Thermal decomposition in polyol : an efficient process for the synthesis of magnetic nanoparticles

L. Van Leuven[1], T. Vangijzegem[1], D. Stanicki[1], S. Laurent[1,2]

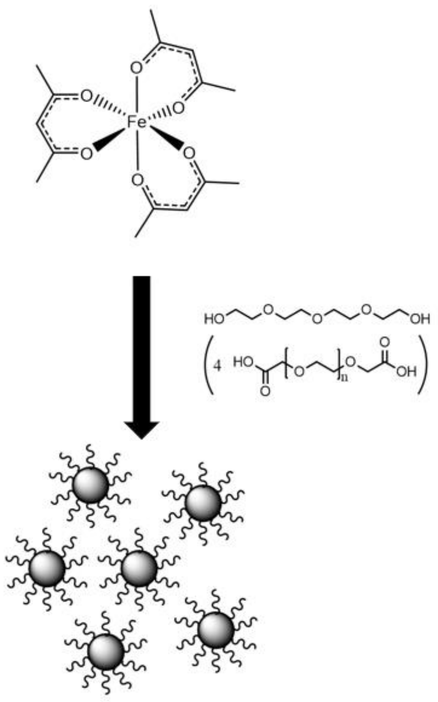
[1]General, Organic and Biomedical Chemistry Unit, NMR and Molecular Imaging Laboratory, University of Mons (UMONS), 7000 Mons, BELGIUM

[2] Center for Microscopy and Molecular Imaging (CMMI), 6041 Gosselies, BELGIUM

Introduction

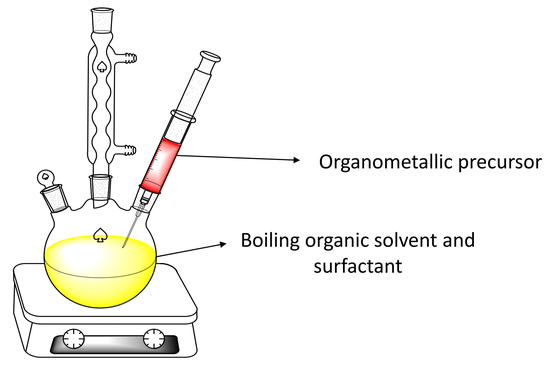
Iron oxide nanoparticles (IONP) are widely studied for their superparamagnetic properties making them suitable as MRI contrast agents. If many synthesis pathways have been described, one of the most popular is the thermal decomposition of organometallic compounds; the success of this process lying on its ability to produce IONPs with a good control over their size, shape, and crystallinity. Typically, the process takes place in high boiling solvent in the presence of hydrophobic surfactants (i.e. oleic acid and/or oleylamine).

As a consequence, the resulting nano-objects need further surface modifications to allow water transfer. To avoid this pitfall, we propose to modify the original process by of changing the hydrophobic materials by hydrophilic ones. Owing to its interesting properties (viscosity and high boiling point), the tetraethylene glycol appears as a suitable solvent for IONP synthesis.

