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# Influence of the amine alkyl-chain upon carbon dioxide absorption in G-L-L reactor

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**Abstract:** Present work analyzes the behavior of several gas-liquid-liquid systems for carbon dioxide separation using chemical absorption. The type of amine center in a solvent with two liquid phases (organic and aqueous) shows a high importance in relation to the overall behavior. The experimental results for these solvents have been compared with a previous study using octylamine in the chemical solvent because it has shown suitable results for carbon dioxide loading, absorption rate and energy cost associated to solvent regeneration. Present study analyzes the carbon dioxide absorption mechanism in multiphase reactors (specifically G-L-L) using different types of amines in order to understand the overall behavior that involves steps of mass transfer and chemical reaction.

**Keywords:** absorption; carbon dioxide; gas-liquid-liquid; separation.

## 1 Introduction

In the last years research studies in the field of carbon dioxide separation, once for purification (e.g., hydrogen production), once for carbon dioxide capture, have been centered on energetic optimization (Mores, Scenna, and Mussati 2012; Wang et al. 2017) and specifically in the reduction of the energy penalty associated to solvent regeneration. This aim is based on the conclusions of previous studies (Feng et al. 2010) that indicate the large cost of solvent regeneration operation (e.g., 70% of the overall cost of the process). In addition it is well-known that carbon dioxide is one of the most important

greenhouse gases that cause de global warming and the different adverse associated effects (Rahimi et al. 2019). In order to reach these aims for carbon dioxide separation several techniques can be used (Rahimi, Riahi, and Abbasi 2020).

In order to reduce the energetic cost of the process, several modifications in the conventional process have been proposed such as changes in the formulation of the chemical solvent using other type of molecules (Azghan, Farsi, and Eslamloueyan 2016; Heldebrant et al. 2011; Zhang et al. 2015), the use of reagents blends (Ling et al. 2019; Xu et al. 2019), improving the regeneration process (Gantert and Möller 2012; Schach et al. 2010), etc. For these reasons the development of low energy solvents that maintain suitable characteristics in separation operation (absorption rate and selectivity) has increased, in order to obtain a new generation of solvents. This third generation of chemical solvents is based on the use of organic solvents in order to replace partial or completely the presence of water (Heldebrant et al. 2011).

During last year more complex solvents that involves a third phase in the absorber have been developed at lab scale in order to contribute improvement in the overall process: in relation with economical issues related with heat transfer processes and solvent regeneration section (Lin and Wong 2014). Recent studies (Kossmann et al. 2015) have evaluated the viability of these solvents (including a second non miscible liquid phase) taking into account an energetic and economical point of view. The reached conclusions were not completely satisfactory because these solvent do not overcome the conventional technology. This proposal implies to feed all solvent flow-rate to stripper. This type of configuration can have some positive issues as the high carbon dioxide physical solubility in the organic phase that can produce a shuttle effect in the overall absorption rate (Carrera, Gómez-Díaz, and Navaza 2018) but the energy required by the reboiler and heat exchangers is high. The positive effects do not allow to overcome the negative ones.

For these reasons phase changing solvents are been taken into account because they can contribute significant improvements. One of positive effects of this type of solvents is centered on the preferential concentration of reaction products in one of the liquid phases present in the

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absorbed. This second phase can be present in the absorber or can be created during the process (a liquid or solid phase) (Zhang et al. 2019). This type of technology can contribute some positive issues by the presence of a CO<sub>2</sub>-enriched liquid phase such as a reduction in the amount of sensible heat and water vaporization and in general a decrease in all heat use elements (Barzagli, Mani, and Peruzzini 2017; Wang et al. 2018). The experimental results using this type of technology (Ye, Wang, and Lu 2015) with a wide number of solvents have allowed to conclude that in some cases the results are better than the conventional operation but the knowledge must be improve.

Taking into account this starting point, present research work proposes to carry out the study of several non-soluble amines in order to generate a gas-liquid-liquid system to take advantage of the special characteristics of these solvents and to analyze the role of the different radicals in the amine molecule.

