micron-sized holes simultaneously remodel the electronic field/ion distribution among the electrode to regulate Zn2+ deposition behavior. In addition, the electric field/ion distribution regulation mechanisms are deeply revealed, achieving dendrite-free Zn anode with significantly promoted lifespan. The advanced 3D printing technology provides a new strategy for the development of high safety, long lifespan and high performance ZIBs.

Keynote (S17-136, Time: Tuesday 10:15, Room: St. Gallen)

Reusable and Rechargeable Biocidal Fibrous Materials for Improved Medical Protection

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The COVID-19 pandemic has drawn great attention from public and researchers to personal protective equipment (PPE), especially to face masks. The current PPE materials are defensive barriers to block penetration of pathogens through the wearable textile materials. Isolation gowns, face masks and filtering facepiece respirators (such as N95 masks) are examples of the PPE used by healthcare worker (HCW) in prevention of potential infections. However, the pathogens can survive on surfaces of the PPE materials for days and weeks and can potentially cross-contaminate and cause human infections during doffing and donning of them. The use of antibacterial and antiviral materials on the masks and medical textiles have been considered as an effective tool to inactivate the pathogens on the surfaces and consequently improve biological protection of professional and public users of PPE. Additionally, the daily uses and enormous consumptions of the PPE during the pandemic have become an environmental burden globally and add subsequent demands on reusable and biodegradable PPE materials. Ideally, reusable, antibacterial, and environmentally friendly materials should be developed for the future PPE materials. In this presentation, progresses in development of reusable biocidal (antibacterial/antiviral) and environmentally friendly textile fibers are reported. Polymer reactive extrusion process was employed to incorporate halamine moieties into olefin fibers to produce chlorine rechargeable and reusable nonwoven fabrics suitable for isolation gowns and water filters; photo-active edible compounds are blended into polymers and co-extruded into fibers and films to provide reusable daylight-induced biocidal functions. The successful fabrication and characterizations of the materials demonstrate feasibility and practicality of commercial production of the materials with anticipated future PPE applications.

Keynote (S17-348, Time: Tuesday 13:45, Room: St. Gallen)

Micro and nano engineered bio-interfaces for diagnostics, therapeutics and public health

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The biological/non-biological interface system is an important cornerstone for the fabrication of a wide range of biomedical devices. Platforms as diverse as lab-on-chip and point-of-care diagnostics, 3D tissue culture scaffolds, organs-on-chips, implants and antimicrobial surfaces all rely on the effective interaction of cells and/or bio-recognition elements (proteins/peptides, enzymes, oligonucleotides, etc.) with non-biological surfaces. Design and engineering of micro/nano patterned interfaces provide powerful tools to study biological phenomena at micro and nano scale and to develop novel technologies for diagnostics and therapeutics. I will present an overview of our research on micro/nano-scale design of novel biomedical coatings and their integration into in vitro systems such as lab on chip, flexible sensing interfaces and antimicrobial coatings as well as in vivo applications to develop efficient medical devices such as catheters and vascular grafts.

Oral (S17-010, Time: Tuesday 10:40, Room: St. Gallen)

Comparison of Droplet deposition manufacturing to Fused filament fabrication additive manufacturing techniques for the production of personalized oral tablets

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Additive manufacturing (AM) or three-dimensional printing (3DP) has enormous pharmaceutical application for the fabrication of personalized medicine in a short time. However, there are several technical challenges that need to be addressed to achieve the desired performance for efficient and sustainable personalized drug release administration. Understanding the influences of processing parameters, material properties, and manufacturing restrictions are essential to optimize the processing parameters and fabricating the final product with high quality. During fabrication of a pharmaceutical object via Fused filament fabrication (FFF) technique, an embedded drug with polymers in filament form is pushed through a heated nozzle of a 3DP. To have a successful fabrication of 3D object via FFF process, filaments with optimum mechanical properties, flexibility, melt viscosity, and thermal stability are required. As a drawback, the extruded filaments of pharmaceutical-grade polymers are either brittle which causes breakage during the process or too soft which can be squeezed aside by the feeding gear. Conversely, Droplet deposition manufacturing (DDM) is designed as a novel industrial additive manufacturing technique. DDM by requiring the plastic granulate as the feed material offers a faster process comparing to the FFF process. Following by melting the plastic granulates in the plasticizing cylinder the tiny plastic droplets layer-by-layer despite from the stationary unit. In this research two additive manufacturing processes of DDM, and FFF were used for fabrication of oral dosage form. Besides, infill density was selected as a variable processing parameter of the fabricated oral tablets. Besides the formulation, the tablets' properties were examined using scanning electron microscopy, Fourier transform infrared Spectroscopy, and ultraviolet. Increasing the infill percentage led to a slower drug release rate. The structure of tablets fabricated with FFF and DDM techniques were significantly different despite the infill percentage remained unchanged. The data confirmed that DDM as a novel technique can be applied for the fabrication of personalized medicine.

Oral (S17-023, Time: Tuesday 11:00, Room: St. Gallen)

Biodegradable 3D-printed/electrospun nanofibrous drug-eluting scaffolds for therapy of alveolar bone defects

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We developed bioresorbable nanofibrous drug-eluting scaffolds for alveolar bone repair using three-dimensional (3D) printing and electrospinning technologies. The scaffolds comprised polylactide (PLA) cages and ketorolac and amoxicillin-loaded poly(lactic-co-glycolic acid) nanofibers that imitated the morphology of the natural extracellular matrix of bone tissues. Characteristics of the printed scaffold and electrospun nanofibers were evaluated. The in vitro and in vivo release characteristics of the drugs embedded in the nanofibers were estimated using a high-performance liquid chromatography assay. In addition, the in vivo efficacies of the PLA scaffold and drug-eluting nanofibers for the treatment of alveolar bone defects were evaluated in a rat model. The experimental data indicated that the nanofibrous PLA scaffold provided a sustained release of ketorolac and amoxicillin for over 4 weeks. The results of the in vivo animal test also indicated that the animals that were implanted with the drug-eluting scaffold exhibited significantly greater movement than the animals with no scaffold. Histological analysis revealed no sign of adverse effects of the drug-eluting scaffolds. By adopting 3D printing and electrospinning technologies, resorbable drug-eluting scaffolds can be successfully manufactured for maxillofacial applications.

Oral (S17-051, Time: Tuesday 11:20, Room: St. Gallen)

Effect of graphene oxide on the mechanical properties and release profile of PLA-drug implants

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Polylactic acid (PLA) based biodegradable implants have some advantages over the non-biodegradable implants. PLA based implants release the entrapped drug and and convert into non-toxic by-products that are naturally eliminated from the body. So, no additional surgical procedure is required for the removal of PLA based implants, unlike permanent drug eluting implants (made from non-biodegradable materials). Despite the benefits, currently PLA based implants are used in limited applications compared to non-biodegradable implants e.g., titanium-based implants. The issues associated with PLA based implants are their lower tensile strength, poor hydrophobicity, poor bioactivity etc. Graphene oxide (GO) which is a 2D material, possess several unique features which can potentially be used to overcome the issues associated with PLA based implants. GO due to its high surface area can enhance the mechanical properties of the PLA. Furthermore, GO is rich with oxygen containing functional groups so when used as a drug carrier it can enhance the biocompatibility of the drug, as well as the bioavailability, and dissolution rate. One of the challenges associated with current drug delivery system is their inability to accommodate higher drug loadings. The lamella structure of GO can facilitate higher loadings of drug and can be used in applications where high drug loadings are essential e.g., cancer treatment. This work aims to investigate whether different loadings of GO can enhance the mechanical properties of a PLA-drug matrix along with investigating the effect of GO loadings on the dissolution rate. In this work, Aspirin was used as a model drug with PLA, and GO was used in three different loadings (0.25 wt.%, 0.5 wt.% and 1 wt.%).PLA-Aspirin and GO were processed using hot melt extrusion process. After processing, the effect of GO loadings on the mechanical properties, and drug release profile was studied. Scanning electron microscope (SEM) testing of the samples was carried out to investigate the dispersion of drug for different loadings of GO. As GO tends to agglomerate into larger particles and this affects the mechanical properties and dissolution rate.

Oral (S17-152, Time: Tuesday 11:40, Room: St. Gallen)

Microfluidic wet spinning of bio-based polymer fibers for biomedical applications

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Microfluidic wet spinning (MWS) has been widely used for the production of microfibers. The "wet" na-ture and mild processing conditions make it attractive for in situ encapsulation of bioactive cargoes such as proteins and living cells in fiber materials for various biomedical applications. For this purpose, different types of microfluidic devices have been developed for manipulating fluid flows in microscale, which is a prerequisite for the production of microfibers via MWS. However, microfluidic devices require complicated and tedious design and fabrication procedures, or rely on costly instrument such as 3D printers and clean-room facilities; or are prone to damage, clotting, and contamination, hence diffi-cult to be re-used. To address these limitations of existing microfluidic devices for MWS, we developed a modular microfluidic platform via facile assembly of capillaries and nozzles in PDMS elastomers. In this contribution, we will introduce the precise control over the laminar flows with such a modular mi-crofluidic platform, thereafter examples of bio-based polymer fibers produced via MWS will be given. With controlled dimensions and different morphologies, such fiber materials have a great potential for different biomedical applications, including medical textiles, biosensors, drug delivery vehicles, and tis-sue engineering scaffolds, etc.

Oral (S17-181, Time: Tuesday 12:00, Room: St. Gallen)

Experimental assessment of the penetration force of injection moulded polymer hollow microneedles in artificial skin

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Hollow microneedles (HMNs) are minimally invasive needle-like microfeatures usually arranged in arrays designed to pierce through the external skin layer for the delivery of drugs or the collection of body fluid. In a recent work, we demonstrated a novel methodology to mass produce hollow polymer microneedles at a low cost. This production technique utilises a femtosecond laser with a cross-hatching strategy and a laser-free zone to create needle-shaped micro-cavities, which are afterwards replicated using polymer injection moulding. For practical use, it is important that the replicated polymer needles penetrate easily into the patient's skin. The penetration force is highly dependent on the needle geometry, which in turn is a result of the geometry of the lasered cavity as well as the quality of the replication process. In order to assess the influence of the needle geometry on the penetration force, a test setup with an artificial skin model made of agarose gel and Parafilm layers is developed and fitted to match the penetration behaviour of a commercially available (34G) needle in porcine skin. Thereafter, this test setup is used to determine the penetration force of polycarbonate hollow microneedles with different designs and produced with different injection moulding parameters. Finally, a link is made between the geometry aspects of the microneedles (i.e. wall thickness, tip radius, taper angle, lumen diameter and replication fidelity) and the force required for penetration, and an optimal hollow microneedle design is proposed.

Oral (S17-184, Time: Tuesday 14:10, Room: St. Gallen)

Therapeutic nanofibers: Stable amorphous solid dispersion of flubendazole with high loading via electrospinning

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Unfortunately, approximately 40% of all marketed drugs and over 90% of drugs in development suffer from poor aqueous solubility, and hence bioavailability. One of the most promising routes to overcome this low aqueous solubility is that of amorphous solid dispersions (ASDs). Nonetheless, the amount of marketed ASDs is still limited. Due to their amorphous nature, many ASDs lack physical stability, over time they crystallize to their less soluble state. In this research, an important step is taken towards the bioavailability improvement of poorly water-soluble drugs, such as flubendazole (Flu), posing a challenge in the current development of many novel oral-administrable therapeutics. Solvent electrospinning of a solution of the drug and poly (2-ethyl-2-oxazoline) is demonstrated to be a viable strategy to produce stable nanofibrous ASDs with ultrahigh drug loadings (up to 55 wt% Flu) and long-term stability (at least one year). Importantly, at such high drug loadings, the concentration of the polymer in the electrospinning solution has to be lowered below the concentration where it can be spun in absence of the drug as the interactions between the polymer and the drug result in increased solution viscosity. A combination of experimental analysis and molecular dynamics simulations revealed that this formulation strategy provides strong, dominant, and highly stable hydrogen bonds between the polymer and the drug, which is crucial to obtain the high drug loadings and to preserve the long-term amorphous character of the ASDs upon storage. In vitro drug release studies confirm the remarkable potential of this electrospinning formulation strategy by significantly increased drug solubility values and dissolution rates (respectively tripled and quadrupled compared to the crystalline drug), even after storing the formulation for one year.

Oral (S17-331, Time: Tuesday 15:10, Room: St. Gallen)

Development of MIP Sensor for Detection of Acetylcholine Neurotransmitter

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Acetylcholine (ACh) is an important neurotransmitter found in the cholinergic neurons and one of the biomarkers of Alzheimer's disease (AD). AD is a neurodegenerative disease and has a role in memory loss and cognitive dysfunction. ACh has a role in memory retrieval and the learning process, but the concentration level of Ach in the case of AD decreases due to the loss of cholinergic neurons. Therefore Synaptic loss is important for the onset of the disease, while the loss of cholinergic neurons is very important for memory and learning, so detecting and targeting acetylcholine can be promising for AD patients. Many techniques exist to detect ACh in serum and CSF, but due to many disadvantages, there are many variations in the detection of concentration levels. MIP (Molecularly imprinted polymer) has a wide range of storage temperature. However, Natural receptors require human body temperature or some at -80 °C, so MIP is a promising sensor compared to the natural antibodies-antigen system to detect ACh owing to easy preparation and specificity to the particular analyte with no or less interference of other biological molecules. The polyaniline emeraldine salt (PES) was synthesized using Aniline as a monomer and Ammonium persulphate as an initiator for the polymerization. PES formation was confirmed by UV-Vis and Raman spectroscopy. PES was used as a non-imprinted polymer (NIP), while in MIP, there are ACh recognition sites in the PES. The ACh was detected using MIP/NIP modified Glassy carbon electrode in the 1X PBS buffer and ferricyanide solution in the linear range of 10-6M-10-10M ACh concentration. Electrochemical techniques such as cyclic voltammetry and electrochemical impedance spectroscopy were used to detect ACh.

Oral (S17-437, Time: Tuesday 14:30, Room: St. Gallen)

Micro-Injection Moulding of PEO, PVP VA64 and PCL for the Production of Extended-Release Tablets of Fenbendazole

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Fenbendazole (FBZ) is a broad-spectrum anthelmintic used to treat gastrointestinal nematodes in ruminants. During anthelmintic therapy, consistent exposure of worms to the drug is desired; however, divided doses can lead to sub-therapeutic concentrations of the anthelmintic, thereby increasing the risk of selecting resistant parasites. There is a need for extended-release formulations of FBZ that may minimize the problems associated with multiple-dosing regimens. Therefore, micro-injection moulding (µIM) was used to produce extended-release tablets composed of poly(ethylene oxide) (PEO), poly(vinylpyrrolidone-co-vinyl acetate) (PVP VA64), and polycaprolactone (PCL) to deliver FBZ. Thermal analysis using differential scanning calorimetry (DSC) and thermogravimetric analysis/derivative thermogravimetry (TGA/DTG) indicated the conversion of the drug to its amorphous state followed by an increase in the drug's thermal stability. Fourier transform infrared spectroscopy (FTIR) analysis did not display any new peak. Images obtained from scanning electron microscopy (SEM) displayed smooth external surface and irregular internal surface with voids and pores, whereas electron dispersive X-ray spectroscopy (EDX) demonstrated a homogeneous drug distribution. Dissolution testing using ultraviolet-visible spectroscopy (UV-Vis) revealed that an extended-release profile could be achieved through a polymeric matrix of PEO, PVP VA64 and PCL. Overall, hot-melt extrusion coupled with µIM proved to be a promising approach towards a continuous automated manufacturing process for the production of extended-release tablets of FBZ.

