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# Methodological evaluation of demixing solvents used in the absorption-regeneration carbon capture process

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# Abstract

Since the beginning of the 21<sup>st</sup> century, the effects of global warming have been more and more visible, especially through extreme climate phenomena. Anthropogenic emissions of greenhouse gases (especially carbon dioxide) have been identified as the main responsible for such climate change. Among the numerous studied technologies to decrease CO<sub>2</sub> emissions, the absorptionregeneration process is the most developed at industrial level. However, the main inconvenience of such a process is its high demand for thermal energy. The solvent used for the CO2 absorption is an important parameter of the process and its choice is a key factor to reduce that required heat. Among the available solvent blends, the demixing solvents allow to reduce the flow needed to be regenerated. Simultaneously, this stream is more concentrated in carbon dioxide. Both contribute to reducing the regeneration energy which causes the highest operational costs of the absorption-regeneration process. For various reasons (e.g., production costs for ionic liquids, unknown water tolerance for water-lean solvents), only aqueous liquid-liquid amine-based mixtures are considered for this evaluation methodology. 30 different blends were identified through a literature review as potential candidates for further possible development. These blends are made of 23 organic compounds in different compositions and/or proportions. 12 of them are ternary blends composed of water and two amine-based molecules. The other mixtures contain a phase-splitter agent in addition to water and amine(s). These chemicals, used to induce or enhance the demixing behavior, are mainly sulfolane and alcohols. The evaluation methodology is composed of two successive steps. The first step is a techno-economic evaluation to classify the reviewed solvents. This evaluation is based on six indicators. The main one remains the regeneration energy as it is the parameter that should be reduced. CO2 absorption capacity and CO2 absorption rate are also considered for the thermodynamic and kinetic aspects. The demixing phenomenon is included in the evaluation through the demixing volume ratio. The two last indicators are the volatility of organic compounds (as it can have impacts on the process design) and the solvent cost (which is the only economic-exclusive criterion). The contributions of these six indicators are weighted using the analytical hierarchy process method and finally give a classification of the 30 identified blends. Among them, 17 solvents are excluded due to the health, safety, and environmental analysis (second step). Indeed, these blends contain at least one compound that presents a serious hazard (mortal hazard, serious fertility danger, corrosivity, high toxicity to aquatic life). Finally, 13 aqueous liquid-liquid biphasic amine-based blends remain and are classified through their score owned after the first step of the evaluation method. The aqueous solvents ranked from the 5<sup>th</sup> to the 2<sup>nd</sup> place are composed of two amine molecules. One contains primary/secondary amino groups (MEA, MAPA, TETA) while the other contains a tertiary amino group (DEEA, DMCA). Nevertheless, among the reviewed aqueous blends, the most promising demixing solvent is composed of triethylenetetramine (30 wt.%) and propan-1-ol (50 wt.%).

Keywords: Carbon capture; CO<sub>2</sub> absorption; Biphasic liquid-liquid solvents; Methodological evaluation

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# 1. Introduction

Since the beginning of the current century, the consequences of the global warming have become more and more visible and intense. The anthropogenic emissions of greenhouse gases are the main source of the current climate changes. The most emitted greenhouse gas remains by far, the carbon dioxide  $(CO<sub>2</sub>)$  with an average global concentration of 424 ppm in 2024 in the atmosphere [1]. It is currently mainly produced by the combustion of fossil fuels (electricity and heat generation, heat transport, residential, …) but also intrinsically by some industrial processes such as cement or lime production (decarbonation of CaCO<sub>3</sub>). Even with low-carbon energy sources, these processes will therefore need CO<sub>2</sub> capture plants to abate their carbon emissions. Three main technologies are currently available to tackle these emissions: pre-combustion, oxy-combustion and post-combustion carbon capture. Among the possible methods in the post-combustion capture sector, the absorption-regeneration process is currently the most developed one at industrial level, using aqueous amine-based solvent (e.g., monoethanolamine-MEA 30 wt.% or methyldiethanolamine-MDEA 30 wt.% with piperazine-PZ 10 wt.%).