## 2 Materials and methods

### 2.1 Materials

Carbon dioxide was supplied by Praxair with a purity of 99.998% and used as gas phase in present work. Dioctylamine (DOA) and octadecylamine (ODA) were supplied by Alfa Aesar with purities of >96% and >97%, respectively and were employed in the organic phase of the gas-liquid-liquid system for carbon dioxide removal. The organic phase was prepared using several organic solvents (cyclohexane, heptane and dodecane) supplied by Alfa Aesar with purities >99%. The gas-liquid-liquid system was completed with double distilled water Figure 1.

### 2.2 Absorption studies

The experiments for carbon dioxide absorption in gas-liquid-liquid contactor were carried out using a bubbling and cylindrical stirred tank contactor with a volume of 0.5 L. Figure 2 shows a scheme of the experimental set-up. The total liquid phase volume used in absorption experiments was 0.25 L. The tank characteristics are: 22 cm of height and 6 cm of diameter. The gas phase was fed in the contactor using a sintered glass sparger that allows to increase gas-liquid interfacial area. A magnetic stirrer was employed to maintain a suitable

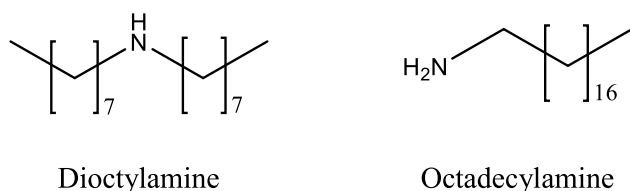


Figure 1: Chemical structure of amines used in present work.

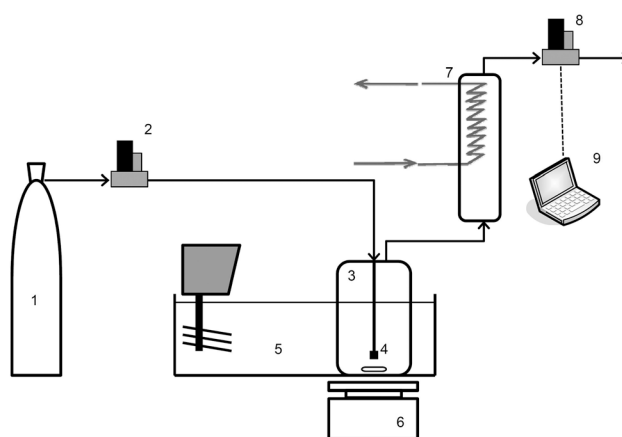


Figure 2: Scheme of experimental set-up used for carbon dioxide absorption experiments. (1) Gas cylinder; (2) Mass flow-controller; (3) Gas-liquid contactor; (4) Gas sparger; (5) Thermostatic bath; (6) Magnetic stirrer; (7) Condenser; (8) Mass flowmeter; (9) Data recorder.

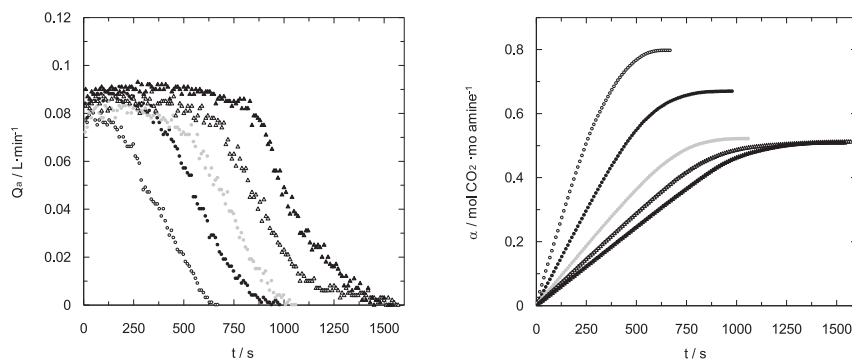
dispersion of both liquid phases. Absorption experiments were carried out at 20 °C using a Frigiterm (Selecta) thermostat. The solvent was introduced in the vessel and stirring until the target temperature was reached. Carbon dioxide absorption rate was determined on the basis of the difference between inlet and outlet mass flow-rates (Alicat Scientific MC-5SLMP-D and Alicat Scientific M-5SLMP-D). These devices were calibrated for the gas flow-rate and pressure employed ranges by the supplier. The working regime was continuous in relation to the gas phase and batch regarding the liquid solvent.