Oral (S17-470, Time: Tuesday 14:50, Room: St. Gallen)

Body temperature reverting shape memory polymer orthopaedic devices

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Orthopaedic fixation devices that change shape following implantation could have functional benefits. A shape memory polymer (SMP) compression screw that shortened in length following implantation in a fracture site could draw bone fragments together and thereby promote bone healing (eg scaphoid repair). Alternatively, an SMP implant that expanded radially following implantation could give improved graft-fixation during ligament repair. We have developed biodegradable and bio-inert plasticised shape memory polymers that are dimensionally stable when stored dry at room temperature but which undergo shape memory reversion when exposed to physiological conditions (saline; 37C). Results show that tributyl O-acetyl citrate (3.5% v/w) plasticised polyLDlactide die-drawn rods (2m/min; draw ratio 3.4) undergo ~25% length reduction when incubated for 55h in air or saline at 37 °C. In contrast, unplasticized die drawn PLDLA rods don't revert in air or saline at 22 °C or 37 °C whilst plasticised rods don't revert when incubated in air or saline at 22 °C (< 2% length reduction after 24h; TAC-PLDLA air 22 °C vs air 37 °C $p = 0.004$). Plasticiser effect on reversion temperature was dose dependent. More hydrophobic citrate esters had greater effect on Tg. Parallel studies showed that 10% v/w triacetin plasticised PET-G gave a bio-inert shape memory polymer that was dimensionally stable when stored dry at room temperature but which reverted when exposed to saline at 37 °C. We are using these SMP materials to develop prototype (i) hybrid metal and bio-inert shape memory polymer devices for ligament repair and (ii) bioresorbable SMP compression screws with zonal reversion properties for small bone fracture repair. The SMP components of the bioinert devices are made using conventional solid-phase die-drawing techniques. The bioresorbable SMP compression screws (which have zonal reversion properties) require computer-controlled 3D printed metal moulds with precise zonal heating, cooling and heat transfer.

Oral (S17-512, Time: Tuesday 16:00, Room: St. Gallen)

A long-lasting sequentially functionalised atelocollagen membrane for Guided Bone Regeneration (GBR) therapy

Tronci Giuseppe[1]

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Collagen-based membranes are widely employed for GBR therapy, aiming to prevent soft tissue infiltration during bone regeneration. [1] However, the fast membrane degradation in the biological environment remains a critical challenge. Here, the sequential functionalisation of medical grade telopeptide-free collagen (i.e. atelocollagen, AC) was hypothesised to generate a UV-cured covalent network with GBR-compliant functionalities, i.e. controlled proteolytic stability, barrier functionality and mechanical competence. [2] The UV-cured product was comprehensively characterised with respect to its gelation kinetics (rheometry), compression properties, swelling ratio and gel content, which confirmed the presence of a crosslinked network at the molecular scale. A significantly higher proteolytic stability was measured in the UV-cured product with respect to Bio-Gide®, which was found to completely dissolve after 14 days in vitro. Following 7-day culture, L929 fibroblasts displayed increased metabolic activity and a surface-confined proliferation, agreeing with the relatively small micropores in the UV-cured sample (Ø≤ 10 μm) and indicating soft tissue barrier functionality. The UV-cured sample displayed material integrity for at least 4 weeks in a subcutaneous model in rats and following 4-week implantation in a GBR model. In the latter case, an island of densely packed tissue was observed at the centre of the GBR defect, supporting the barrier functionality and the integration of the collagen material with the surrounding bone in vivo. The presented design strategy proved to successfully generate a collagen material with competitive macroscopic properties and proteolytic stability with respect to Bio-Gide®, and could offer a simple and regulatory-friendly platform for the manufacture of long-lasting membranes for GBR therapy. [1] Sanz, M. et al., J. Clin. Periodontol., 46(S21):82-91 2019 [2] Liang, H. et al., Acta Biomater., 140(1): 190-205, 2022

Oral (S17-536, Time: Tuesday 16:20, Room: St. Gallen)

Polycaprolactone fibers/bioactive glass nanoparticles composites for Guided Bone Regeneration

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Fibrous membranes comprising of polycaprolactone (PCL) and bioactive glass nanoparticles (BGn), at different amounts, were prepared by the green electrospinning method for potential use in guided bone regeneration (GBR). The characteristics of the membranes were assessed first by FTIR, SEM, TGA, and DSC. Wettability was studied by measurements of the static water contact angle. In vitro biodegradability and bioactivity were evaluated through immersion in phosphate-buffered saline (PBS) and simulated body fluid (SBF) solutions, respectively. TGA, FTIR, and SEM analysis showed that the BGn were homogeneously dispersed in the PCL matrix. Furthermore, BGn's introduction enhanced the uniformity and alignment of fibers and helped reduce the hydrophobicity of PCL. DSC analysis indicated that an increase in PCL's crystallinity was induced due to the nucleation effect of BGn. In addition, in vitro studies demonstrated that biodegradability and bioactivity could be tailored as a function of the BGn content. Indeed, excellent biomineralization was obtained at higher BGn concentrations (10-15%wt) with a matching biodegradation rate. These results suggest that the electrospun composite mats could be used as barrier membranes in GBR.

Oral (S17-594, Time: Tuesday 16:40, Room: St. Gallen)

Novel biodegradable subcutaneous implants manufactured via high-resolution 3D-printing

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35.2% of the adult population in Europe suffer from at least one chronic disease. These diseases need to be treated over a long period of time or even throughout life with medications that are usually administered orally. However, only half of these patients take their medications as prescribed, making improving adherence a priority on the public health agenda. Subcutaneous implantable drug delivery systems (SIDDS) represent a promising approach to solve this problem. SIDDS are implanted into the subcutaneous tissue and deliver drugs over prolonged times. They are typically manufactured using a hot-melt extrusion process and follow a one-size-fits-all approach in terms of daily dose and duration of use. However, an individualized treatment plan that considers patients' physical differences and disease progression is critical for a successful treatment. In the present study, we present an innovative processing platform, enabling the personalized production of SIDDS for the first time via 3D-printing. Novel biodegradable thermoplastic polyurethane multiblock copolymers custom-synthesized for fused filament fabrication are processed at nozzle temperatures as low as 135°C to produce rod-shaped implants. The implants have a cross-sectional diameter of 2 mm and are 40 mm in length, which corresponds to the size of commercial products. With nozzle sizes of 100 µm and layer heights as small as 25 µm, porous implants with complex internal structures can be printed flawlessly at economic print times. These results set the basis for the production of SIDDS that can be tailored towards the patients' needs in terms of daily drug release and degradation period through the adaption of 3D-printing parameters.

Oral (S17-624, Time: Tuesday 17:00, Room: St. Gallen)

Fabrication of a collagen-based hydrogel wound dressing with visual infection-sensing capability

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Chronic wounds fail to proceed through an orderly self-healing process and represent a major healthcare burden worldwide. Bacterial infection is one of the major causes of wound chronicity, generating risks of gangrene and amputation. Cost-effective technologies enabling prompt detection of bacterial infection and integration into wound dressings are therefore needed to assist with therapeutic decisions and minimise risks of antibiotic misuse. We investigated whether the design of a theranostic dressing could be realised by integrating an infection-responsive system, targeting the pH as a biomarker of wound infection, with a collagen-based wound contact layer with previously-demonstrated wound healing capability. Bromothymol blue (BTB), a halochromic dye, was integrated into the dressing to introduce long-lasting visual infection-sensing capability through the retention of BTB within the dressing. Dressings had an average BTB loading efficiency of 99 wt.% and displayed a colour change within one minute of contact with simulated wound fluid. Samples retained up to 97 and 85 wt.% of BTB after 96 hours in a healthy and near-infected wound environment, respectively. Long-lasting dye confinement and durable dressing colour change are attributed to the formation of secondary interactions between the collagen-based hydrogel and the BTB. Evidence of this was seen as an increase in collagen denaturation temperature (DSC) and red shifts (ATR-FTIR). The addition of BTB did not affect the morphology and mechanical properties of the collagen hydrogels. Dressings were shown to be cytocompatible, given the high L929 fibroblast viability in sample extracts, 92 %, after 7 days. Therefore, this simple, scalable, and cell-/regulatory-friendly wound theranostic design offers a new platform for the development of advanced dressings; aiming to support wound healing, reduce hospitalisation time, and enable informed and personalised variations in clinical care.

Poster (S17-204, Time: Thursday 17:00, Room: Foyer)

Fabrication of layer-by-layer polyelectrolyte coated spray-dried PLGA microparticles for trametinib sustained delivery

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Recent strategies for the development of drug delivery systems suggest the use of biobased and biodegradable aliphatic polyesters, for the effective administration of a broad range of active ingredients. Poly(lactic acid) (PLA), poly(ε-caprolactone) (PCL), poly(alkylene succinate)s and polyglycerol hyperbranched polyesters have already found various applications in the field of pharmaceutical technology. Among them, poly(lactic-co-glycolic acid) (PLGA) has attracted significant attention due to its tunable biodegradability and versatility. Numerous PLGA-based drug-carriers have been developed over the past decades, whilst controlling their therapeutic effect and sustaining the drug-release rate remains a challenging issue, particularly in the fight against cancer. In the present work, trametinib (GSK1120212) that strongly inhibit the activities of MEK1 and MEK2 kinases and is currently under investigation for its effectiveness as anticancer agent, was encapsulated in commercial PLGA (with 65/35 w/w lactide to glycolide ratio) microspheres. The preparation was performed via spray-drying method to obtain nicely formed spherical particles in microscale, with high drug loading capacity. The prepared microparticles were then coated with a multilayer polyelectrolyte complex of chitosan/alginate (CHI/ALG) to further tune the sustained-release behavior. The formulations were characterized using Fourier-Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction Analysis (XRD), and Scanning Electron Microscopy (SEM) and their in vitro dissolution rate was studied. Their ex vivo effect on MEK1/2 activity was evaluated by kinase translocation reporter (KTR) assay using the GFP-ERF fusion protein in HeLa cell. Subcellular localization was analyzed by confocal microscopy.

Poster (S17-416, Time: Thursday 17:00, Room: Foyer)

Synthesis and in-vitro characterization of new PLA-b-PHSu microparticles for long-acting injectables of risperidone drug

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Long-Acting Injectables (LAIs) are controlled-release drug delivery systems that are administered intramuscularly, creating a "depot" of drug which is available at the required concentration in the human body. In this work, microparticles from poly(lactic acid)/ poly(hexylene succinate) block copolymers (PLA-b-PHSu) with encapsulated risperidone drug have been synthesized. The scope of this research was to synthesize new risperidone microparticles that will overcome the slow hydrolysis that the commercial form of the risperidone drug delivery system has. PLA and PHSu are biocompatible and non-toxic thus they are perfect candidates for medical applications. Moreover, PHSu was obtained to enhance the degradation rate of PLA. Spray-drying was used to synthesize risperidone-loaded microparticles, which were thereafter studied in vitro. The drug loading and the drug entrapment efficiency of microspheres were calculated by HPLC measurements. Moreover, the dissolution rate of risperidone from the prepared microparticles was investigated. Scanning Electron Microscopy was used to examine the structure and surface of the prepared microparticles.

Poster (S17-438, Time: Thursday 17:00, Room: Foyer)

Laser processing of thermoplastic polymer for precision sealing of microfluidic medical devices

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The microfluidic medical industry is moving towards a thermoplastic platform for fabrication due to its ease of production. With the current trend towards functionality integration and miniaturization, the laser has been demonstrated to have possible applications as a lithography-based fabrication tool for micromachining as well as additive manufacturing of thermoplastics. In this study, we take a look at the laser material interaction using a Nanosecond pulsed laser and cyclic olefin copolymer (COC). Effect of laser parameters on the material melting and the heat affected zone changes with different parameters. This study is aimed to better understand the possibility to integrate laser as a production tool for cutting, making channels/wells and sealing COC-based microfluidic devices and how to evaluate such a study. This could potentially lead to a one-step production for thermoplastic-based microfluidic medical devices.

Poster (S17-657, Time: Thursday 17:00, Room: Foyer)

Ultrafast laser processing of high-performance polymers for rapid prototyping applications

Haasbroek Pieter Daniel[1]

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Subtractive manufacturing offers high speeds, efficacy, and superior surface quality for a broad range of materials. Whereas the ablation behaviour of metals, glass and ceramics is well understood, the laser micromachining of polymers is still poorly understood. We have studied the ultra-short pulse laser ablation of PI, PEI, PC and PEEK as potential candidates for cost-effective 'rapid prototyping' of moulds for injection moulded medical devices. Making use of these strong and heat-resistant polymers would allow accelerated mastering during the development of a new medical device and other surface-textured applications. However, there still exists a lack of sound fundamental understanding of laser micromachining of polymers, especially for thermoplastics. Phenomena like the saturation effect at high fluence processing, non-linear absorption of thermal energy, and laser beam de-focussing due to the Kerr effect, adversely affect the surface integrity of polymers, thereby hampering their performance. The process window and pulse energy range for laser micromachining of said polymers have been determined using a femtosecond laser source. Results from single-pulse, multiple-pulse and areal ablation experiments are correlated with the thermal and optical properties of the polymers. This not only provides a deeper understanding of the regimes of controlled ablation (linear increase in ablation depth with the number of laser pulses) and further optimisation concerning pulse energy per irradiated area (Fluence), but also provides the basis for the development of predictive numerical models that can describe the laser ablation process in thermoplastic polymers. Ultimately, such a prediction would be used to inform the laser control software to make the necessary adaptations to the energy and scanning parameters, in-process, to laser machine polymer surfaces with maximal accuracy.

Keynote (S18-047, Time: Tuesday 10:15, Room: Bodensee)

Experimental Measurements and CFD-Simulation of a Viscous Adhesive Injected into an Unsealed Channel

Jousset Pierre[1], Gutgsell Michael[2], Kuster Max[1], Wenig Sabine[3]

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Adhesive joints are increasingly being used in car body construction and in the industry in general. In order to enable high load-bearing capacity of large joining surfaces and tolerance compensation, adhesive bonding technology is used as a joining technology for complex and highly stressed components. In a semester work at the OST/IWK together with the company Sika, an epoxy based adhesive from Sika has been applied by injection into a laterally open channel. At this stage and before having cured, the adhesive is not in a solid state and exhibits a pasty and viscous behaviour. This adhesive is characterized by particularly controlled spreading during injection in unsealed channels. It has a unique temperature-dependent viscosity behavior which makes it very attractive for industrial applications using an injection process. To analyze the spreading behavior of the adhesive and the limits of the injection process, the flow behavior of the adhesive was modelled using Finite-Element (FE) simulation. Using material parameters identified from an inverse identification process, a suitable material constitutive model has been calibrated and the prediction of numerical results have been compared with experimental measurements performed on real tests. The sensitivity of the process parameters potentially influencing the rheological behavior of the adhesive, such as adhesive and substrate temperature, injection speed, channel geometry and tolerances, were investigated in relation to the spatial and temporal propagation of the flow front. Results have been validated using a laterally open injection channel test fixture especially developed for this purpose. The FE-simulations results show a correlation between the process parameters and the shape and extent of the adhesive during its spreading in and beneath the unsealed injection channel. This behavior was confirmed in the experimental tests carried out, despite some deviations. As a conclusion, results of this work settle the basis for a guideline for the use and flow simulation of this adhesives, helping car body manufacturers to design their joints and production processes in a more targeted manner.