However, the absorption-regeneration process requires a considerable amount of heat for the regeneration of the CO2-absorption solvent. For the reference solvent (MEA 30 wt.%), this required heat is generally held between 3 and  $4 \text{ GJ/tCO}_2$  at a temperature level of around 120 °C and a pressure of 2 bar [2,3]. Multiple improvements of the process can be used to reduce the thermal energy required such as an intercooling stage, a rich or a lean vapor compression before the stripper column [4,5]. The solvent itself used in the process also has an important impact on the operative costs, especially on the heat requirements. The carbon dioxide absorption can be achieved using physical or chemical solvents, respectively mainly driven by the CO<sub>2</sub> partial pressure or the reactivity of the solvent with CO<sub>2</sub>. Hybrid solvents (e.g., ionic liquids) form a more recent category of solvents but their cost related to the production method slows down their large-scale development [6]. The chemical solvents remain the most used at industrial level and are mostly composed of amine(s) blend. Some solvents (e.g., based on amino acid salts) can be implemented to capture CO2 [7]. These mixtures can form a precipitate, leading to a liquid-solid phase separation. However, the presence of solid phase is a particular challenge, especially for the transportation of this CO2-rich phase. Therefore, such phenomenon should be avoided for a process using liquid-liquid solvents.

The schematic flowsheet of the carbon capture process using a biphasic liquid-liquid solvent is represented in Fig. 1. Between the absorption and the regeneration columns, the core of this process is the phase separator device (also called decanter). After the  $CO<sub>2</sub>$  absorption, this equipment is the place where the two liquid phases are separated. The  $CO<sub>2</sub>$ -lean phase is directly sent to the absorber. Only the  $CO<sub>2</sub>$ -rich phase is carried to the regeneration part of the process where it is heated to release the carbon dioxide from the solvent. After this process part, the regenerated flow is mixed with the CO<sub>2</sub>-lean phase to be recirculated to the absorption column. The two main advantages of this phase separation are on one side to regenerate a smaller liquid flow and on the other side that the solvent to be regenerated has a higher  $CO<sub>2</sub>$  concentration. These characteristics of the flow to be regenerated lead to a significant lower energy needed at the reboiler.



Fig. 1. Typical flowsheet of an absorption-regeneration process using liquid-liquid biphasic solvent (adapted from Liu S. et al. [8])

Many chemical mixtures have been studied and tested to be used as liquid-liquid biphasic solvents in a postcombustion carbon capture process, but mostly at laboratory scale. Some of them have the potential to reduce the heat requirements by more around 50% [9]. However, the process energy parameter is not the only parameter to consider identifying the most interesting solvents that should be studied for an implementation at industrial level. After a literature review of the biphasic liquid-liquid solvents, this work therefore sets out to evaluate the various mixtures using a method involving several indicators. This leads to a broader assessment, allowing to select the most promising blends for further study to pilot and finally commercial scales.

# 2. Literature review

The demixing solvents have been studied in the past, mostly at a small scale. This section contains a summary of the literature review concerning these liquid-liquid blends. More recently, non-aqueous (or water-lean) have been studied as a new category of potential biphasic solvents. However, water is generally present in the industrial flue gas itself and/or used to cool it (through a direct contact cooler). The utilization of a blend without water can be so critical as the flue gas should be dried before the absorption. Otherwise, the water will progressively go to the solvent phase in the absorption column. The water tolerances of some of these solvents have been estimated but most of them remain unknown. Moreover, no pilot-scale experiments have been reported until now to verify these estimations [10]. Facing such uncertainties, the present work only focuses on the aqueous biphasic liquid-liquid blends. In this field, the installation with the highest technology readiness level is an industrial pilot unit built at the ArcelorMittal plant in Dunkirk, France. This unit uses the DMX™ solvent, an aqueous mixture of two amines, developed by IFPEN [11]. However, the properties of these components remain largely unknown, causing difficulties to correctly evaluate this solvent and compare it to others. Like the DMX™ solvent, some aqueous liquid-liquid biphasic solvents are composed of two amine molecules.

In the literature review, 12 blends of them are reported and generally include a primary and/or secondary amine with a tertiary amine. Different reactional mechanisms of  $CO<sub>2</sub>$  absorption exist according to the type of the amino group. It induces different properties. The primary/secondary amines are useful for their  $CO<sub>2</sub>$  absorption kinetics [12] while the tertiary amines have a higher  $CO<sub>2</sub>$  absorption capacity and a lower reaction energy (promoting the regeneration) [13]. Diethylethanolamine (DEEA) is part of 7 reviewed mixtures containing 2 amines, as regeneration promoter (tertiary amine), such as in Liu F. et al. [14] or You J. et al. [15]. The summary of the different types of biphasic blends reported in the literature is presented in Table 1 with the chemicals composing the blends. The exact composition of the blends is available in Appendix A.