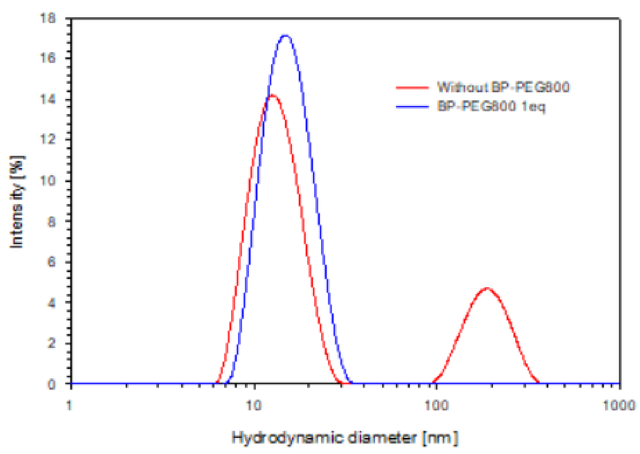


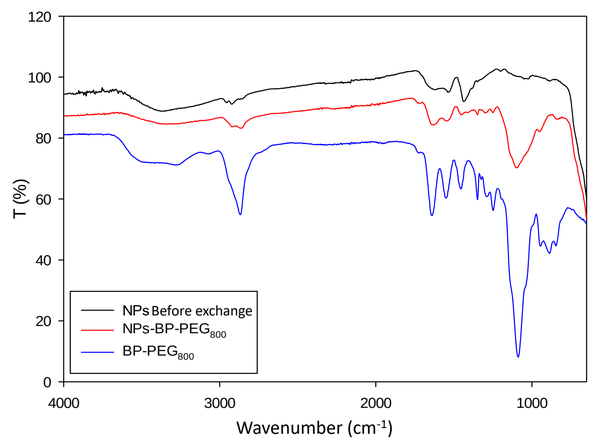
Method and results

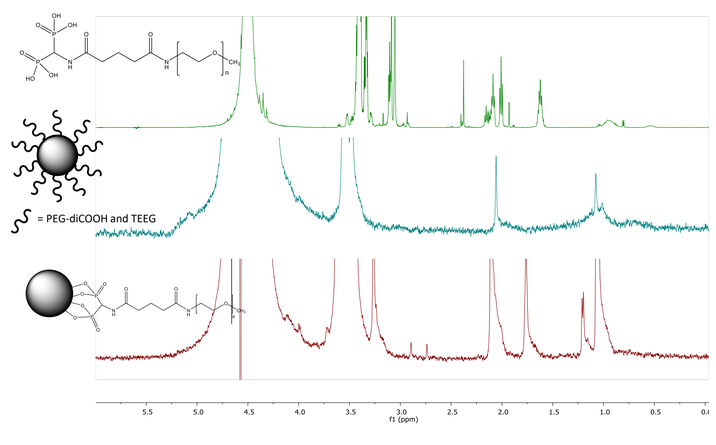
$Fe(acac)_3$ in tetraethylene glycol (50mM) was injected in a pre-heated solution of TEG. Reaction time and temperature were fixed at 5 minutes and 250°C respectively. Different precursor/PEG-diCOOH ratios were tested to evaluate their influence on the properties (size, magnetic properties and colloidal stability) of the resulting objects.



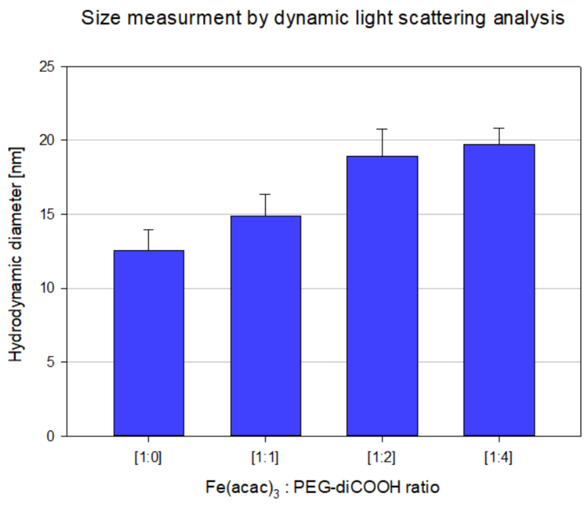
After synthesis, the particles were easily isolated and transferred in water. The as-obtained particles were stabilized by mean of ligand addition process, using a “home-made” system (i.e. BP-PEG800). The efficiency of the process was assessed by FT-IR and NMR (HR-MAS) spectroscopy. By proceeding this way, stable suspensions were obtained.

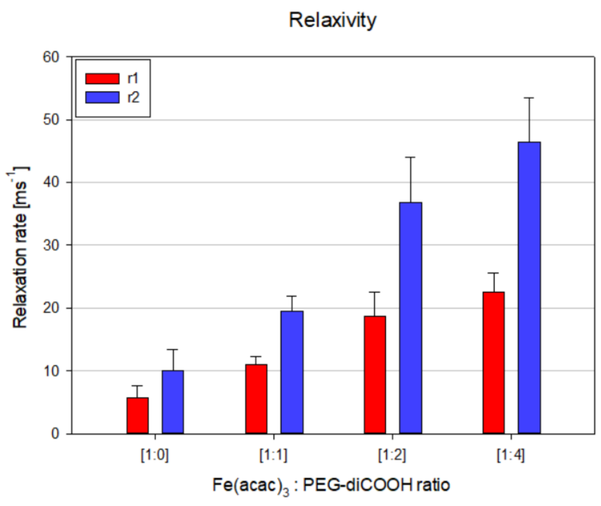






Interestingly, we observed that the presence of PEG-diCOOH in the solution have an influence on the physico-chemical properties of the resulting IONPs. This observation could be correlated to the ability of such structure to modify the decomposition kinetics, influing thus the germination and particle’s growth steps.





Conclusion

In conclusion, the described protocol enables to synthesise hydrophilic IONP with interesting magnetic properties. The use of PEG-diCOOH stabiliser during the process clearly impact these properties. For further studies we would like to adapt this synthesis pathway to flow chemistry to gain more control over synthesis parameters such as heating time and pressure. Furthermore, flow synthesis should allow better reproducibility and scaling-up.

Acknowledgement

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