Keynote (S18-320, Time: Tuesday 13:45, Room: Bodensee)

Atmospheric gliding arc plasma deposition: A novel tool to selectively change the wettability, roughness and surface chemistry of substrates

Michl Thomas Danny[1], Goel Alok[1], Sroka Karolina[1], Neuhaus Sonja[1]

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Controlling the three key surface properties, wettability, roughness and surface chemistry, is essential for many industrial applications. Such industrial applications can range from glueing, inking, corrosion control to selective attachment of active molecules on the sur-faces. It is crucial to control these three sur-face properties without changing the materi-al's bulk properties. Recently, atmospheric plasma processes have leapt forward and thus gained widespread attention. Like the low-pressure plasma processes, atmospheric plasmas are fast, solvent free and environ-mentally friendly. However, unlike their low-pressure counterparts, atmospheric plasmas can be readily implemented into existing production lines and easily scaled up. Specifical-ly, atmospheric gliding arc plasma (AGAP) combines homogeneity and high-power den-sities compared with other atmospheric plas-ma methods. We hereby demonstrate how a commercially available system (Plasmatreat©) can be used to rapidly deposit novel thin film coatings. These thin-film coatings selectively change surface wettability, roughness and chemistry. This deposition requires only elec-tricity, benign & readily available gases and small amounts of monomer precursor. By avoiding the use of noble gases or toxic pre-cursors, contrary to other atmospheric plasma methods, AGAP is a viable and safe option to run on an industrial scale to coat a variety of substrates; ranging from nonwovens to met-als. We expand our knowledge by connecting the macroscopic effects, such as adhesion, with the microscopic; namely by extensive surface analysis using XPS and TOF-SIMS. Keywords: Surface engineering, surface modification, thin film coatings, atmospheric plasma, gliding arc plasma, wettability, roughness, surface chemistry, XPS, TOF-SIMS, surface analysis

Oral (S18-013, Time: Tuesday 10:40, Room: Bodensee)

Alternating current based electrochemical deposition of pure emeraldine salt redox state of polyaniline to modify Mw-CNT/polyester microfiber nonwoven based flexible electrode for microbial fuel cell

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In the present study, we propose the fabrication of a high-performance electrode by the utilization of alternating current (AC) for electrochemical polymerization of aniline to form a uniform coating of dark green colored amorphous chains of pure polyaniline (PANI) emeraldine salt redox state. Multiwalled carbon nanotubes (MW-CNT) based electrically conductive ink modified polyester microfiber nonwoven substrate was employed as working electrode in a three-electrode system immersed in-stock solution of aniline/aqueous HCl based electrolyte to obtain emeraldine salt redox state of PANI. A low-frequency AC signal of known set voltage and amplitude via electrochemical impedance spectroscopy was employed. Development of emeraldine salt redox state-based PANI-MW-CNT ink modified polyester microfiber nonwoven textile improved the conductivity, surface roughness, along with hydrophilicity, and biocompatibility of the electrode material which can be used in a microbial fuel cell (MFC). Industrial-scale operation of the developed electrode material in MFC would reduce the overall costs of wastewater treatment systems due to its ability to provide energy positive or at least energy neutral treatment ability. Finally, the operationally cost-efficient flexible electrode would promote its utilization to treat high COD wastewaters (E.g. textile wastewater), which became a serious cause of water pollution and severely affected the lives of human beings and animals. Keywords: Polyaniline; Flexible electrode; Electrochemical polymerization; Microbial fuel cell; Nanocomposite

Oral (S18-243, Time: Tuesday 11:00, Room: Bodensee)

An Experimental Study of Polymer-Polymer Interdiffusion under Co-Extrusion Processing Conditions

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Interdiffusion – defined as the mutual interpenetration of macromolecules to form a diffuse interphase – is observed at polymer-polymer interfaces in a wide range of plastics processing techniques. This transport phenomenon has been studied extensively on static interfaces (i.e., the polymers and, in particular, the interface is not subject to deformation rates), for instance, in plastics welding, 3D printing, and consolidation of thermoplastic UD tapes. In contrast, interdiffusion under shear-loaded conditions as present in stratified flows during co-extrusion has been studied scarcely to date. Under real co-extrusion processing conditions, employing a two-layer co-extrusion demonstration die and the material combination PMMA and SAN, this work presents the effect of interfacial contact time, of extrusion melt temperature, and of the magnitude of the interfacial shear stress on the interdiffusion rate. The implemented model-based digital process twin enabled accurate adjustment of the magnitude of aforementioned process parameters in the die. The diffuse interphase of the calibrated two-layer sheets was then characterized spectroscopically by means of confocal Raman microscopy. By measuring the breadth of the diffuse interphase region and deriving the mutual interdiffusion coefficient we revealed that – similar to static conditions – interdiffusion during co-extrusion flows scales with contact time and temperature according to Fick's laws and an Arrhenius relationship, respectively. Moreover, already low interfacial shear stresses significantly boosted the rate of interdiffusion compared to static conditions. Though, a systematic variation of interfacial shear stress levels between approximately 1,000 and 40,000 Pa showed no further impact on the mutual interdiffusion coefficient (i.e., rate of interdiffusion).

Oral (S18-248, Time: Tuesday 11:20, Room: Bodensee)

Plasma etching of recycled PET film in Roll-to-Roll (R2R) processing

Amberg Martin[1], Höhener Marion[1], Perret Edith[1], Rupper Patrick[1], Hanselmann Barbara[1], Hufenus Rudolf[1], Hegemann Dirk[1]

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Europe is one of the most advanced regions regarding plastics collection and recycling, recycling rate is at 32%, 43% is energy recovered and 25% is still landfill. European countries lack the capacity to manage increasing amounts of plastic waste in circular and sustainable ways. In this study a PET heat reflector film for cargo containers is replaced by rPET film material. The metallized rPET film is thermoformed into a honeycomb-like structure as used in the main component of the cargo container wall. The question discussed here is: Can a low-pressure Ar/O2 plasma pre-treatment of rPET be used to achieve the desired adhesion for the metallization comparable to virgin PET, suitable for a thermoformed heat reflecting layer? Low-pressure plasma etching of rPET is therefore investigated in comparison to virgin PET and polypropylene (PP) using a large R2R plasma reactor with capacitively coupled radio frequency (RF) excitation. Mild plasma conditions at floating potential are selected to preserve the material properties of the polymers and to achieve an industrial two-sided treatment. The etch rates of the polymer samples are determined at floating potential and directly on the electrode for different Ar/O2 gas mixtures, etching duration, and sample size. Thermoanalytical techniques are used to characterize the polymer before and after the plasma etching treatment. The morphology of the polymer substrates is investigated with respect to their position in the plasma, i.e. at the RF electrode or at floating potential (facing the electrode and opposite side), which depicts the influence of particle bombardment from the different reactive plasma species. Finally, adhesive-tape peel tests prove that excellent adhesion of silver coatings can also be achieved on recycled PET film using R2R plasma processing, which opens up the industrial use of recycled polymer material for cargo containers and further applications.

Oral (S18-264, Time: Tuesday 11:40, Room: Bodensee)

Surfaces forces on nano-porous plasma polymer films

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Nano-porous SiOx plasma coatings can be used for a broad range of applications such as anti-icing or self-cleaning surfaces, taking into account the unique properties of the coating such as the wettability of the film [1] or their function as a diffusion layer [2]. Plasma-enhanced chemical vapor deposition (PECVD) gives the possibility to fabricate highly networked plasma polymer films (PPFs) derived from HMDSO. The PECVD processes allow to tune the plasma operating parameters such as gas flow rate or power, resulting in hydrophobic and nano-porous PPFs [2]. The porous films form interconnected voids due to a Si-O ring network structure, allowing the diffusion of water molecules into the nano-porous structure at room temperature [3]. The result is a nanometer thin polymer film that can be hydrated. Despite extensive investigations on HMDSO-based coatings, there is still a lack of comprehensive understanding of the surface forces occurring on such nano-porous surfaces, especially in contact with aqueous liquids.

To detect how this polymer layer affects the vicinal liquid, we study the surface forces appearing on nano-porous SiOx plasma polymer surfaces. Two different techniques, namely atomic force microscope (AFM) and extended Surface Force Apparatus (eSFA) are used to investigate physical phenomena occurring at different scales. The studied SiOx (CHx) coatings derived from HDMSO/Ar/O2 plasmas were deposited on thin ruby mica substrates in a capacitive coupled parallel-plate RF reactor. The measurement of the forces on the surfaces corresponds to the change of the free Gibbs energy of the fluid, which in this case is expected to vary with time. Solute adsorption measurements of the SiOx plasma polymer coatings in water can reveal information about the expected dipolar orientation of water molecules on such surfaces on nanometer scale.

[1] Ma et al., International Materials Reviews, 2022, https://doi.org/10.1080/09506608.2022.2047420
[2] Gergs et al., Plasma Process. Polym. 2022, https://doi.org/10.1002/ppap.202200049;
[3] Hegemann et al., Plasma Process. Polym. 2021, https://doi.org/10.1002/ppap.202000176;

Oral (S18-324, Time: Tuesday 12:00, Room: Bodensee)

Transfer of PECVD Coating Processes to other reactors by Means of Plasma Diagnostics and Coating Characterization

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Due to low heat impact plasma enhanced chemical vapor deposition (PECVD) processes are predestined for surface engineering of plastics. Different monomers, such as silicon based hexamethyldisilazane (HMDSN) and hexamethyldisiloxane (HMDSO), have been investigated to alter the surface and barrier properties of plastics. Transferring the results from laboratory scale to an industrial application has been a challenge since plasma properties are highly dependent on the reactor dimensions, energy distribution, monomer flow and distribution, etc. Therefore, each reactor has its own unique working point for the desired coating properties and a working parameter set has to be determined by extensive testing. Finding a reactor independent correlation between fundamental plasma properties and the resulting coating properties and understanding their dependencies is a viable tool to increase reproducibility as well as prediction of coating properties and monitoring of PECVD processes. In this study plasma properties in two reactors with different means of introducing microwave radiation were investigated. Optical Emission Spectroscopy (OES), Multipole Resonance Probe (MRP) measurements were therefore correlated with investigations of coating properties such as morphological, chemical and physical properties. An intersection of the parameter space of both reactors was found where the plasma properties such as electron density, electron temperature, etc. and the monomer and auxiliary gas fluxes are nearly equal. Investigations by Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM) and Infrared Spectroscopy (IR) of the coatings are carried out in order to analyze causes for different coating properties.

Oral (S18-325, Time: Tuesday 14:10, Room: Bodensee)

Cerium oxide nanoparticles deposition on poly(lactic acid)- tricalcium phosphate by different methods: high power impulses electrophoresis deposition and plasma surface activation

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Pessan Luiz Antonio[2], Costa Lidiane Cristina[2]

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The poly(lactic acid) (PLA)-tricalcium phosphate (TCP) composite is a promising material for bone tissue regeneration because of its good mechanical properties, body temperature stability, and biocompatibility. However, its low surface energy and low hydrophilicity make cell growth and attachment difficult. Nanoparticles can be deposited on the scaffold's surface to overcome its limitations. Cerium oxide nanoparticles (CeNPs) are gaining attention due to their antibacterial and anti-inflammatory properties. This work aims to evaluate different CeNPs deposition methods on the PLA-TCP scaffolds surface. We applied two different deposition techniques: high power impulses electrophoresis deposition (HI-EDP) and plasma surface activation followed by deposition in solution. In addition, the influence of the solvent (water and ethanol) on the homogeneity of the deposition was evaluated. The impulsed electrical current consists of a pulsed train with five positive pulses around 1200 V, with 1 µs duration and 200 µs between pulse trains. Two HI-EPD configurations were used: the scaffolds were in the solution, which was mechanically stirred throughout the process, and the scaffolds were tied on a steel grid placed on the unbiased electrode, with the solution immersed in an ultrasound bath to ensure homogeneity. Plasma surface activation was carried on a glass reactor containing an active screen to minimize the damage on the scaffold's surface, powered by a DC source and pure Ar as the gaseous precursor. Microscope analysis was performed to verify the dispersion quality after the deposition. It was observed that both the surface activation mode (HI-EDP or plasma) and the solvent affect the dispersion quality.

Oral (S18-350, Time: Tuesday 14:30, Room: Bodensee)

Interfacial Anchoring of Chiral Nematic Photonic Films of Organic Acid Doped Cellulose Nanocrystals in Biocomposites Laminates

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One of the key components in optical, bioengineering, and sensing applications is an organic photonic film. It is challenging to produce chiral nematic biopolymer composites with outstanding mechanical strength and flexibility without sacrificing their spectacular iridescence (structural color). Herein, we have proposed an approach to preserve and lock the self-assembled chiral cellulose nanocrystal (CNC) films based out of agro-waste between the layers of poly (lactic acid) (PLA)/ polycaprolactone (PCL) biocomposite blend. The photonic properties of the films were tuned by doping with different organic acids (OAs). The multilayer films were studied for the interfacial interactions between CNCs and polymer blends. The effects of OAs with different acidic strengths on the morphology, optical and mechanical properties were studied. The films exhibited high mechanical strength and a selective reflection band from UV to the near-IR region. The composite films displayed good visual color sensing properties with moderate antibacterial activity for Escherichia coli bacteria. Overall, the results demonstrated that the layered composite film could be a candidate material for bio and optical engineering having implications in intelligent packaging. Keywords: Cellulose nanocrystals; Smart-nanostructures; Self-assembly; Bio-Photonics; Layered composite

Oral (S18-395, Time: Tuesday 14:50, Room: Bodensee)

Sub-micrometer scale cross-sectional analyses of the polymer flow modifier effects on metal-polymer injection molded direct joining

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Joining light metals and polymers is a tendency in the transport industry to reduce CO2 emissions and costs. Injection molded direct joining (IMDJ) is one type of high-speed metal-plastic direct joining methods, which first forms small textures on the metal surface and then injects melted polymer onto the surface via injection molding, leading to the strong joints due mainly to the mechanical interlocking. We perform microblasting to form micro-scale textures and then use hot water treatment (HWT) to form nano-scale textures on the blasted structure as a metal pretreatment method. This pretreatment method is suitable for an IMDJ mass production because of the low cost and low environmental load. The method indicates relatively good performance (20 MPa) with the aluminum/PA6 joint. However, further joining performance improvement is needed. This research aims to develop a method to improve joining strength effectively and clarify the factors for the improvement. Higher polymer fluidity can yield a better mechanical interlocking effect at the joint, which increases joining performance. The additive of flow modifier can achieve the fluidity improvement effectively by release intermolecular bonds. We here mix an additive (flow modifier OSGOL FM-11) into PA6 (polycaprolactam) to raise the fluidity of the polymer. We manufacture single lap joints by IMDJ and found that the best joining strength increased up to 35 MPa with 10wt% additives. To clarify the mechanisms of joining performance increasing and joining phenomenon, we then studied the infiltration ratio difference by TEM cross-section observation and chemical interaction status difference by AFM-IR analyzation, which can detect the IR spectra of sub-micrometer scale area. The results showed the better polymer infiltration and possibility of chemical interaction occurrence in the sample with additive, leading to a more robust joint. The results contribute to the understanding of hybrid joining phenomena.