Table 1. Types of biphasic liquid-liquid blends



The demixing behavior can also be enhanced or even induced by the presence of a phase-splitter agent. Sulfolane is the most used molecule to achieve this goal with demixing blends. 7 different blends containing sulfolane have been reported in the literature (e.g. DETA – SULF [16]). Alcohols (namely, propan-1-ol and butan-1-ol) are also involved in the biphasic behavior of several solvents. The most common reported case is the replacement of a part of the water content by alcohol to cause the demixing phenomenon (e.g., MAE – BUT [17]). Some other phase-splitting agents like ethers have also been tested with amines (e.g., TEPA – DGME [18]). 2-amino-2-methyl-1-propanol (AMP) is a specific compound as it acts as a phase-splitting agent but also contains a sterically hindered amino group. This type of amine has specific properties and combines the fast kinetics of primary/secondary amine with a smaller regeneration energy (through the instability of the produced carbamate after the  $CO<sub>2</sub>$  absorption). In this literature review, AMP is part of two mixtures (already demixing without AMP) as enhancer of the demixing phenomenon (e.g., DMCA – MCA – AMP [19]).

# 3. Evaluation methodology

Some examples of evaluation process of non-demixing amine blends used for carbon capture have already been presented using various indicators such as the absorption capacity, the regeneration energy or the volatility of the solvents [20,21]. However, until now, no study has been published, specifically regarding the classification of demixing solvents. In this work, the developed methodology includes two steps: a techno-economic evaluation and a health, safety and environment (HSE) assessment. The first step classifies the 30 reviewed blends while the second step eliminates the dangerous mixtures from the previous classification.

# 3.1. Techno-economic evaluation

The techno-economic step is based on six indicators related to the solvent. The main indicator remains the regeneration energy (generally expressed by amount of produced  $CO<sub>2</sub>$ ) as it is the biggest part of the operative costs of such process. The reduction of this energy is the main goal of process or solvent improvements in such absorptionregeneration process. This amount of energy can be estimated by the sum of three contributions: the reaction enthalpy of the CO2 desorption, the energy used to heat the solvent until the regeneration temperature and the latent heat related to the vaporization of the solvent. For this last term, only the vaporization of water is generally considered. The advantage of a smaller flow going to the regeneration column (caused by the demixing phenomenon) is mostly visible in the reduction of the two last contributions. For the present methodology, the regeneration energy of each solvent is normalized by the regeneration energy of MEA 30 wt.% (3.99 GJ/tCO<sub>2</sub> [22] was used this method).

The  $CO<sub>2</sub>$  absorption capacity of the liquid solvent is also a key parameter to appreciate the performances of the solvents. It represents the concentration of carbon dioxide in the liquid phase at equilibrium for defined temperature and CO2 partial pressure conditions. It is therefore dependent on the structure of amine molecules and their concentrations in the solvent. A high absorption capacity is desired as it reduces the solvent flow needed in the absorption column for a fixed flue gas flow. For monoethanolamine, the stoichiometric absorption capacity is 0.5 mol CO2/mol amine due to the reactional mechanism for primary amines [23,24]. The corresponding values for MEA 30 wt.% (5 M) are 2.5 mol CO<sub>2</sub>/L or 2.46 mol CO<sub>2</sub>/kg [25] and are used to normalize the values of demixing solvents for the indicator in the methodology.

In addition to the thermodynamic aspects, the  $CO<sub>2</sub>$  absorption rate is also fundamental to evaluate the solvents properties. This has a particular importance for the estimation of the column's heights in the process. For the comparison, the particular challenge is that the  $CO<sub>2</sub>$  absorption rate can be measured through different parameters such as the overall mass transfer coefficient, the absorbed flux or the pseudo-first order constant of the absorption reaction. Some of them depend on the experimental device and conditions. Therefore, the different values reported in the literature for the different biphasic solvents are divided by the value of the corresponding parameter measured for MEA 30 wt.%. Kinetics are also dependent on the carbon dioxide concentration already present in the liquid. For this evaluation, the value for a clean solvent without absorbed  $CO<sub>2</sub>$  has been retained.