### 2.3 Regeneration studies

The experimental technique for solvent regeneration consists of the use of a three-necked flask (volume of 0.5 L), a double condenser, a heating mantle (Selecta Fibroman) and a temperature sensor (Selecta Sensoterm). The solvent was boiled during 15 min and after this thermal treatment, a new absorption experiment was carried out in order to evaluate the behavior of this type of solvent. Five absorption/regeneration cycles were carried out.

## 3 Results and discussion

The first part of solvent characterization studies in present work is centered on the analysis of carbon dioxide absorption using DOA and ODA with different concentrations. The ratio between organic and aqueous phases is maintained at 80/20 respectively (20% of water). Cyclohexane is used as organic solvent. The experimental results using DOA (a secondary amine) for absorption curves and carbon dioxide loading are shown in plots included in Figure 3. The increase in amine concentration causes similar behaviors for these two parameters than using homogeneous solvents: slight increase in absorption rate



**Figure 3:** Influence of DOA concentration upon absorption rate and carbon dioxide loading. (○)  $C_{DOA} = 0.1 \text{ mol L}^{-1}$ ; (●)  $C_{DOA} = 0.2 \text{ mol L}^{-1}$ ; (◐)  $C_{DOA} = 0.3 \text{ mol L}^{-1}$ ; (◑)  $C_{DOA} = 0.4 \text{ mol L}^{-1}$ ; (◒)  $C_{DOA} = 0.5 \text{ mol L}^{-1}$ .  $C_{H_2O} = 20\%$ .  $Q_G = 0.1 \text{ L min}^{-1}$ .  $T = 293 \text{ K}$ .

and a higher time to reach solvent saturation. A decrease in carbon dioxide loading is observed until reaching a constant value. The increase in absorption rate is produced because a higher amount of amine is available and it increases reaction rate. The absorption rate increases slowly due to other parameters such as gas-liquid interfacial area and gas-liquid and liquid-liquid mass transfer coefficients that limit the overall rate.

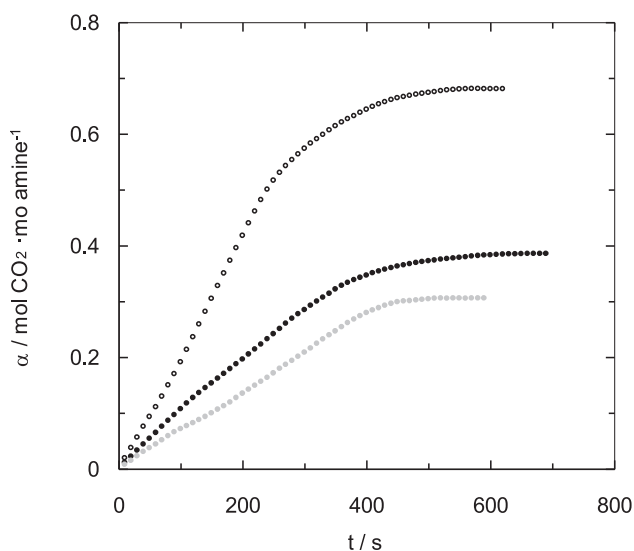
The decrease in carbon dioxide loading is a typical behavior for chemical absorption processes. When low amine concentrations are used, the weight of carbon dioxide physically absorbed is high and it reaches higher values for loading. DOA concentrations higher than  $0.3 \text{ mol L}^{-1}$  shows a constant final value for carbon dioxide loading corresponding to chemical absorption. The value of this parameter reaches a value close to  $0.5 \text{ mol CO}_2 \text{ mol amine}^{-1}$  that is in agreement with the reaction mechanism for this type of amines (secondary type) and the fact of the reaction takes place in the organic phase: carbamate production.