Oral (S18-513, Time: Tuesday 15:10, Room: Bodensee)

Determination of Hansen Solubility Parameters using Inverse Gas Chromatography

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Inverse Gas Chromatography (iGC) is a commonly used technique to characterize solid surfaces. Different physicochemical properties of solid materials that can be determined with iGC including surface energy, Lewis acid and base constants and therefore the specific pair interaction parameter (Ka, Kb, Isp), solubility parameter (δT, δD, δP, δH), glass transition temperature (Tg), Work of Cohesion and Adhesion (Wcoh, Wadh) and so on. The importance of surface energies is well-known and proved by many scientific papers in the case of composites. The Surface Energy Analyzer (SEA) is the new generation of inverse gas chromatography instrumentation, which would allow all the mentioned properties to be measured directly and automatically with its unique injection system and software. Solubility parameters are directly related to the cohesive energy density of a material. The latter is a measure of the strength of interaction between molecules or atoms, therefore, solubility parameters are related to stability and other physical properties. Inverse Gas Chromatography provides a fast and reliable route for the measurement of these parameters. This paper describes the determination of the Hildebrandt solubility parameter of PMMA and investigates the influence of different types of Chromosorb, which is used as a carrier for the coated polymer.

Oral (S18-636, Time: Tuesday 16:00, Room: Bodensee)

Cold Plasma Treatment of Glass Fiber/Epoxy Composites to Enhance Surface Free Energy for Adhesive Joint Applications: A Central Composite Design of Experiment

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Adhesive joints are extensively used in wind turbine blades and their failure has become one of the leading causes of the blade’s malfunction. Since the composite/adhesive interface is critical for load transfer in bonded joints, maximizing the bonded joint's load-bearing capacity with the surface's enhancement is necessary. While plastics and composite materials are difficult to finish, some treatment methods such as the plasma technique have shown promise in preparing a contaminant-free surface with higher surface energy. This study aims to evaluate the evolution of surface characteristics of glass-fiber/epoxy composite in response to cold plasma treatment. A Central Composite Design (CCD) of experiment was designed and implemented to quantify the impact of three main factors, i.e., flow rate, time, and power on the surface contact angle and the surface free energy. The CCD was employed to study the multivariable system with linear and nonlinear main effects as well as the potential interactions between variables. The contact angle was measured using two liquids, i.e., glycerol and water. The surface energy was calculated using the OWRK/Fowkes method which applies the geometric mean to combine polar and dispersive components. The results revealed that overall plasma treatment increased the surface free energy and its polar component, which should provide better interfacial adhesion. Moreover, all the main effects and the two-factor interactions were found to be very significant and strong nonlinear relations were observed between the main effects and the response. The DOE also suggested optimal values for all flow rates, time, and power to maximize the surface free energy.

Oral (S18-654, Time: Tuesday 16:20, Room: Bodensee)

Studies of Surface Tensions of Molten Polymers: Influence of the surface rheological properties

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Surface tensions (γ) of polydimethylsiloxane (PDMS) from high-MW systems to oligomers were performed by mean of the pendant drop method at different temperatures. It was found that the surface tension increased with the molecular weight (i.e. the bulk viscosity) and decreased with the temperature with trend dependence in agreement with Sauer and Dee results [1]. In addition, the curves $\gamma(\eta,T)$ have been superimposed to give a master curve allowing the prediction of the surface tension ($\gamma(T)$) for ultra-high molecular weight (i.e. high viscosity) molten PDMS systems. On the other hand, the surface shear viscosities (ηs) and elongational relaxation times (τ) of different PDMS fluids have been measured using a new homemade biconical interfacial setup and using the pulse rising drop method respectively [2-4]. It was found that both $\eta s(T)$ and $\tau(T)$ exhibited a linear correlation with the melt viscosity and that the contributions due to polymer chains transport/mobility at the polymer/ air surface are not negligible. This is in disagreement with the Cahn-Hilliard density gradient theories, which takes into consideration only bulk liquid properties [5]. The experimental methodologies developed in the present study represent a major interest in the measurement and modeling of the surface tension of liquids and can be transposed to other molten amorphous or semi-crystalline polymer systems. References [1] B. B. Sauer and G. T. Dee, Macromolecules, vol. 24, no. 8, pp. 2124-2126, 1991. [2] Y. El Omari, M. Yousfi, J. Duchet-Rumeau, and A. Maazouz, Polymers, vol. 14, no. 14, p. 2844, 2022. [3] Y. El Omari, M. Yousfi, J. Duchet-Rumeau, and A. Maazouz, Rheologica Acta, vol. 61, no. 8, pp. 613-636, 2022. [4] Y. El Omari, M. Yousfi, J. Duchet-Rumeau, and A. Maazouz, Polymer Testing, vol. 101, p. 107280, 2021. [5] G. T. Dee and B. B. Sauer, Journal of colloid and interface science, vol. 152, no. 1, pp. 85-103, 1992.

Poster (S18-584, Time: Thursday 17:00, Room: Foyer)

Novel Triarylamine-Based Intrinsic Microporous Polyamides for Electrochromic Supercapacitor: Diffusion Dynamics and Charge-Discharge Studies

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To investigate the counter ions diffusivity of polymers with intrinsic microporous structures during the electrochemical process, Tröger's base (TB), with the non-planar and V-shaped moiety, was introduced to facilitate the formation of diffusion channels. The TB-incorporated polyamides (TPPA-TB and TPPA-Me-TB) revealed enhancing electrochromic properties due to the higher diffusivity, which could effectively narrow down the electrochemical redox potential difference (ΔE), resulting in the higher switching response speed (ʋ) while maintaining similar transmittance change (ΔT). Intriguingly, the triarylamine-based polyamide TPPA-Me-TB demonstrated both excellent charge-discharge ability with a high specific capacitance (Csp) of 165.3 F/g at 1.0 A/g and distinct two-stage color changes from colorless to green (0.8 V) and to blue (1.2 V), that could monitor the content of charge capacity. Therefore, these novel redox-active polyamides should be attractive and suitable for electrochromic supercapacitor (ECS) materials.

Poster (S18-704, Time: Thursday 17:00, Room: Foyer)

Treatment of flax fibers by cold plasma

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The main challenge regarding the development of biocomposite materials, i.e. biodegradable polymer matrix plus plant-fiber fillers, is to work out a 'real' eco-friendly way of manufacturing in order to be considered as fully green materials. Fiber pretreatments can be done using various surface modification techniques [1]. To avoid using polluting chemical treatment processes, we choose to experiment and evaluate a cold plasma treatment process applied to natural fibers. Thereby, surface modification of flax fibers in a cold plasma reactor working in a pressure range of 0.1-1 bar was studied by following a similar approach to other related fields [2]. In this first work, process gas composition, plasma density and energy characteristics of the plasma were the main variables of the study. IR spectroscopy was used to characterized the flax fibers surface. A differential analysis of the surfaces after plasma treatment allows to draw some preliminary hypotheses on the nature and mechanisms of the induced modifications. Our approach was comforted by the recent reviews done by Gupta et al. (2021) [3] and Alonso-Montemayor et al. (2022) [4], in which the effects of different plasma treatments on the fiber surface were enough promising, going up to overall mechanical characteristics modification, to justify even more advanced research. [1] Baltazar-y-Jimenez A., Bistritz M., Schulz E., Bismarck A. Doi: 10.1016/j.compscitech.2007.04.028 [2] Rohani V., Bauville G., Lacour B., Puech V., Duminica F.D., Silberberg E., Doi: 10.1016/j.surfcoat.2008.06.135 [3] Gupta U.S., Dhamarikar M., Dharkar A., Chaturvedi S., Kumrawat A., Giri N., Tiwari S., namdeo R. Doi: 10.1016/j.matpr.2020.11.973 [4] Alonso-Montemayor F.J., Navarro-Rodriguez D., Delgado-Aguilar M., Neira-Velazquez M.G., Aguilar C.N., Castaneda-Facio A.O., Reyes-Acosta Y.D., Narro-Cespedes R.I. Doi: 10.1007/s10570-021-04361-0 Acknowledgments: ANR Carnot MINES Paris PSL, project entitled "RecCombios: Recyclabilité de Composites Biosourcés - Recyclability of Biobased Composites") 2022

Plenary (S19-802, Time: Wednesday 09:10, Room: Sulzer Chemtech)

Micro- and nanoplastics: sources and release

Nowack Bernd[1]

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The presence of micro- and nanoplastics in all environmental compartments brings up the question where these materials actually come from: how much is released in the micro- or nanosize and which part has been produced by fragmentation/degradation in the environment? Detailed knowledge about polymer flows into the environment is essential for a better management of plastic pollution. Currently, release estimates are often based on simple assessments, focusing mostly on only a selected number of products and are generic for large regions. In addition, only limited knowledge about specific polymer flows is available although it is well known that different polymers are used in very different applications and may therefore have very different release potentials.

This presentation will show how detailed material flow analysis models can be used to get mass flow information for 12 different polymers and rubber. As plastic releases are not evenly distributed across space, a spatially resolved release model is needed. Such models can also be used as input to environmental fate models that describe the further behavior of plastics in the environment.

This presentation will also show data on release of both micro- and nanoplastics from textiles which constitute one of the main sources of microplastics to the environment. Release can occur during washing, abrasion and after exposure to UV light and the different types of fibers that can be formed will be discussed. It will also be shown how technical processes during manufacturing are responsible for microplastic fiber formation. As nanoplastics have come into focus recently, this presentation will also shed some light on their release from textiles and the analytical issue complicating their identification.

Keynote (S19-495, Time: Wednesday 10:15, Room: Sulzer Chemtech)

Considerations and implications for regulations of intentionally added microplastics in the EU

Mitrano Denise M.[1], Amberg Stefano[1]

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Plastic pollution in the environment has been rapidly increasing for decades and the environmental impacts can be related to material size where macro-, micro- (MPs, < 5mm) and nano- (< 1mm) plastics have different fate, transport and biological interactions. Most MPs originate from the degradation of plastic litter or release from normal use of polymer-containing materials (i.e., secondary MPs), but MPs are also intentionally added in a number of products (i.e., primary MPs). While some reduction of secondary MPs can be targeted through improved waste management policies, more targeted measures can be used that focus specifically on intentionally added MPs. The European Chemicals Agency's proposed regulation of all primary MPs under REACH considers the wide variety of uses, physical and chemical properties, and applications collectively. The challenges and implications of these restrictions will be discussed. Alternatively, implementing the Essential Use Concept may help to balance environmental health, technical feasibility and innovation of new materials better than the currently proposed approach. Here we present a systematic framework of how MPs could be categorized and regulated based on their use. In some cases, substitutions are both politically and technically feasible. In other cases, substitutions may come with more uncertainty such as significant performance questions and monetary costs. A holistic and transdisciplinary alternatives assessment is therefore needed that considers MPs risks and benefits in particular use cases as well as the existence, quality, costs, and hazards of replacement materials. Regulations need a precise focus and must be enforceable by measurements. Policy must carefully evaluate under which contexts MPs use may be warranted and where incentives to replace certain MPs can stimulate innovation of new, more competitive and environmentally conscious materials.

Keynote (S19-640, Time: Wednesday 13:45, Room: Sulzer Chemtech)

Sustainable alternatives to single-use plastics and circular economy

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“Plastic waste” is a global pollution problem that pops up daily on news headlines in our changing world with microplastics (>5 mm in size) even showing up in Mount Everest’s snow. ”New Plastic Economy” by Ellen MacArthur’s Foundation targets ”100% reusable, recyclable or compostable plastic packaging by 2025”. Plastics find applications in many sectors including but not limited to food packaging, transportation, medical, housing, and other high-end products. Packaging is the major sector in plastic use; ~50% of which is in single-use products like grocery bags to takeout containers, straws, six pack rings and plastic cutlery. Plastic wastes account to ~300 million tons annually, which is equivalent to the weight of the entire human population (United Nations). Thus, immediate and vigorous actions are required in reducing plastic wastes. There remains a huge challenge in alternative solutions for three categories of packaging – multi-layer packaging, mixed-material (plastic, metal, and paper) and mono-material packaging that are impractical to recycle. Biodegradable plastics show leading trends in sustainable packaging. Many misunderstandings, myths and confusions around bioplastic pose a challenge in its commercialization. The biodegradable plastic market expects to grow to ~$ 5 billion market in 2025. The end-of-life solution for biodegradable plastics should grow and should be expanded through government and industry leaders as well as policy makers. Special interest is growing around home compostable and soil biodegradable products over industrial composting. Disruptive technology in creating high barrier biodegradable packaging can help in reducing food wastes. This presentation will highlight on challenges and opportunities towards a zero-waste plastics economy in supporting circular economy. The combination of circular economy with bioeconomy can lead to advanced sustainability.

Oral (S19-038, Time: Wednesday 10:40, Room: Sulzer Chemtech)

Primary and secondary polyolefin-related microplastics: A lifecycle perspective

Gahleitner Markus[1], Eder Gabriele[2], Huber Miriam[3], Pomakhina Elena[1], Archodoulaki Vasiliki-Maria[3], Washüttl Michael[2], Zinöcker Erich[1]

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The issue of microplastics has been growing in scientific discourse and public perception massively in the last 5 to 10 years. As polyolefins, mostly polyethylene (PE) and polypropylene (PP), make up more than 50% of the global plastics production, the related industry also needs to take a close look at this subject. In order to get a complete perspective on sources, presence, consequences and final fate of those particles, Borealis is engaging at various levels, supporting collaborative projects and doing own research. In terms of microplastic sources, the whole product lifecycle from primary production through conversion and application to recycling must be considered, not ignoring the regrettable fact of littering and waste dispersion from landfills. Reliable detection and identification are equally relevant along this path as mechanisms leading to breakup into secondary microplastics, but possibly also their decomposition. Examples for activities at several stages will be shown.

Oral (S19-199, Time: Wednesday 11:00, Room: Sulzer Chemtech)

From Macro- to Microplastic: Determining Degradation Rates and Microplastic Release of Commodity Polymers

Ruckdäschel Holger[1], Menzel Teresa[1], Meides Nora[1], Mauel Anika[1], Senker Jürgen[1], Strohriegl Peter[1]

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When polymers enter the environment, molecular degradation at the surface is triggered by various environmental factors, such as sunlight, water, and temperature. The gradual decomposition eventually degrades macroscopic plastic parts into smaller pieces called secondary microplastics (MP). Althoughstrategies have been developed to reduce the release of primary MP already produced in µm-size, the formation of secondary MP by macroscopic plastic debris is inevitable. Therefore, a thorough knowledge of the degradation mechanisms and rates is urgently needed to develop future abatement strategies. In this work, we exposed macroscopic amorphous polystyrene (PS), semi-crystalline polyethylene (PE) and polypropylene with additives to accelerated weathering in a long-term experiment [1,2]. We analyzed the depth-dependent degradation pathways and identified the driving factors for embrittlement based on polymer structure. Comparison with natural weathering experiments confirmed our results and also allowed us to determine an acceleration factor for the degradation of PS, PE and PP in natural environments. By combining the quantitative data on the formation of MP and the acceleration factor, we calculated the specific surface degradation rates for all polymer types. Finally, an extrapolation answers the question of the residence time of macroplastic waste based on component geometry before it is completely degraded to MP. Our results cover the pathway from macro- to microplastics and give first indications of possible avoidance strategies for the formation of secondary MP by commodity polymers. References [1] Meides, N., Menzel, T., Pötzschner, B., Löder, M.G.J., Mansfeld, U., Strohriegl, P., Altstädt, V., and Senker, J., Environmental Science and Technology, 55(12), 7930-7938 (2021) [2] Menzel, T., Meides N., Mauel, A., Mansfeld, U., Kretschmer, W., Kuhn, M., Herzig, E.M., Altstädt, V., Strohriegl, P., Senker, J., Ruckdäschel, H. Science of The Total Environment (2022)

Oral (S19-351, Time: Wednesday 11:20, Room: Sulzer Chemtech)

Elaboration of weathered model microparticles of polyethylene suited for transport study, characterized in Raman, FTIR and NMR spectroscopies.