In the chosen methodology, the demixing phenomenon is characterized by the biphasic volume ratio or the volumetric flow rate ratio (for a continuous process). This equilibrium parameter is here defined as the volume ratio between the CO2-rich phase and the CO2-lean phase. The demixing ratio cannot be compared to the MEA (30 wt.%) solvent as this reference case does not present a demixing behavior. This indicator (mostly depending on temperature and CO2 concentration) should be as low as possible to reduce the required energy at the reboiler.

For the process design, the volatility of the solvent is also important as it will determine the required efficiency of a water-wash step of the treated flue gas after the CO<sub>2</sub> absorption. Indeed, the components of the solvent can be transferred to the gas phase. To quantify this tendency for each blend, the volatility of all organic compounds is simulated at atmospheric pressure and boiling temperature through the AspenPlus™ software (model NRTL-RK). These estimated values of the volatility of the organic fraction of the blends are normalized to the equivalent value for MEA 30 wt.%.

The sixth indicator is the solvent cost and is the only parameter uniquely linked to the economic aspect of the evaluation. Its place in this selection process is caused by the need of regular solvent replacement due to loss in the treated flue gas or degradation, especially through thermal or oxidative processes. This indicator has been calculated through the commercial prices of VWR™ provider while the water is considered to be costless. Even though the prices at the industrial level are certainly lower, the ranking between the different solvent costs could be considered quite similar.

The six indicators are reported in Table 2 with their units, values for MEA 30wt.% and best and worst values for the identified demixing solvents as well as the corresponding relative indicators and their respective weights. These weightings have been calculated using the Analytical Hierarchy Process (AHP) methodology [26]. The principle of this method is based on relative importances for each couple of indicators (from equal to very different relative importance). One of its main advantages is to assure the consistency of all these relative importances and therefore, a coherence between the resulting ponderations. For each indicator (RE, AC, AR, DR, OV and SC), a scoring scale is established from 1 (the best score) to 5 (the worst score). According to this scale, a rate is assigned to each reviewed solvent for each indicator. When the data are not available for one or more indicator(s), the corresponding score is attributed by analogy with another blend and using theoretical consideration (for regeneration energy, absorption capacity and absorption rate). For the demixing volume ratio, an unknown value involves a score of 5 as the precise value is impossible to estimate without experimental data. The final score of each solvent is calculated by the weighted sum of the six indicators. Therefore, the best solvents are those with the lowest final score.



Table 2. Key indicators of the techno-economic evaluation with their respective weights

#### 3.2. Health, safety and environment analysis

The health, safety and environment (HSE) analysis is based on the hazard statements of the pure chemical components of the blends. The data are taken from the database of the European Chemical Agency and concern two classifications (the harmonized classification and labelling from the CLP Regulation and the notifications according to the Registration, Evaluation, Authorization and restriction of Chemicals (REACH) legislation) [27,28]. The reported hazards statements are classified in three categories regarding health, physical and environmental risks. The base principle of this step is the rejection of blends containing at least one component having at least one serious hazard. Indeed, the risks inherent to a single component can have consequences for the whole process as well as during the storage of the chemicals or the preparation of the blends. Among the data available for the components of the reviewed solvents, seven hazard statements are considered as an exclusion reason in the present methodology. These are related to a mortal hazard (H330), an important hazard for reproduction (H360FD, H360Df, H360D), the corrosivity to metals (H290) and the high toxicity to aquatic life (H410).

# 4. Results and discussion

After the techno-economic step, the 30 reviewed solvents get a score from 1.49 for the best to 4.55 for the worst promising solvent. Twelve solvents obtain a score under the mean global value (score 3). Among them are six 2-amine blends and six blends with the presence of a phase-splitter agent. The scores after this first step of the evaluation methodology are reported in the last column of the table presented in Appendix A. The exclusion step according to the HSE evaluation concerns 17 blends which contain AEEA, SULF, DETA, DGME, MCA, EAE or DGM. There are 13 remaining blends classified from 1.49 to 4.14 with six global scores under 3 and are represented in Fig.2.