In order to analyze the absorption behavior it is necessary to take into account that DOA is a secondary amine with a high degree of sterical hindering. On the basis of a previous article (Bougie and Iliuta 2010), the presence of an important amount of bicarbonate ion in the reaction products is probable. This fact is produced due to the low stability of carbamate that tends to suffer hydrolysis. This type of reaction mechanism produces in general value of carbon dioxide loading close to  $1 \text{ mol CO}_2 \text{ mol amine}^{-1}$ . Figure 3 shows values far than expected ones. The experimental values of carbon dioxide loading tend to reach values close to  $0.5 \text{ mol CO}_2 \text{ mol amine}^{-1}$  that is compatible with the presence of carbamate as the unique reaction product avoiding the formation of bicarbonate.

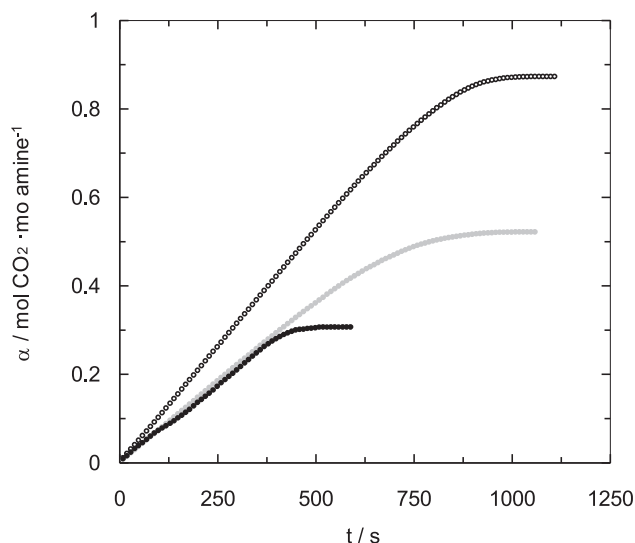
ODA is also used as reagent in the chemical solvent. This amine in comparison with OA (octylamine) maintains the same amino center with a larger chain length. The absorption curves show important differences in comparison with the secondary amine (DOA) previously analyzed.

Figure 4 shows also a resume of carbon dioxide loading experimental data. Only chemical solvents with ODA concentration lower than  $0.3 \text{ mol L}^{-1}$  are used because high concentrations show large deviation from homogeneous bubbling flow pattern. Carbon dioxide loading shows an important decrease when ODA concentration increases in the organic phase of the solvent. DOA solvents also showed a decrease of carbon dioxide loading with concentration related with the importance of carbon dioxide physical absorption in diluted solutions and in agreement with previous studies (Bonenfant, Mimeault, and Hausler 2005) and. The main difference when ODA solvent is the magnitude of carbon dioxide loading because it reaches values lower than  $0.5 \text{ mol CO}_2 \text{ mol amine}^{-1}$  that is an anomalous behavior this type of solvents.

When OA is used in this type of heterogeneous solvents, the proposed hypothesis for the reaction mechanism is centered on the carbamate formation in the organic



**Figure 4:** Influence of ODA concentration upon absorption rate and carbon dioxide loading. (○)  $C_{ODA} = 0.1 \text{ mol L}^{-1}$ ; (●)  $C_{ODA} = 0.2 \text{ mol L}^{-1}$ ; (◐)  $C_{ODA} = 0.3 \text{ mol L}^{-1}$ .  $C_{H_2O} = 20\%$ .  $Q_G = 0.1 \text{ L min}^{-1}$ .  $T = 293 \text{ K}$ .



**Figure 5:** Influence of amine substituent type upon carbon dioxide loading. (O) OA (Brea et al. 2019); (●) DOA; (●) ODA.