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In the environment, macro and micro-size plastic waste undergo climatic ageing, which can lead to oxidations (formation of carbonyl functions, etc.), modifications of their surface hydrophilicity, as well as chain scissions modifying their chemical structures. This ageing and weathering can affect their thermal properties (glass transition temperature, enthalpies and temperatures of crystallization and melting) as well as their mechanical properties such as Young's modulus, threshold, breaking stress and elongation at break. As a consequence, those polymer wastes can be considered rather different from their initial polymer state. In particular, the evolutions associated with weathering can be characterized by techniques such as Fourier Transform Infrared, Raman, Nuclear Magnetic Resonance spectroscopies and microscopies. Our work aims at elaborating model microparticles of polyethylene, having undergone UV ageing for several exposition conditions and mechanical abrasion, characteristic of urban stimuli. With the objective of : i) having reference data in Raman, FTIR, and NMR spectroscopies aiming at refining the interpretation of characterization results of micro-particles from natural environments, ii) designing model microparticles, representative of those present in natural media, for laboratory transport studies.

Oral (S19-377, Time: Wednesday 11:40, Room: Sulzer Chemtech)

Microplastics pollution caused by the face masks, their evaluation and associated product safety

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The Covid-19 pandemic has added a significant burden to the environment as most of the personal protective equipment (PPE) like face masks, surgical gowns, etc. are discarded after single use. Major constituents of PPE are the synthetic polymers like polypropylene, polyester and polyethylene. With such a high volume of PPE disposed of created additional problems of microplastics released to the environment. These microplastics are a danger to the environment and can end up contaminating resources. A microplastic can be defined as a plastic polymer with a size of 1-5μm. In addition to microplastics, masks also release toxic chemicals like formaldehyde which are used during finishing of masks. The release of microplastics from face masks was investigated in three commonly used masks, namely cloth, surgical and respiratory N95. The Gelbo flex lint tester with particle counter was used to measure the release of microplastics in dry state while compressing and twisting the masks. Since masks are worn next to the skin, another aim to determine product safety, by evaluating release formaldehyde and pH was evaluated as per the OEKO-TEX standard 100 for PPE materials. Fragment particle sizes in the range of 1-25μm were detected in surgical and respiratory masks with major contributing particles within the 1-5μm range. The number of particles in the 1μm, 3μm, and 5μm for surgical masks were 173837, 75512 and 25059. In the case of respiratory N95 masks, these values were 120678, 42190 and 15069. The number of microplastics released by surgical masks was higher than that of respiratory N95 masks. This could be attributed to the release of loose fibres while flexing action as well as somewhat porous structures of the surgical masks. The pH of masks was found to be within the recommended limit of 4-7.5. For the formaldehyde test, nothing was detected in all the masks. The results confirmed the release of microplastics and product safety are within the specified limit.

Oral (S19-446, Time: Wednesday 12:00, Room: Sulzer Chemtech)

Coupled techniques: the best tool for the identification of microplastics

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[1]AIMPLAS, Paterna, Spain

It is estimated that approximately 5% of plastic production ends up in the environment and part of it does so in the form of micro and nanoplastic (MNPs). The main problem is that there is still no legislation. This is largely due to the difficulty in a standard methodology for this analysis and identification. In AIMPLAS, we have adequate methodologies for the analysis of MNPs depending on the type of substrate, sludge, soil, biota, drinking water etc. Coupled techniques are the best tool for the identification of MNPs and increasingly used. In this work we present different practical cases using pyrolysis gas chromatography mass spectrometry (Pyr-GC/MS), µ-FTIR and SEM-EDX for the identification of MNPs in sludge sample of a wastewater treatment plant, water of an effluent and biota. Using these coupled techniques, we can cover the identification depending on the information that is desired to obtain: • µ-FTIR: allows visualizing the morphology, size, color, and identification of MNP > 10 µm. • SEM-EDX: allows to visualize the morphology, size, and elemental identification of particles > 10 µm • Pyr-GC/MS: quantitative and qualitative identification of MNP > 1 µm It is important to combine different techniques and analytical procedures in order to cover a wide range of chemical formulations, sizes, types and morphologies of MNPs, ensuring the quality of the analysis. We resort to coupled techniques that allow us to obtain the maximum possible information employing the minimum resources.

Oral (S19-522, Time: Wednesday 14:10, Room: Sulzer Chemtech)

Biofilm-influenced weathering of polypropylene films in various aqueous environments

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As concerns of plastic pollution rise, threatening global water sources, there has been an influx of microplastic research focused on their fate and effects, in hopes of remediation. Polymers in aquatic ecosystems are susceptible to degradation by natural weathering where abiotic factors, such as salinity or pH, and biotic factors, such as microbes, induce changes to the physiochemical properties of the materials. The extent of plastic weathering is dependent both on the material and the location (i.e., body of water and geographic location) though few studies have quantitatively considered the joint effects of abiotic and biotic factors present in different environments on a single plastic material. This study utilized microscopy, Fourier-transform infrared spectroscopy (FTIR), and goniometry to evaluate biofilm growth through morphological and chemical changes on polypropylene (PP) incubated in filtered water, lake water, and ocean water. Biofilm growth was confirmed on all samples however the most notable changes occurred to films immersed in ocean water. After removal of biofilm, PP degradation was also confirmed. Consequently, salinity was investigated under controlled conditions but deemed not solely responsible for the degradation due to a lack of measurable difference after immersion in various saline solutions. Finally, RNA analysis on samples immersed in ocean water revealed high abundances of Ralstonia species, followed by a shift to a Zhongshania dominated community. This work suggests that biofilm-influenced degradation may accelerate polyolefin aging in the environment, especially in the presence of ocean microbial consortia.

Oral (S19-547, Time: Wednesday 14:30, Room: Sulzer Chemtech)

Shape as a controlling factor in weathering of PET as a pre-cursor to microplastic formation

Pinlova Barbora[1], Borzi Aurelio[2], Nowack Bernd[1]

[1]Empa, St. Gallen, Switzerland
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Plastic debris is exposed to a range of factors in the environment that lead to weathering of the material. Weathering results in changes in the material and is considered a precursor to fragmentation and microplastic formation. However, the understanding of the mechanism of the chain of these events is limited, including the effect of the form of the plastic. Therefore, a comparative study of the weathering process of polyethylene terephthalate (PET) was performed. PET in the forms of fibers, films, and pellets was exposed to UV light for three months. Moreover, the effect of hydrolysis was studied by sub-merging half of the samples in water. The progress of weathering was investigated with scanning electron microscopy every 15 days. Additional analysis was performed on selected samples by Raman spectroscopy, x-ray diffraction analysis, and gel permeation chromatography. It was concluded that the weathering conditions as well as the specific properties of the plastics influence the development of the features related to weathering. It was shown that the combination of photo-oxidation and hydrolysis quickened the process. The samples exposed to UV light while submerged in water started displaying signs of damage after 15 or 30 days of the weathering. While pellets immediately started to crack, films and fibers were first covered with pits, which then transformed into cracks. Each PET form showed a unique crack pattern, likely related to the production process. Moreover, the crack pattern was recognized to be a template to the surface layer ablation which was observed when additional mechanical stress was applied. Therefore, the size and quantity of the fragments could be estimated. It was suggested that 1.4-7.9 million microplastic fragments can be released from one cm2 of a weathered PET film. Moreover, additional particles were seen emerging on the surfaces of the weathered samples that could be an additional source of microplastics produced during weathering

Poster (S19-259, Time: Thursday 17:00, Room: Foyer)

Effects of nanoplastic trophic transfer in food chain with three marine organisms

An Youn-Joo[1], Kim Lia[1]

[1]Konkuk University, Seoul, Korea

Plastic wastes is one of the environmental issue and marine ecosystem is especially high contaminated by these small debris. Microplastics continuously into marine ecosystem and living organisms have some threat to be affected by these kinds of microplastics. Considering the real transfer of microplastics, small sized particles could be transferred from low to high trophic level organisms; this study investigated the effects of microplastic exposure through marine trophic transfer from the microalgae or zooplankton to fish. The polystyrene sphere was initially adsorbed to the cell wall of microalgae (Dunaliella salina) and rolled as the food of Artemia franciscana. The fish was fed A. franciscana as food that microplastics were eventually transferred to the most high-level organism, Larimichthys polyactis. Through the trophic transfer of microplastics, L. polyactis was exposed to the microplastics found in their gut, disturbed on its swimming ability or digestive ability by measuring α-amylase activity. Fish are consumed as the human food resource and the results of this study indicate that human could be eventually exposed to microplastics by consuming the food resource.

Poster (S19-707, Time: Thursday 17:00, Room: Foyer)

Textile microplastics: perspectives for changes in the current scenario

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[1]University of São Paulo, São Paulo, Brazil

In environmental plastic pollution, for more than a decade, domestic washings have been recognized, as an activity that promotes textile fragments. More studied than other sources, they are annually responsible for the emission of significative amounts of fibers in water bodies. Due to its ecologic and socioeconomic consequences, beyond academy, they receive attention from both, the public and private sectors. In this way, to deal with its outcomes, several factors and actors take place. Here, main strategies rely on washing machine devices, equipment choices and modes of operation, wastewater treatment infrastructure and textile parameters. About the latter, there is a higher potential of action as it is on the base of this type of pollution. Thus, product development and manufactures represent the utmost turning point. In this scenario, this study analyses and reunites the main issues related to fibers emission, especially the synthetics ones. Starting from a textile perspective, based on literature review, it purposes interventions for mitigating microplastic and other particles release. It also highlights knowledge gaps to address in future studies. Overall, it intends to amplify the textile perspective, promoting next steps to modify actual pollution taxes. These analyses to be complemented with simultaneous actions, from involved sectors.

Keynote (S20-241, Time: Tuesday 12:00, Room: Sulzer Chemtech)

Polar polymers in actuators, sensors, and generators

Opris Dorina M.[1], Sheima Yauhen[1], von Szczepanski Johannes[1], Danner Patrick M.[1], Caspari Philip[1], Wolf Jana[1], Adeli Yeerlan[1], Owusu Francis[1], Nüesch Frank[1]

[1]Empa, Dübendorf, Switzerland

High dielectric permittivity polymers are an important material class that may be applied in actuators, sensors, and energy harvesting and storage. Their glass transition temperature (Tg) can be tuned at will by the type and content of the polar groups used and the polymer backbone on which they are grafted. Proper cross-linking of the low Tg polymers allowed us to achieve a novel class of polar elastomers with unique dielectric properties. In contrast, poled high Tg polymer nanoparticles in an elastic network allow for achieving piezoelectric elastomers. These novel materials are used as dielectrics in elastic capacitors, which can serve as actuators, sensors, or generators. This presentation will show the synthesis of polar polymers, their processing into thin elastic films, and their use in different devices.

Keynote (S20-381, Time: Tuesday 17:00, Room: Sulzer Chemtech)

Locally Programmable Multi-stimuli Dually-responsive Intelligent Woven Structures

Kamkar Milad[1], Panahi-Sarma Mahyar[2], Xiao Xueliang[2]

[1]University of Waterloo, Waterloo, Canada
[2]Jiangnan University, Wuxi, China

Using metallic composite yarns and polymeric/thermochromic microcapsules composite fibers, processed via a melt-spinning-technique, an electrothermally multi-responsive fabric is woven. The resulting smart-fabric transfers from a predefined structure to an original shape while changing color upon heating or applying an electric field, making it versatile for different applications. The shape-memory and color-changing features of the fabric can be precisely controlled by rationally controlling the micro-scale design of the individual fibers in the structure. Thus, the fibers' microstructural features are optimized to achieve color-changing behavior along with shape fixity and recovery ratios of 99.95% and 79.2%, respectively. More importantly, the dual-response of the fabric by electric field can be achieved by a low voltage of 5 V, which is far smaller than the previously reported values. Above and beyond, the fabric is able to be meticulously activated by selectively applying a controlled voltage to any part of the fabric. The precise local responsiveness can be bestowed upon the fabric by readily controlling its macro-scale design. A biomimetic dragonfly with the shape-color dual-response ability is successfully fabricated, broadening the design and fabrication horizon of smart groundbreaking materials with multiple-functions.

Keynote (S20-675, Time: Tuesday 13:45, Room: Sulzer Chemtech)

Bio-Inspired Artificial Muscle-Tendon Complex with Proprioception Made of Liquid Crystal Elastomer and Room-Temperature Liquid-Metal Soft Sensors

Park Yong-Lae[1], Cho Jiyeon[1], Lee Minhee[1], Wang Yang[2], Cai Shengqiang[2]

[1]Seoul National University, Seoul, South Korea
[2]University of California San Diego, California, USA

Muscle-tendon complex (MTC), a collection of muscle and tendon fibers, is a representative actuating mechanism of biological system. The muscle spindle and Golgi tendon organ (GTO), proprioceptors of the MTC, transduce information on the changes in length and tensile load of the muscle, respectively, enabling low-level feedback control at central nervous system. This concept of integrated sensing can be applied to development of artificial muscles. Liquid crystal elastomer (LCE), a special type of polymer artificial muscles, is a stimulus-responsive material with anisotropic mesogens. When the temperature increases above its transition point, the orientation of the mesogens changes from an aligned state (nematic state) to a disordered state (isotropic state) which in turn causes prominent deformation. In order to actuate LCE using electrical power, we printed a liquid metal (LM) pattern on an LCE substrate as a performer of joule heating, which has high conformability to complex movement of soft materials. Since the electrical resistance of the LM changes as the LCE contracts and extends, the heater can also be used as a strain sensor, similar to a muscle spindle. However, due to the characteristics of the LCE material, such as high viscoelasticity and soft elasticity, the stress-strain relationship of LCE is nonlinear and time-dependent, showing a property like slow stress relaxation. This material complexity makes it difficult to reflect the accurate stress state of the specimen although we have the strain information. In order to solve this problem, we introduce fixed isotropic LCE tendons connected in series with the contractile component. Since the isotropic LCE shows a higher performance in terms of elasticity, we can use it for detecting the applied load with a LM circuit directly printed on it, similar to GTO. In conclusion, the proposed LCE MTC has a capability of self-sensing and self-heating for contraction, enhancing the performance of feedback control.