Among these mixtures, the four mixtures ranked from  $3<sup>rd</sup>$  to  $6<sup>th</sup>$  position all contain the tertiary amine DEEA (diethylethanolamine). Two of them are composed of various concentrations of DEEA and MAPA (positions 3 and 5). The blends in position 4 and 6 respectively contains MEA and DMBA. Nevertheless, the two most promising solvents according to this methodology are composed of triethylenetetramine (TETA) with respectively propan-1-ol (PROP) and N,N-dimethylcyclohexylamine (DMCA). The solvents positioned from 3<sup>rd</sup> to 5<sup>th</sup> place get relatively similar scores (between 2.38 and 2.43) while the most promising blends obtain significantly lower scores. With a rate of 1.91, the second most promising solvent contains, with the primary/secondary amine TETA (15 wt.%), a tertiary amine DMCA (35 wt.%). The association of these two categories of amines combines their respective advantages. For the blend in first position, propan-1-ol (50 wt.%) is used to induce the demixing phenomenon with TETA (30 wt.%). no-economic step, the 30 reviewed solvents get a score from 1.49 for the best to 4.55 for the worsend with the presence of a phase-splitter agent. The scores abe moments with the presence of a phase-splitter agent. The sc **SCESSORE 10**<br>
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Fig. 2. Classification of the blends resulting of the methodology

The final score for each solvent represented in Fig. 2 is a combination of the separate scores for the six indicators of the first step of the evaluation methodology. For the six most promising solvents, Fig. 3 shows the score for each techno-economic indicator. The lower the score is for each indicator, the better it is for the current evaluation method. This representation can be used to identify the strengths and weaknesses of a given solvent. For example, the biggest weakness of the TETA-PROP blend is the volatility of the organic compounds (mainly driven by the volatility of propan-1-ol) while the other indicators are good, or even excellent. On the contrary, the 6<sup>th</sup> position of the DEEA-DMBA blend (with a global score of 2.77) is only driven by its estimated low regeneration energy. The main weakness of the DEEA-MEA blend is the low availability of information concerning the demixing ratio which badly impacts the global score. For the three other blends (DEEA-MAPA and DMCA-TETA), some indicators show excellent results but the most obtain good or mean scores.



Fig. 3. Indicator scores for the six most promising solvents

### 5. Conclusion

The aim of this work was to classify the aqueous demixing solvents present in literature. 30 blends were identified as already studied at small scale. The first step of the evaluation based on six techno-economic indicators classifies the 30 solvents by giving each a global score from 1 to 5. The indicators were chosen by their potential impact on the process design and include regeneration energy, CO2 absorption rate, CO2 absorption capacity, demixing volume ratio, volatility of the organic compounds and solvent cost. The data related to each indicator were preferably found in literature. For the unavailable values, a score was still attributed by analogy with similar solvents or by application of theoretical rules. Even if the more accurate classification is done using experimental values, the estimation of the unknown technical properties allows to apply this method for solvents with few data (low technology readiness level). A potential further investigation could be to experimentally determine the missing values. Other properties (e.g., solvent degradation) were not included in this study due to missing information for most of the chemicals. A specific investigation of the solvent behavior under degradative conditions could be interesting to comfort or modify the present classification.

The second step excluded 17 solvents containing at least one chemical presenting a very serious hazard (such as death hazard or serious fertility problem). 13 aqueous blends remain from which the most promising one is composed of triethylenetetramine (30 wt.%) and propan-1-ol (50 wt.%). The high proportion of alcohol is the cause of the good biphasic properties as it reduces the  $CO<sub>2</sub>$ -rich phase. On the contrary, the main challenge for such a solvent is to deal with the high volatility of propan-1-ol. Such blends containing alcohol have been studied only for some years. Even if 2-amine blends are studied for longer, some of them present also good properties. Indeed, the four next solvents (classified from  $2<sup>nd</sup>$  to  $5<sup>th</sup>$  place) are composed of a tertiary amine (DMCA or DEEA) and a primary/secondary amine (TETA, MAPA or MEA). With the results of this study, the next developments regarding the implementation of aqueous biphasic liquid-liquid solvents should be the test of these solvents (especially, TETA – PROP) in a micropilot scale unit. Such experiments with large scale simulations could confirm the position of these solvents as one of the promising technologies to be used to tackle  $CO<sub>2</sub>$  emissions and limit climate change.

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# Appendix A. Reviewed solvents mixtures

The following table summarizes the 30 aqueous solvents mixtures reviewed in the literature with their respective mass fractions. Some blend compositions are directly presented in the scientific articles in mass fractions while the others are expressed in concentration units (e.g., mol/L). The corresponding mass fractions were calculated using the software Aspen Plus™ to homogenise units in the present table. The penultimate column of the table reports the global score of the solvents according to the techno-economic classification. The tick character in the last column signifies that the blend successfully passes the HSE analysis.



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