$C_B = 0.3 \text{ mol L}^{-1}$ ,  $C_{H_2O} = 20\%$ ,  $Q_G = 0.1 \text{ L min}^{-1}$ ,  $T = 293 \text{ K}$ .

phase and the hydrolysis of this compound in the aqueous phase that allow to increase carbon dioxide loading. The previous work of our research team (Brea et al. 2019) confirmed that this reaction mechanism takes place and the carbamate hydrolysis was observed in the organic/water interface but in the interface of water droplets in the continuous phase. By NMR studies was confirmed the formation of a water-in-oil microemulsion. These water droplets are stabilized by the carbamate and protonated amine molecules produced in the organic phase during the carbon dioxide chemical absorption. Figure 5 shows a comparison between the absorption curves of OA with amines used in present work.

When DOA is used in the solvent the formation of microemulsions during chemical absorption is not observed and an important turbidity was produced. This fact avoids the increase of liquid-liquid interfacial area reducing the possibilities of carbamate hydrolysis. These conclusions are in agreement with the previously presented experimental data for carbon dioxide loading. On these bases it is possible to conclude that carbon dioxide chemical absorption by DOA is carried out in the organic phase with the production of carbamate and protonated amine as unique reaction products.

The experimental results using ODA as reagent for carbon dioxide chemical absorption show (see Figures 4 and 5) a higher reduction in carbon dioxide loading reaching values lower than  $0.5 \text{ mol CO}_2 \text{ mol amine}^{-1}$  (specifically values close to  $0.3 \text{ mol CO}_2 \text{ mol amine}^{-1}$ ). These results allow to think that chemical equilibrium does not allow to increase the conversion in the reaction between

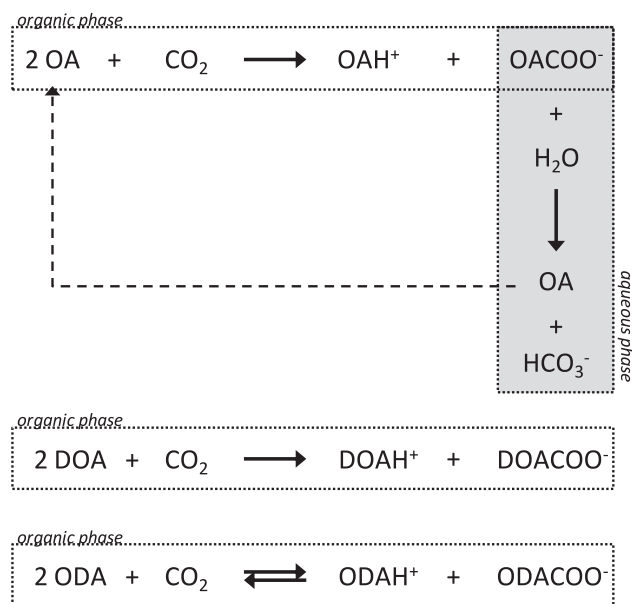
carbon dioxide and ODA. This type of limitation reduces the interest of this non-soluble amine to be used in gas-liquid-liquid absorbers.

On the basis of experimental results using gas-liquid-liquid contactor with DOA (two alkyl radical with eight carbons) and ODA (one alkyl radical with 18 carbons) in comparison with the solvent based on OA (one alkyl radical with eight carbons) indicate that the presence of a higher number of radicals (DOA) or a larger chain length (ODA) reduce or avoid the action of water molecules (hydrolyzation) upon carbamate previously produced in the organic phase by the reaction between carbon dioxide and amines. This fact causes the stabilization of carbamate and it does not allow to increase carbon dioxide loading. In the case of ODA-based solvents the high chain length causes a very important steric hindrance that increases the difficult for the reaction between carbon dioxide and nitrogen group of amine (Benítez et al. 2011).

On the basis of experimental results obtained in present work for heterogeneous solvents with DOA and ODA in comparison with OA-based solvents, the proposed reaction mechanisms of each amine are shown in Figure 6.

Taking into account the negative characteristics of ODA-based solvents both in chemical reaction extension and undesirable heterogeneous flow pattern, this amine has not been considered in the other studies included in present work.