Keynote (S20-696, Time: Tuesday 15:10, Room: Sulzer Chemtech)

Landing on A Wall with Morphologically Adaptive Soft Robots

Jusufi Ardian[1]

[1]Empa, Dübendorf, Switzerland

Perching on vertical surfaces could be a critical ability for multi-modal robots since it facilitates station-keeping, i.e., to hold a stable position above the ground, without the need for continuous hovering, hence lowering the energy consumption. However, landing on vertical substrates is challenging, especially in the absence of thrust generation capability. Dynamic landing on vertical surfaces in challenging environments is a critical ability for multi-modal robots. Based on original discovery of the geckos perching on a rainforest tree (Siddall, Byrnes, Full, and Jusufi 2021), and the development of an active robotic tail, we continue to decipher materials and mechanically mediated landing involving the Fall Arresting Response using soft bodied physical models. Here we measure the landing performance as a function of varying body and appendage stiffnesses. The capability associated with a Materials-mediated Crash-Landing on a wall is derived from the careful study of the unique landing manoeuvre of the Asian flat-tailed geckos (Hemidactylus platyurus) are observed to glide and perch on vertical surfaces by relying on their tail and body morphology, potentially reducing the control effort to perch. This novel perching mechanism using a bioinspired physical model is discussed and its tail and body parameters to determine their influence on perching success and the kinematics of the gecko's dynamic landing maneuver are adjusted. Perching performance is evaluated by changing the model's torso and tail stiffness. Combining a compliant torso and stiff tail enables the model to passively perch on a vertical substrate with a success rate > 90 %, compared with ≈ 10 % without a tail attached. A compliant torso is necessary to absorb the in-flight kinetic energy and accommodate the uncertainties in approach conditions (Chellapurath, Khandelwal, Rottier, Schwab and Jusufi 2022). Similar to the geckos' perching strategy, the stiff tail pushes against the substrate, preventing the model from falling backward head over heels. These findings highlight the critical role of tail and back material stiffness for perching and a robust mechanism for soft robotic lander

Keynote (S20-699, Time: Tuesday 10:15, Room: Sulzer Chemtech)

Embodiment Design of Autonomous Material Systems

Shepherd Robert F.[1]

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I will talk about what makes a material autonomous, and how to evaluate levels of autonomy within autonomous material systems. Further, I will describe what should be possible now, and what will be possible if additive manufacturing techniques progress. Finally, I will discuss examples of autonomous material systems, with particular focus on embodiment of sensing, actuation, computation, and energy.

Oral (S20-137, Time: Tuesday 10:40, Room: Sulzer Chemtech)

Synthetic growth: a new way to grow a soft robot

Hausladen Matthew M.[1], Zhao Boran[1], Kubala Matthew S.[1], Francis Lorraine F.[1], Kowalewski Timothy M.[1], Ellison Christopher J.[1]

[1]University of Minnesota, Minnesota, USA

Endowing soft robots with the ability to be grown, instead of simply manufactured, can enable more life-like qualities in soft robotic systems. We present a novel synthetic growth method inspired by the growth of plants and fungi, extrusion by self-lubricated interface photopolymerization (E-SLIP) [1]. In this method, profiled photopolymer parts are processed continuously through simultaneous pressure-driven flow and photopolymerization of liquid thiol-ene monomers. Through the incorporation of poly(dimethylsiloxane)-block-poly(ethylene oxide) (PDMS-PEO) copolymer surfactant in the monomer solution, a lubricant layer is formed in situ, permitting extrusion at low pressures. Using E-SLIP, high-aspect ratio parts with a variety of profile geometries and tunable mechanical properties can be fabricated. By employing E-SLIP as the synthetic growth method, growing soft robots are capable of lengthening by thousands of percent (>2,700% demonstrated) and at rapid growing speeds (up to 12 cm/min). We present the fundamental parameters that govern the growth of the robot and define the operating space that permits successful growth. The growing soft robot parallels capabilities of plants and fungi, such as exploration of constrained environments, burrowing, and navigation of tortuous paths. These results establish E-SLIP as a potential platform for on-demand manufacturing, exploration, and sensing in a variety of environments. 1. M.M. Hausladen, B. Zhao, M.S. Kubala, T.F. Kowalewski, L.F. Francis, C.J. Ellison. "Synthetic growth by self-lubricated photopolymerization and extrusion inspired by plants and fungi." Proceedings of the National Academy of Sciences, 119, 33 (2022).

Oral (S20-214, Time: Tuesday 11:00, Room: Sulzer Chemtech)

Sim-to-Real Transfer of Soft Robotic Navigation Strategies That Learns from Visual Perception

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[1]CUHK, HK, China

To steer a redundant soft robot precisely in an unconstructed environment with minimal collision remains an open challenge. When the environments are unknown, prior motion planning for navigation may not be available in simulation or operation. This work presents a novel Sim-to-Real method to guide a cable-driven 3D-printed soft robot in a static environment under the Simulation Open Framework Architecture (SOFA). The scenario aims to resemble one of the critical steps during a simplified transoral tracheal intubation process where a robotic endotracheal tube is guided to the upper trachea-larynx location by a flexible video-assisted endoscope/stylet. In this work, a 6.2-mm-diameter steerable soft-bodied robot is presented to work as a stylet. In SOFA, we first describe the robot morphologically with its soft-rigid material properties, detailed geometry, and actuation mechanics. Next, we employ the quadratic programming inverse solver to obtain collision-free motion strategies for the endoscope/stylet manipulation based on the robot model and encode the virtual eye-in-hand vision. Then, we associate the anatomical features recognized by the virtual vision and the joint space motion using a closed-loop nonlinear autoregressive exogenous model (NARX) network. Afterward, we transfer the learned knowledge to the robot prototype, expecting it to navigate to the desired spot in a new phantom environment automatically based on its eye-in-hand vision only. Experiment results indicate that our soft robot can efficaciously navigate through the unstructured phantom to the desired spot with minimal collision motion according to what it has learned from the virtual environment. The results show that the average R-squared coefficient between the closed-loop NARX-forecasted and SOFA-referenced robot's cable and prismatic joint space motion are 0.963 and 0.997, respectively. The eye-in-hand visions demonstrate satisfactory alignment between the robot tip and the glottis.

Oral (S20-316, Time: Tuesday 11:20, Room: Sulzer Chemtech)

Biomimetic multilayer e-skin with mechanoreceptive and thermoreceptive sensory capabilties

Georgopoulou Antonia[1], Hardman David[2], Thuruthel Thomas George[2], Iida Fumiya[2], Clemens Frank[1]

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[2]University of Cambridge, Cambridgeshire, United Kingdom

In natural organisms, tactile sensing enabled by the skin involves a combination of mechano- and thermoreceptive neurons. Mechano- and thermoreceptive sensing of the skin can be mimicked using flexible thermoplastic elastomers with a conductive filler, like carbon black. In this study, thermoplastic material extrusion additive manufacturing (MEX-AM) was used to fabricate a multi-layer skin with integrated mechano- and thermoreceptive sensing elements. Soft sheets with four unidirectional mechano- and thermoreceptive elements were cross-ply laminated to achieve the multi-layer e-skin structure. A robotic manipulator with a heated probe was used for probing the e-skin structure 4'500 times in random locations with varying temperatures and probing depths between 30 and 100 °C, 0 and 4 mm, respectively. Neural network processing was applied to process the sensor output data, successfully. Interestingly, when both outputs from the deformation and temperature sensing elements were combined, the sensory stimulus localization predictions were more accurate. Lateral accuracies of 1.8 mm, depth errors of 0.22 mm, and temperature errors of 8.2 °C were achieved for the multi-layer e-skin structure. Overall, 3D printed e-skin in combination with neural networks will be a key issue for future closed loop control in applications like soft robotics and wearable electronics devices.

Oral (S20-346, Time: Tuesday 11:40, Room: Sulzer Chemtech)

Flexible Z oriented Columnar Piezoelectric Composite Films as Flat-Panel Transparent Loudspeakers: Application and Modeling

Cakmak Mukerrem[1], Grant Jesse[1], Wang Yimin[1], Liu Yangfan[1]

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Flexible Z oriented Columnar Piezoelectric Composite Films as Flat-Panel Transparent Loudspeakers: Application and Modeling Jesse Grant, Yiming Wang, Yangfan Liu, Alex Roth, Rahim Rahimi, Guochenhao Song, Miko Cakmak# Highly anisotropic piezoelectric composites promise a new frontier in electroacoustic devices by combining the advantageous properties of both piezoceramics and polymers. Fundamentally, piezoelectric loudspeakers employ the converse piezoelectric effect to convert electrical to mechanical energy. Quasi-1–3 piezoceramic/polymer composites represent a novel approach to flat-panel loudspeakers that are tunable in modulus, with opportunities for mechanical flexibility, optical transparency, and large-area coverage. This 1-3 composite is formed of "Z" (thickness) oriented columnar morphology that is created by the application of electric field to the photocurable polymer precursor dispersed with piezoelectric relaxor ferroelectric lead magnesium niobate–lead titanate (PMN–PT)micro/nanoparticles by dielectrophoresis. In this study a range of design parameters, including the particle loading, matrix modulus, film thickness, film size, and electrode-material are explored. Material properties critical for dielectrophoresis are characterized and loudspeakers were fabricated based on the optimal processing conditions. Electroacoustic characterization studies included the effect of loudspeaker size, substrate stiffness, the microphone distance, the piezoceramic material, and the matrix modulus. Finally, finite element (FE) modeling of the electromechanical behavior validates the natural frequencies and the modes shapes of the loudspeakers via the analytical solution and the frequency response to electrical and mechanical excitation. Good correspondence between the predicted electroacoustic performance and the experimentally validated model are observed.

Oral (S20-525, Time: Tuesday 14:10, Room: Sulzer Chemtech)

Soft robots with mechanical intelligence via nonlinear networks of inflatable actuators

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[2]KU Leuven, Leuven, Belgium

Recent advances in soft robotics have been made possible by the developments of elastic inflatable actuators (EIAs), due to their simple fabrication process, low costs, large deformations and fast responses. EIAs have the dual function of storing elastic energy and doing mechanical work, exhibiting the desired multifunctional behavior envisioned by the concept of Embodied Energy. However, most of the energy supplied to EIAs is typically stored as elastic energy and then released without performing useful work. A way to partially convert stored energy into useful mechanical work consists of designing elastic inflatable actuators with a nonmonotonic characteristic to form a mechanical instability incorporated in the actuator. When the driving force or an external force triggers the instability, part of the elastic energy stored in the structural elements of the actuators is released to power a fast output motion. This can be used to make soft robots jump or to trigger a change in the internal system of the robot. Moreover, when multiple nonlinear actuators are interconnected they can also embody the control function, by leveraging the local negative stiffness of the actuators to drive their motion out of phase, achieving a mechanical intelligence that allows soft robots to locomote using only a single input. In this work, we report on a soft articulated tetrapod that walks in an underactuated configuration and a system of bending actuators that move with a metachronal pattern inspired by biological cilia using a single pressure input.

Oral (S20-660, Time: Tuesday 14:30, Room: Sulzer Chemtech)

Soft robotic fish platform with a novel control strategy for stiffness control

Schwab Fabian[1], Rezaei Seyedreza[1], Jusufi Ardian[2]

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Robots are excellent at performing complex tasks in controlled situations, but they lack the adaptability and flexibility needed for exploration or search and rescue missions in irregular surroundings. The use of an animal's musculoskeletal system and proprioceptive awareness is one of the most distinctive aspects of animal locomotion. On a fundamental, bio-mechanical level, little is known about the various biological systems that enable animals to exhibit this behavior of seemingly unaffected exploration of unfamiliar environments. With the help of robophysical models, biologists and engineers can work together productively to find answers to those unanswered questions about adaptive motion. These biorobotic platforms can act as "model animals" for biological research to advance our understanding of nature, while also giving mobile robots realistic capabilities for navigation and exploration. A soft robotic fish platform was fabricated and experiments in air and water, with differing undulation frequencies were performed. In order to provide bio-inspired feedback, we combine soft sensors and flexible actuators. Our goal is to replicate not only the animal's locomotion strategies but also their unique material properties. We then combine this with new control techniques. To investigate the effect of co-contraction on body stiffness control, the soft sensory performance of the robotic fish with a new control design known as [m, m]-Padé approximants in implementing repetitive learning controls for a soft robotic subcarangiform fish is investigated. Compared to the previously used PID-Controller, results show improvements in reducing the output tracking error concerning the previous design performance in a more challenging reference signal as the robotic fish motion. This bio-inspired fish tail robotic experimental platform allows for the discovery of the effect of tail stiffness and frequency on thrust generation.

Oral (S20-695, Time: Tuesday 14:50, Room: Sulzer Chemtech)

RoboTwin: a platform to study hydrodynamic interactions in fish schools

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By living and moving in groups, fish gain many benefits, such as heightened predator detection, greater hunting efficiency, more accurate environmental sensing, and energy saving. Although the benefits of hydrodynamic interactions in schooling fish have drawn growing interest in fields such as biology, physics, and engineering, and multiple hypotheses for how such benefits may arise have been proposed, it is still largely unknown which mechanisms fish employ to obtain hydrodynamic benefits, such as in increased thrust, or improved movement efficiency. One main bottleneck has been the difficulty in collecting detailed sensory information, corresponding locomotory responses, and hydrodynamic information from real schooling fish. In this paper, we present the RoboTwin platform designed to aid such data collection: it allows us to replay the 3D movements and body posture kinematics of real fish in fish-like robots, allowing us to measure power cost, thrust, and detailed flow fields. To demonstrate the capabilities of our platform, we conduct experiments with two goldfish (Carassius auratus) swimming in a flow tank, from which 3D positions and corresponding body postures are quantified. By replaying these in RoboTwin, we reveal large benefits in thrust (around 50% compared to swimming alone) obtained by the follower exploiting vortices shed by the leader. Our results show the effectiveness of our design. We outline future applications of this system for the study of hydrodynamic interactions in schooling fish.

Oral (S20-701, Time: Tuesday 16:20, Room: Sulzer Chemtech)

Soft robotics and the quest for modeling embodied intelligence

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Soft robotics is largely motivated by embodied intelligence, or the emergence of sensory-motor behaviour from the interaction with the environment [1]. A soft body can deform under external interaction forces, in addition to the internal ones generated by its actuators, for obtaining the intended movements [2]. In order to take advantage of embodied intelligence in soft robot design, we need a formal mathematical description of it, which is still an open challenge in the field [3]. Modeling the internal and external interactions in a soft robot [4] would help both design and control of soft robots and would bring the field a step forward, in the transition from a pioneering trial-and-error field [5] to a model-informed discipline. [1] R. Pfeifer, M. Lungarella, F. Iida, "Self-Organization, Embodiment, and Biologically Inspired Robotics", Science 318:1088, 2007. [2] C. Laschi, B. Mazzolai, and M. Cianchetti, "Soft robotics: Technologies and systems pushing the boundaries of robot abilities," Science robotics, vol. 1, no. 1, pp. eaah3690, 2016. [3] H. Hauser, A.J. Ijspeert, R.M. Füchslin, R. Pfeifer, W. Maass, "Towards a theoretical foundation for morphological computation with compliant bodies", Biological Cybernetics 105:355–370, 2011. [4] G. Mengaldo, F. Renda, S.L. Brunton, M. Baecher, M. Calisti, C. Duriez, G.S. Chirikjian, C. Laschi, "A concise guide to modeling the physics of embodied intelligence in soft robotics", Nature Reviews Physics, 4(9):595-610, 2022. [5] E.W. Hawkes, C. Majidi, M.T. Tolley, "Hard questions for soft robotics", Science Robotics 6(53), 2021.