The use of heterogeneous solvents tries to take advantage of certain characteristics of each liquid phase and then the effect of liquid phases' ratio upon carbon

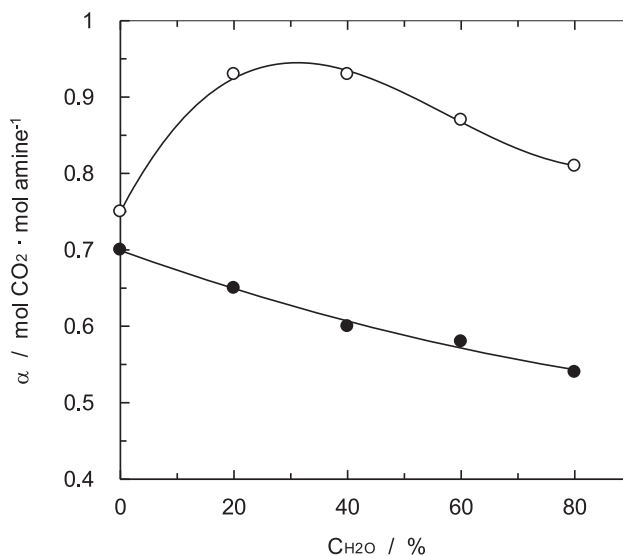


**Figure 6:** Proposed reaction mechanisms for the different amines in GLL system.

dioxide absorption rate and carbon dioxide loading is an important study. Figure 7 shows an example of experimental data for carbon dioxide loading along time using different aqueous/organic phases ratio. An increase in the amount of water causes a decrease in the absorption rate (slope at the beginning).

Figure 8 shows clearly the influence of water presence in the gas-liquid-liquid reactor upon carbon dioxide loading for DOA and OA based solvents. The observed behavior using OA showed a maximum value between 20 and 40% of water. The use of DOA shows a different behavior. A monotonic decrease in the carbon dioxide loading is observed when water presence increases in the reactor. This behavior for DOA-based solvents confirms that water does not hydrolyze the carbamate and it reduces the importance of water in the overall process. This fact is in agreement with the proposal of reaction mechanism in Figure 6. On the other hand the decrease in carbon dioxide loading observed in Figure 8 is assigned to a decrease in carbon dioxide physical solubility because it is lower for water in comparison with the organic solvent (Gómez-Díaz and Navaza 2008).

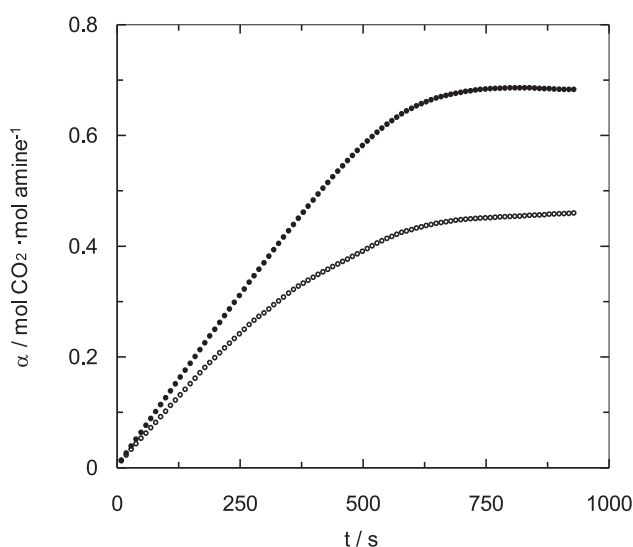
Present research work analyses the influence of gas flow-rate fed to the contactor with the aim of analyzing the effects caused upon absorption rate and loading (see Figure 9). Carbon dioxide absorption rate increases with gas flow-rate. This behavior is based on hydrodynamic issues and mainly in relation with the increase in gas-liquid interfacial area. This increase in gas-liquid interfacial area is due to the presence of a larger volume of gas that