Oral (S20-702, Time: Tuesday 16:40, Room: Sulzer Chemtech)

Soft Robotic Arm with Extensible Stiffening Layer

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When talking about soft robots, softness is considered the most important feature, which brings dexterity and safety in interactive tasks with humans and environments. Such softness sometimes limits the real application of soft robots because load capability and rigidity are widely needed on many occasions. Often the inspiration for soft robots, soft animals such as octopus and sea cucumbers still preserve the ability to turn rigid when needed, and, more importantly, they can rapidly turn rigid at any deformed states. Intrigued by these capabilities, differently from existing stiffening technologies that do not adapt to soft robots' variety of morphological changes, we explored a new approach to create a flexible layer that can easily stiffen soft robots without affecting their original flexibility. We built an extensible chain of particles with a peculiar geometry, which can jam and stiffen. We enclosed it in a flexible membrane so that we can stiffen the structure by removing air. The overall structure is flexible enough and can withstand large elongation. So, it can be used around a soft robot arm for stiffening it in different deformed states. Experimental results show that such a stiffening layer does not affect the flexible motion of the soft robot, the stiffening function works even under the robot's 90% elongation state, and the stiffness can be increased by 15 times. By adopting the stiffening layer and different end effectors, we show that an assistive soft robot can simultaneously have the flexibility to collect food and the rigidity to hold it and feed a patient. The results from this work may provide new design and application insights into the creation of soft robots with both flexible and rigid features.

Oral (S20-722, Time: Tuesday 16:00, Room: Sulzer Chemtech)

Pneumatic bending actuators with ligaments and tendons

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Well-established actuators for macroscopic soft-machine applications use embedded pneumatic networks (PneuNets). Due to their design and soft bodies, grippers with classical PneuNet bending actuators must be continuously actuated to grasp and hold an object. Inspired by the passive viscoelastic properties of the human hand, we developed a novel bioinspired pneumatic bending actuator with integrated ligaments. The introduction of ligaments into the design specifically exploits the high design freedom of 3D printers and the high tensile strength provided by state-of-the-art soft filaments for FDM. Unlike traditional bending actuators, our design straightens and stretches its ligaments when pressure is applied and bends by relaxing the ligaments when pressure is removed. Using this principle, grippers only require energy input to open, while gripping or holding an object is powered by the relaxation of the ligaments and the energy stored there. To further improve the system, inspired by the digital tendon locking mechanism of the avian foot, we implemented a soft pneumatic tendon sheath and tendon to significantly reduce the maximum air pressure required to fully straighten the actuator. Locking the tendon increased the stiffness and block force of the actuator, while releasing the tendon allowed the actuator to straighten with less air pressure.

Poster (S20-393, Time: Thursday 17:00, Room: Foyer)

A multi-material robotic finger with integrated piezoresistive sensor elements

Clemens Frank[1], Georgopoulou Antonia[1], Hamelryckx Stijn Jacques[1], Junge Kai Christian[2], Hughes Josie[2]

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Robotic fingers with rigid and soft components and integrated sensors have not been explored so far because extensive manual work is required. We demonstrate the development of such devices using multi-material fabrication approaches that allow the 3D printing of fingers with incorporated sensing elements in one step. In this work, we propose utilizing a hybrid quad extruder 3D printer with combined pellet and filament-based extruders to enable the fabrication of a sensorized multi-material finger without additional manual manufacturing steps. Different designs of the finger joints were investigated to optimize the bending behavior. The investigation showed that by reducing the stiffness of the joints, the required force decreased, while the sensory performance (relaxation and sensitivity) and range of function was improved. The materials used for the bone and skin of the finger and the sensing elements consisted of thermoplastics and thermoplastic elastomers.

Poster (S20-694, Time: Thursday 17:00, Room: Foyer)

Soft Active Tail Enhances the Climbing Capabilities of the Robot

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In the past three decades, climbing robots have been developed for use in a variety of tasks, including inspection, cleaning, and maintenance of tall structures. In the last few years, several climbing robots have been reported, in a variety of sizes and weights and utilizing many different technologies. Among them, bio-inspired mechanisms used in climbing robots for locomotion have gained special attention. Biomechanical studies have shown the advantages of having a tail in climbing animals. The climbing experiments on the Asiatic gecko (Cosymbotus platyurus) have shown that they use their tails to adjust contact force actively and sufficiently to counter the animal's body pitch-back. In this study, we explored the role of stiffness and morphology of tails on the climbing performance by using a bioinspired robot as a test platform. The bioinspired robot consisted of whegs to drive the robot forward in steep inclined surfaces. The robot had a soft active tendon-driven tail to stabilize the pitch back and reduce the force required by the whegs to support the weight of the robot. We optimized the stiffness and morphology of the tail by testing the climbing performance of the robot by using a multiple 3d printed soft tails with different stiffness, coefficient of friction, and tail length. An optimised tail has made the robot more versatile regarding its operational conditions.

Poster (S20-721, Time: Thursday 17:00, Room: Foyer)

Soft Robotic Fish with Tail Fin Shape Changes to resurrect ancient Marine Species Thrust

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Animals have evolved highly efficient terrestrial, aerial, and aquatic locomotion capabilities. Even the most sophisticated robots of today are limited in scope and cannot swiftly adapt to abrupt changes in the environment. Our project aims to help close the distance between artificial and natural swimming locomotion.

Swimming animals can modify the amplitude of their undulations and the rigidity of their bodies to optimize their performance at a variety of velocities. Investigating the effect of bilateral muscle activation on propulsive output in free-swimming, living fish is difficult due to the difficulties associated with control experiments involving the manipulation of muscle activation in living animals. To examine the effect of cocontraction on body stiffness control, we propose using soft robotic model animals as experimental platforms to resolve the soft sensory performance and gain insight into the swimming behavior of subcarangiform fish.

We utilize our knowledge of soft robotic control and fabrication to create a soft robotic platform with embedded soft sensors capable of mimicking the locomotion of animals.

In a recirculating flow tank, we investigate the swimming performance of a bio-inspired soft robotic fish equipped with two pneumatic actuators and soft strain sensors at undulation frequencies between 0.3 and 0.7 Hz and flow rates between 0 and 20cm/s.

We intend to evaluate various tail morphologies and foil stiffnesses to determine their effect on swimming performance.

Combining physical modeling of experimental biomechanics performance with robotic replications of the agile and robust movements of animals, this project takes an interdisciplinary, multi-modal approach.

Poster (S20-724, Time: Thursday 17:00, Room: Foyer)

The Venus Flyflap: Combining two snap trap motion principle into one plant inspired artificial Venus flytrap demonstrator

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Plants, as sessile organisms, are exposed to their environment without any protection but still cope with changing conditions. Especially in soft robotics, properties like adaptation to environments, ingenious materials systems, and energy-effectiveness of plants provide an extremely rich source of inspiration to develop new technologies - and many of them are still in the beginning of being discovered or transferred. Snap traps plants are of particular interest here, as these are to catch fast-moving insects without any central control unit. The translation of the functions of snap traps enables the development of systems with embodied (physical) intelligence and embodied energy. Current artificial Venus flytraps inspired by carnivorous snap-trap plants enable fast, hinge-less movements in plant-inspired robotic systems.

Our novel bioinspired "artificial Venus flytrap" demonstrators incorporate snap-trap motion principles of two carnivorous plants (Venus flytrap and watertrap) in one system, exhibiting adaptive responses to various environmental triggers. The presented systems are examples of a successful implementation of several principles based on plant motion and deformation in a versatile, adaptive, and technically compliant multi-material system. The systems and novel motion sequences are characterized in terms of kinematics, energy requirements and overall performance.

The aim of our project is to showcase the practicality of our material systems, which have dynamic, life-like, and non-equilibrium properties, through the use of bioinspired technical devices such as an "artificial Venus flytrap". This will serve as an initial step towards introducing innovative technologies into industrial products and everyday life applications.

Keynote (S21-108, Time: Tuesday 10:15, Room: Mettler Toledo)

SLS 3D Printing for Polymer Self Healing Lattice Structures

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Lattice structures are topologically ordered, three-dimensional open-celled structures composed of one or more repeating unit cells, which is popular for 3D printing. The designed lattice structures not only can achieve the lightweight function by introducing the pores into the matrix, but also endow the materials digitally-controlled functions to meet various applications. However, lightweight could weaken the materials strength, fatigue and reliability. Especially, the beam or strut with a small diameter in the lattice structure is easily broken during repeatedly deformation, for a reliable and durable lightweight device, the self-healing lattice (SHL) structure is of great value. In the past few year, we have developed a series of flexible polymer materials suitable for SLS 3D printing, such as TPU, PDMS and their composites powders. The relationships between polymer structure, powder morphology, melt rheological behavior, processing parameters and final material properties were studied in order to improve the printing qualities and mechanical properties. We developed the covalent adaptable networks (CANs) of polyurethane and polydimethylsiloxane containing dynamic bond for SLS 3D printing. The interlayer interaction is significantly improved by dynamically chemical crosslinking and also the printed parts have the excellent self-healing function. Furthermore, we realized the construction of conductive and self healing lattice structure by SLS printing. The diversified lattice structures have different strain distributions during stretching or compressing, which will affect the electrical resistance behavior. The relationship between lattice structures and piezoresistive properties was investigated. Interestingly, we found Schwarts structure has the highest Gauge factor compared to Gyroid- and Diamond- structures. The fundamental mechanism was investigated by Finite Element Modeling. References: 1.Z.C. Li, Z.H. Wang, X.P. Gan, D.H. Fu, G.X. Fei, H.S. Xia, Macrom

Keynote (S21-629, Time: Tuesday 13:45, Room: Mettler Toledo)

In-situ monitoring of powder bed fusion of polymers

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[2]ETH Zurich, Zurich, Switzerland

Powder bed fusion of polymers allows to produce complex and customized parts that are impossible to make using traditional polymer processing technologies. Process monitoring can be the key to effectively exploit the high production time of AM to gather relevant data on the part itself, which can be used to qualify it on the fly. In this work, laser profilometry and microwave tomography are integrated in a powder bed fusion of polymers machine: the former allows to monitor the powder bed quality, but also to spot and quantify relevant process issues such as curling, whereas the latter enables the measurement of the temperature history of a component during cooldown, while it is surrounded by powder, possibly allowing the prediction of its future crystalline structure.

Oral (S21-015, Time: Tuesday 11:40, Room: Mettler Toledo)

Energy conversion aspect in laser powder bed fusion of nanoparticle supported PA12 powder feedstocks

Rudloff Johannes[1], Grünewald Moritz[1], Popp Kevin[1], Chehreh Abootorab[2], Kusoglu Ihsan Murat[3], Barcikowski Stephan[3], Nowicki Alexander[4], Schuffenhauer Thomas[5], Bastian Martin[1]

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[4]University of Hamburg, Hamburg, Germany
[5]Bayrisches Laserzentrum GmbH, Erlangen, Germany

Additive Manufacturing (AM) offers a high freedom of design and the possibility to shape complex parts cost-efficiently, especially for small lot numbers. Currently, powder bed fusion using laser beam (PBF-LB) process is best suited to produce polymer components that fulfil the industrial requirements. Even though AM is about to shift from a prototyping stage to the production of technical components, the existing material range is still very limited. To counteract this problem, available powders can be modified through additives, e.g. nanoscale materials to adapt the material properties. For a better understanding of how to process these nano- additivated materials at best, analytical considerations of the PBF-LB process are a powerful tool. In this study, material and process data are combined to determine the energy conversion with dimensionless numbers. Therefore, we investigated the influence of nanoparticles experimentally on the polyamide-12 (PA-12) powder feedstock properties and the resulting processing behaviour. The surface of PA-12 powder was additivated with silver and carbon black nanoparticles. For this purpose, monolayers with different surface energy densities are printed and their thicknesses are measured via dial gauge. The process is described by a dimensionless energy input value, which sets the introduced surface energy density by the laser in relation to the melting enthalpy and the resulted monolayer thickness. The respective values represent the energy turnover during processing. Values slightly above 1 indicate, that almost all laser-energy is converted to melt the polymer. The results indicate that the presence of nano-additives has a massive impact on the material properties, e.g. on the laser absorptance. Analytical based optimised process parameters were then chosen to manufacture multilayer samples. These were used to measure the influence of the nanoparticles on the pore-volume by µCT. Additionally, mechanical properties were obtained from tensile testing.

Oral (S21-084, Time: Tuesday 11:00, Room: Mettler Toledo)

Ageing Behavior of Polyamide Powders for Powder Bed Fusion

Freihart Karl[1]

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Additive Manufacturing with thermoplastic polymer powders is emerging into industrial applications. As a result of economic and ecologic demands in serial applications, a major focus needs to be placed on sustainability. In terms of refreshing of used powders and circular economy, a deep understanding of the ageing behavior of polyamide powders for Powder Bed Fusion (PBF) processes is key. Furthermore, to extend the life span of the polymer material, knowledge on ageing mechanisms is also base for high value recycling of parts. In a comparative study, different grades of commercial polyamide powders (PA11, PA12 with different chain growth stabilizers) have been aged under conditions mimicking time and temperature dependencies in the PBF process. The samples have been analyzed in order to evaluate chain growth and crosslinking, degradation and annealing effects. The work focusses on these analytical approaches to allow prediction of process behavior of polymer powders throughout the lifecycle within PBF and beyond. In comparison, these analytical data are compared to PBF production processes with the respective material to prove relevance of the simulative conditions and allow derivation of process adaptions for future products or to extend the lifecycle of existing materials in PBF.

Oral (S21-088, Time: Tuesday 11:20, Room: Mettler Toledo)

Powder-based 3D printing with focused ultrasound transducers

van Berlo Frank[1], Anderson Patrick D.[1], van Breemen Lambert[1]

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Powder-based 3D printing techniques have a larger potential for developing and manufacturing prototypes and customized products as compared to other printing techniques, since components with near-net-density and near-net-shape can be produced from fine powders [1]. However, conventional techniques like SLS require powders with uniform sizes and good flowability to produce these dense parts [1]. Processing a variety of materials becomes limited due to the availability of materials in the desired powder form. Another limitation of SLS printing is the rapid sintering of particles that involves high heat transfer and (moving) components with temperature gradients, which require a high level of control [2]. To tackle the aforementioned limitations, a new type of powder-based 3D printer is being developed. Here, the energy source for melting is a collection of many focused ultrasound sources positioned on a side of an enclosure. The transducers focus on a small spot inside the enclosure. As in phased array transducers, the position of this focus spot can be moved through the volume. Hence, a volumetric 3D scan can be performed to selectively fuse polymer particles. So, in this multi-transducer design, no moving components are required to spread or sinter the powder. We aim to use a model-based approach to generate a design and prototype of the ultrasound 3D printer. In this work, we determine the model's acoustic input parameters and the conversion of ultrasound into heat. To do so, a setup is built in which liquids, solids, and most important powders can be acoustically characterized in terms of speed of sound and energy loss (attenuation). [1] N. Jeyaprakash, et al., Advances in Additive Manufacturing, Tapei (2021). [2] Y. Chivel and I. Smurov, Phys. Procedia. 5B (2010).