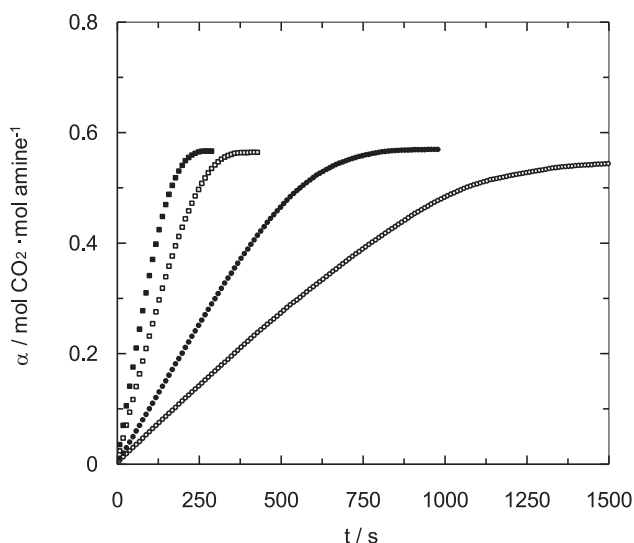
**Figure 8:** Influence of water presence upon carbon dioxide loading. (O) OA (Brea et al. 2019); (●) DOA.  $C_B = 0.2 \text{ mol L}^{-1}$ ,  $Q_G = 0.1 \text{ L min}^{-1}$ ,  $T = 293 \text{ K}$ .

increases significantly the number of bubbles and then the gas hold-up. On the other hand there is non-influence of this variable upon carbon dioxide loading that confirms the absence of relation between gas flow-rate and reaction mechanism.

After the analysis of the role of water in the heterogeneous solvent, type and concentration of the amine employed in carbon dioxide chemical absorption, other study centered on the organic phase has been carried out:

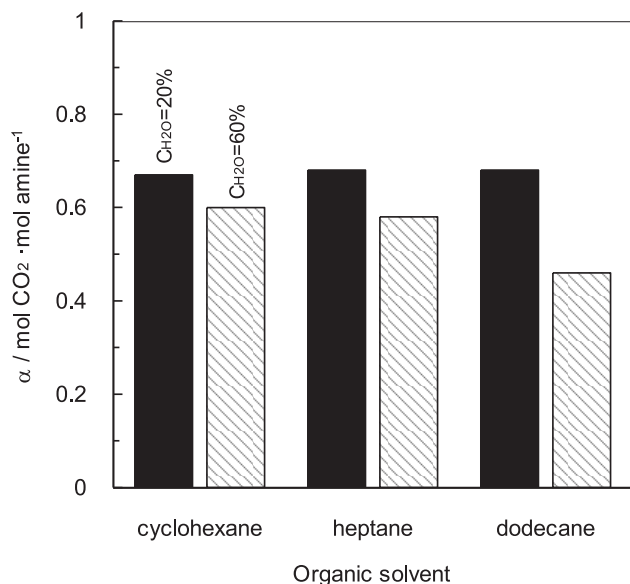


**Figure 7:** Influence of water content upon carbon dioxide loading using DOA-based solvent.  $C_B = 0.2 \text{ mol L}^{-1}$ ,  $Q_G = 0.1 \text{ L min}^{-1}$ , (●)  $C_{H_2O} = 20\%$ ; (O)  $C_{H_2O} = 60\%$ .  $T = 293 \text{ K}$ .



**Figure 9:** Influence of gas flow-rate upon carbon dioxide loading using DOA-based solvent.  $C_B = 0.3 \text{ M}$ ,  $C_{H_2O} = 20\%$ . (O)  $Q_G = 0.05 \text{ L min}^{-1}$ ; (●)  $Q_G = 0.1 \text{ L min}^{-1}$ ; (□)  $Q_G = 0.2 \text{ L min}^{-1}$ ; (■)  $Q_G = 0.3 \text{ L min}^{-1}$ .  $T = 293 \text{ K}$ .