Oral (S21-128, Time: Tuesday 10:40, Room: Mettler Toledo)

Experimental and numerical investigations on the influence of the interlayer time in powder bed fusion of PA 12

Grünewald Moritz[1], Popp Kevin[1], Rudloff Johannes[1], Hochrein Thomas[1], Bastian Martin[1], Bierwisch Claas[2]

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In the field of plastics, powder bed fusion using laser-beam (PBF -LB) represents the industrially most relevant additive manufacturing technique. In order to increase the productivity of the PBF-process, reduced productions times are of great interest. Shorter build-times are, thus, economically desirable but their influence on e.g. mechanical properties are not fully understood. The densification in PBF is slow due to the small ratio of surface tension and viscosity. The application of a subsequent powder layer prior to full densification of the layer below might lead to a significant residual porosity in the part. In contrast, faster repeated laser irradiation can lead to higher melt temperatures and thus faster compaction. In our work we investigated the influence of the inter-layer time (ILT; time gap between two recoating steps) on the resulting part properties of polyamide 12. An experimental test setup was developed to study the stepwise thickness increase of the first ten 100 µm layers. Moreover, four different ILT between 30 s and 90 s were set in order to examine the influence of the time gap on densification. Micro-computed tomography measurements were used to characterise the pore formation. Mechanical properties were obtained by tensile tests. The results indicate that vertically and horizontally oriented tension rods are affected differently. In parallel to the experimental investigations, numerical simulations were conducted. A simulation tool was developed and implemented for comparison to experiments. The one-dimensional finite difference solver takes into account laser irradiation and melting of the powder bed as well as heat conduction, densification and crystallization of the polymer melt as functions of the building direction. Several layers can be simulated under realistic process conditions. We found that the competition of the time scales of densification and crystallization and the ILT give rise to a wide range of porosity profiles.

Oral (S21-129, Time: Tuesday 12:00, Room: Mettler Toledo)

Novel Bio-Based Poly(3-hydroxybutyrate-co-3-hydroxyhexanoate) Powders for Selective Laser Sintering Additive Manufacturing

Colucci Giovanna[1], Giubilini Alberto[1], De Trane Giorgio[1], Lupone Federico[1], Badini Claudio[1], Minetola Paolo[1], Bondioli Federica[1], Messori Massimo[1]

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Additive manufacturing (AM) is playing a crucial role in the world of manufacturing due to its high flexibility, geometrical accuracy, reduced time and costs, and minimal waste production. Selective laser sintering (SLS) is considered the most favorable AM approach for plastics. SLS is a powder bed fusion process where the fabrication of 3D objects is achieved by layer-by-layer selective melting of powders by employing a laser. It requires good powder flowability, which critically depends on the particle shape. Novel powders of good processability are essential to broaden the application fields of powder-based AM process. Bio-based polymers which have attracted great attention due to the increasing interest in sustainable materials can be good candidates for SLS applications. This research focuses on the preparation and characterization of bio-based microspheres, obtained by oil-in-water emulsion solvent method, with the purpose to use them for the first time as powders for SLS, starting from poly(3-hydroxybutyrate-co-3-hydroxyhexanoate) (PHBH) as polymeric matrix, dissolved in chloroform and added drop-by-drop to an aqueous solution of poly(vinyl alcohol). The results revealed that microspheres showed specific particle size and sphericity useful to be processed by AM. The PHBH microspheres were used to study their effective 3D printability using geometries varying from simple shapes to more complex architectures. We realized samples with monolayer geometries with increasing complexity, and samples with more complicated structures and a higher number of layers. This study certainly deserves further investigation to better evaluate the scalability of the microsphere fabrication process, and to optimize the additive manufacturing process in order to obtain more complex 3D structures. With this preliminary research work, we have opened promising future scenarios for novel applications of this environmentally friendly polymer in additive manufacturing.

Oral (S21-209, Time: Tuesday 14:10, Room: Mettler Toledo)

Advanced Characterisation of the Ageing Effects of Polymer Powders for Additive Manufacturing

Marsh Shona[1], Rudolph Natalie[2], Duffy John[3], Agostini Serena[3], Majewski Candice[4], Strasser Claire[2]

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Additive manufacturing is a group of fast-growing technologies that are rapidly maturing. An important part of this development are the insights material characterization provides to understand and optimize the processes as well as developing new materials. This is particularly true for powder bed fusion (PBF) of polymers, where the vast majority of the market is dominated by nylon-12 materials. During PBF, solid powder forms the support of a melted part shape. This powder remains at elevated temperatures, just below the melt, for significant periods of time; sometimes many hours at the bottom of the build. Therefore, they are at risk of ageing with consequences on processing behaviour and part performance. Typical materials used in PBF are nylons and some TPUs with more and more new materials, including commodity polymers such as polypropylene, being under investigation. We have used characterization techniques including Differential Scanning Calorimetry (DSC), Rheology and Gel Permeation Chromatography (GPC) to assess indicators of ageing, such as molecular weight or property changes, of well-understood polyamide materials to compare with polyamide with fillers and polypropylene powders as more novel materials. Rheological measurements on the different aged PA powders indicated that complex modulus, cross-over frequency of G′ and G″ as well as GPC and DSC data provide valuable information about ageing. For example, it showed polyamides are more sensitive than polypropylenes. Both rheology and DSC was utilised to study the crystallisation and flow behaviour of the PA powders during powder application and the coalescence phase during the process. The results from this study collectively show that ageing powders may increase the elastic response of the material, influencing the powder application in the powder bed fusion process. Consequently, this might mean different processing parameters are needed in order to accommodate different levels of aged powder.

Oral (S21-269, Time: Tuesday 14:30, Room: Mettler Toledo)

Thermal conductivity of glass/talc filled Polyamide 12 as function of tapping level

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Selective laser sintering is a layer-by-layer process that allows complex shapes to be processed but requires specific properties for the powder used to build the final object. The advantages of free crucible processing and freedom of design makes this process interesting for several applications. Despite the large number of SLS printer available in the market, a deep understanding of the physical phenomena of this process is still lacking, due to the number of parameters involved, directly influencing the final part's properties through the microstructure. In this work, glass beads or talc were added to Polyamide 12 (PA 12) matrix in order to evaluate these filler's influence on powder bed. We started with PA 12 and glass beads (PA90/GB10 in wt%) untill 70/30 wt%, with a 5 wt% increment. As SLS operates with CO2 laser working at 10.6µm to melt powders, it is fundamental to investigate how heat will be conducted through the different sintered layer. Various parameters can influence thermal conductivity of filled Polyamide 12 powder bed such as particle shape, aspect ratio, chemical composition, presence of impurities and also the packing of the particles from powder bed. Hence, porosity is such an important parameter as air inside is highly insulating the material and reduces its thermal conductivity by convection between pores. This convection is also governed by the pores size. (Rayleigh criterion) Several experiments were carried out in order to evaluate the thermal conductivity as a function of porosity which highly depends on how particles are settled, thus how the powder bed is tapped. These results were obtained using a hot wire conductivity meter from Neotim which can give thermal conductivity through a powder bed. To determine samples porosity, we used a powder tester which returned powder densities for different tapping levels. As we are working with filled polyamide 12 matrix, a huge drop of flowability has to be considered upon addition of filler which can cause issues during the process. Changes of processing parameters could be required due to significant modifications of the thermal behavior resulting from the presence of fillers.

Oral (S21-329, Time: Tuesday 15:10, Room: Mettler Toledo)

Powder bed fusion of poly(ethylene terephthalate) (PET)

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Powder bed fusion (PBF) is a method of additive manufacturing where the raw material is a powder (polymer or metal). For polymers, there are in fact two methods: selective laser sintering (SLS) and high speed sintering (HSS). In SLS, an infrared laser scans the powder bed in the areas of the cross section to be fused. This is done layer by layer. In HSS, the powder bed is ink-jet printed with a carbon black ink, and then an infrared lamp is passed over the bed in a single pass, and the blackened area gets hotter selectively and fuses. As no rastering with a laser is needed, HSS is faster and it is hoped it can compete with injection moulding production speeds. However, whereas most polymers are tolerant to the injection moulding process, one major obstacle for PBF is that the process is intolerant to most polymer powders. Amorphous polymers are less suitable than semi-crystalline polymers as the heat diffuses out and softens the surrounding powder outside the object envelope. Semi-crystalline polymers do not have this problem, but show various difficulties arising from shrinkage on crystallisation, such as curl, warpage and delamination of layers. Besides extrinsic properties (like particle size and shape), the intrinsic properties of the polymer like melting and crystallisation narrow the number of polymers that are process-tolerant for PBF. The most commonly used polymer for PBF has been polyamide 12, with ~90% market share. Polyamide 11, polyamide 6, polypropylene and poly(butylene terephthalate) have also been introduced commercially. In this paper, we shall show that PET polyester powder (developed originally by SABIC) has the intrinsic properties needed for SLS and HSS. Good mechanical properties are shown and the ability to build complex parts with good finish is demonstrated. PET has a higher service temperature than PA 12. The PET powder and the printed articles can be recycled without contaminating the PET recycling stream.

Oral (S21-444, Time: Tuesday 14:50, Room: Mettler Toledo)

Impact of layer time and hatch conformity on the behavior of thin-walled components in powder bed fusion of polymers

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The effects of process variables like hatch conformity and layer time are often overlooked and, as a result, are not fully understood. This is particularly true for thin-walled components. This work specifically addresses the impact on thin structures, which are critical for maximizing the potential of the laser-based powder bed fusion process. Therefore, tensile specimens of various thicknesses and orientations were manufactured, and the hatch conformity and layer time were altered. For a more profound process understanding, infrared thermography is used to analyze the developing exposure temperatures. While the hatch count indicated an impact on the resulting exposure temperatures, the impact on the resulting part properties is minimal. The layer time shows a notable influence on the part geometry and density. When considering the mechanical properties, young's modulus can be described as constant, independent of the layer time and hatch conformity. The elongation at break shows no influence of the hatch conformity but the layer time. Overall, the most significant changes occurred due to variations in the part thickness and must be considered in part optimization.

Oral (S21-481, Time: Tuesday 16:00, Room: Mettler Toledo)

Local integration of electrically conductive paths using an in situ x-y positioning system in selective laser sintering

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In additive manufacturing there are some disadvantages, you have to deal with. For example, highly filled material systems cannot be processed in additive manufacturing, especially in selective laser sintering. However, it has already been shown that manufacturing with low-filled material systems can succeed. So far, the focus was in the functionalization with regard to to improving mechanical properties on a global scale. In this study, the functionalization potential is to be investigated with regard to the generation of locally deposited electrically conductive paths by means of an x-y positioning system. In addition to various filler systems, copper, solder powders and aluminum will be used for this purpose. Before deposition, these powders are mixed volumetrically with the polyamide powder used in the process and fed into the nozzle intended for deposition. In order to investigate the influence of the track width on the electrical conductivity, the process parameters of the x-y positioning system are also varied. This allows active influence to be exerted on the track geometry in terms of its width and height. The adjustable parameters here include the traversing speed, the adjustable height between the powder bed and the nozzle, and the frequency and amplitude of the piezo element. The piezo element controls the vibration of the nozzle and therefore the amount of deposited onto the poweder bed surface.The deposited tracks are processed in the SLS into individual layers and examined by means of microscopy for the geometric and morphological properties as well as the connection of the track to the individual layer. The morphological and light microscopic analysis is followed by the investigation of the electrical conductivity. It is found that the tethered tracks are both parameter and filler dependent with the respect to the resulting geometry of track. The bonding quality differs significantly with variation of the filler contents in the deposited traces.

Oral (S21-592, Time: Tuesday 16:20, Room: Mettler Toledo)

Investigating the influence of heat treatment in a pressurised atmosphere on the ductility of SLS-printed PA12

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In the last decade, Selective Laser Sintering (SLS) of Polyamide 12 (PA12) has evolved into a production method for end-use components in non-demanding applications. There is growing interest from the industry to apply SLS-printed PA12 in demanding end-use applications such as machine parts, orthopaedics and aerospace components, where safe operation is vital and high ductility is required. SLS-printed PA12 is strong but brittle compared to moulded PA12, which is commonly attributed to porosity of the printed material. The state-of-the-art SLS process cannot produce fully dense parts, so post-processing techniques were studied for their effect on part density, in particular Hot Isostatic Pressing (HIP). By means of elevated temperature and isostatic pressure, HIP is used to mould dense materials from metal, ceramic and polymer powders and also to increase density and ductility of printed metal components. Experiments with HIP were performed to gain insight into the influence of heat treatment in a pressurised atmosphere on the part density and mechanical properties of SLS-printed PA12. Parts were treated for 4 hours at 175°C at pressures of 18 and 1500 bar in separate experiments. Parts mostly retained their shape and under specific conditions a small increase in part density was observed, however the ductility and other mechanical properties decreased. Although porosity of SLS-printed PA12 undoubtedly affects ductility, the results of the HIP experiments lead to the question whether porosity is the leading cause for low ductility. The melting phase in SLS is brief compared to common moulding processes, and it may have a negative impact on the bond between particles. Further heat treatment experiments performed above the peak melting temperature resulted in a dramatic increase in ductility of the SLS-PA12 specimens and indicate that the brief melting process of SLS, and not part porosity, is the leading cause for low ductility of SLS-printed PA12.

Oral (S21-692, Time: Tuesday 16:40, Room: Mettler Toledo)

Selective laser sintering on the way to industrial application: A holistic approach for a digitized demonstration factory

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At present, additive manufacturing with polymers already allows the economical production of components in individual pieces or small quantities with good application-specific properties and a high degree of individualization and complexity. However, additive manufacturing processes are still associated with many manual postprocessing steps and thus high costs. Consequently, series applications for additively manufactured parts fail today in most cases due to the high process costs as well as the lack of quality assurance tools. For the economic use of additive series production, the above-mentioned hurdles must be overcome. In order to be able to investigate powder-based additive manufacturing processes comprehensively up to the depowdered component, the research institution Neue Materialien Bayreuth GmbH (Germany) is currently setting up a demonstration factory for selective laser sintering with polymers. This contribution presents the concept of this unique research environment. The demonstration factory represents the entire processing chain including automated de-powdering, surface cleaning, part identification and inline quality assurance. All subcomponents are connected to a central IOT platform. In addition to the parts workflow, the closed powder circuit is also the subject of consideration. With detailed in-line and at-line analytics along the process chain, the entire SLS process can be evaluated, potential problems during processing can be identified, process stability can be improved, and part properties can be precisely assigned. This paper presents the relevant methods. End-to-end digitization enables a transparent visualization of all media and material flows as well as a part-related life cycle assessment. The research infrastructure described here offers the optimal prerequisites for the establishment of robust manufacturing strategies and thus an economical use of SLS technology for series components.

Oral (S21-697, Time: Tuesday 17:00, Room: Mettler Toledo)

Polypropylene in Laser-Based Powder Bed Fusion of Polymers

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Currently, the majority of laser powder bed fusion (PBF-LB/P) processes use polyamides, which prevent the components from exhibiting the mechanical characteristics of traditional manufacturing techniques; as a result, highly ductile materials are needed to cover all potential application areas. The preferred material thereby is polypropylene with its various structures and settings. Available studies show that isotactic Polypropylene is processable, but exhibits brittle behavior, therefore other structures are needed. To meet this challenge and realize the full potential of PBF-LB/P, this work first includes an analysis of the material properties to provide a statement on the processing performance; then the processing and characterization of the mechanical performance of fitting polypropylene types is investigated.