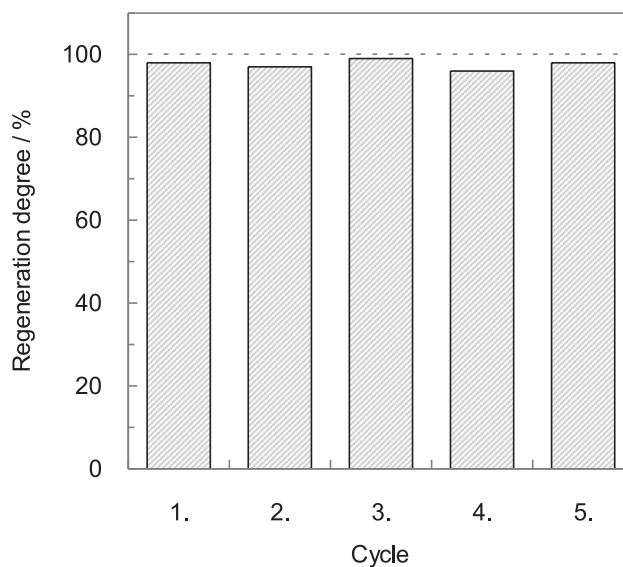




**Figure 10:** Influence of the type of organic solvent and organic/water ratio upon carbon dioxide loading.  $C_B = 0.2$  M.  $Q_G = 0.1$  L min<sup>-1</sup>.  $T = 293$  K.

the effect of type of organic solvent upon the chemical absorption process. The change of continuous phase can produce modifications in the reaction pathway and the weight of each reaction involved in the overall process. Figure 10 shows examples of the experimental data corresponding to this study. The carbon dioxide loading is used to compare the solvents with different solvents. Figure 10 shows a lack of influence of the type of organic phase when a 20% of water was employed in the multiphasic reactor. A similar value close to 0.7 mol CO<sub>2</sub> mol amine<sup>-1</sup> is obtained for all the organic compounds employed as organic phase. Previous studies have concluded that the change in the type of solvent can produce important changes in the pK<sub>a</sub> that is related with the reactivity between carbon dioxide and amines (Um, Lee, and Jeon 2002). On the basis of experimental data the nature of organic solvents does not show an important effect upon the chemical absorption.

When a large amount of water is employed (60% of water) a decrease in carbon dioxide loading is always observed, but the degree of this reduction is different depending on the organic phase employed in the chemical solvent. The reduction in chemical absorption is higher when dodecane is used as organic phase while the use of heptane and cyclohexane show similar values. Taking into account the non-influence of organic solvent upon the reactivity (when 20% of aqueous phase is used), the different behavior observed when 60% of water is employed could be related with differences in the internal dynamics in order to generate microemulsions or other



**Figure 11:** Regeneration degree of DOA-based solvent.  $C_B = 0.5$  M.  $C_{H_2O} = 20\%$ .  $Q_G = 0.1$  L min<sup>-1</sup>.

type of aggregates that reduce the presence of reaction products in the medium (organic phase) (Brea et al. 2019).

The last study in present work is centered on the evaluation of DOA-based solvent during the regeneration operation by stripping. In general a decrease in the carbon dioxide loading is observed when a previously loaded solvent is regenerated (Li et al. 2012). Figure 11 shows the experimental results obtained for this chemical biphasic solvent. These data indicate a suitable behavior of this solvent because the regeneration degree (defined as the carbon dioxide loading reached in relation with the value obtained for fresh solvent) maintains a constant value and similar than the value for fresh solvent.

## 4 Conclusions

The modification of number or length of radicals in the amino center of reagent for carbon dioxide chemical absorption causes significant modifications in the behavior of gas-liquid-liquid absorbers related with the reaction mechanism (inhibition of carbamate hydrolysis).

A water-in-oil microemulsion is not produced and it decreases liquid-liquid interfacial area and it tends to reduce mass transfer rate between both liquid solvents.

An increase in aqueous/organic phases ratio does not allow increase carbamate hydrolysis and shows negative effects upon absorption rate and carbon dioxide loading.

The effect of organic solvent type shows non influence upon carbon dioxide absorption with low aqueous/organic ratio but an increase in this ratio shows better results for

cyclohexane in comparison with the other organic solvents.

The DOA-based solvents shows a stable behavior during absorption-regeneration cycles without a lost in carbon dioxide absorption rate and loading.

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