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Determination of kinetics of CO_2 absorption in unloaded and loaded DEEA+MAPA blend

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Abstract

Alkanolamine blends are of high interest as solvents to enhance the CO_2 capture technology in comparison to the traditional 5M MEA. In the present work, the mass transfer and kinetics coefficients of the unloaded and loaded blend 3 M DEEA+ 2 M MAPA are measured from 30 to 70ºC in a Double Stirred Cell (DSC) under pseudo-first order conditions. Needed physical properties, as density, viscosity and Henry's Law constants of N_2O are measured from 25 to 80ºC. Results are extracted using the zwitterion mechanism and two-film theory.

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Keywords: CO_2 capture; Chemical Absorption; DEEA; MAPA; kinetics; mass transfer

1. Introduction

The solvent-based post-combustion capture is considered the most mature technology available at small industrial scale. It is possible to retrofit traditional power plants, making it one of the most viable choices for Carbon Capture [1].

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Aqueous primary amines are shown as the main solvents for CO_2 absorption. They form stable carbamate at a high reaction rate. Especially, MEA has been widely used due to its low volatility, thermal stability and high absorption rate. Nevertheless, both process improvements and alternative solvents for MEA are widely studied due to are to the high cost of solvent regeneration and corrosion at high concentrations. . Tertiary amines have appeared as an alternative option because a reduced amount of energy is needed for its regeneration [2]. However, the absorption rates are lower and promoters are needed. Thus, use of amine blends is one, widely studied way to minimize the energy requirements in the process maintaining and acceptable absorption rate [3]–[6].

N,N-diethylethanolamine (DEEA) appeared as one of the amines with the lowest amount of energy needed for its regeneration [7]. DEEA can potentially absorb CO_2 up to 1 mol CO_2 per mole DEEA in presence of enough pressure of $CO_2$and is obtained from renewable sources [8]. In order to increase the absorption rate, previous works have studied the use of N-methyl-1,3-propane-diamine (MAPA) as promoter of DEEA solutions [7]–[10].

The study of kinetics is indispensable to compare novel solvents and design the absorption and desorption columns. Values of rate constants can be extracted experimentally with different apparatus that take into account the gas-liquid mass transfer and reaction in the liquid phase, as previously reported with wetted wall columns [11], string of discs ([12]–[14]) or stirred cells [15]. Another option available is the mixing method of Hartridge and Roughton, which excludes the mass transfer influence [16].

Monteiro et al. [13] studied the CO_2 absorption in several unloaded DEEA+MAPA solutions. In her work, all the blends formed two phases and loaded solutions were not measured. Recently, Ciftja et al [9] showed a list of blends that proved to be one phase solutions. One of them, 3M DEEA + 2M MAPA showed good results and it has been selected for this work. Mass transfer and kinetic constants from 30 to 80ºC are measured using a double stirred cell (DSC). In this work, measurement with with 3M DEEA +2M MAPA, was performed from 30 to 80ºC. Furthermore, needed densities, viscosities and N_2O Henry's Law constants are measured from 25 to 80ºC.

Nomenclature

AARD	Average Absolute Relative Deviation
DEEA	N,N-diethylethanolamine
DSC	Double Stirred Cell
Ha	Hatta number
MAPA	N-methyl-1,3-propane-diamine
MEA	Monoethanolamine
R_{CO2}	Absorption Rate of CO_2
VLE	Vapour-Liquid Equilibrium
α	CO_2 loading (mol CO_2/mol amine group)
α'	CO_2 loading (mol CO_2/mol primary amine)
ρ_l	Density of loaded 3M DEEA+ 2M MAPA solutions
ρ_u	Density of unloaded 3M DEEA+ 2M MAPA solutions

2. Experimental apparatus and procedure

Densities of unloaded and loaded solutions were measured from 25 to 80ºC using an Anton Paar DMA 4500 M densitometer. The apparatus uses a Xsampler 452 H heating attachment to control the temperature with temperature variability of 0.01 ºC. More detailed description of the equipment and method can be found elsewere [17]- [18]. Two measurements were done for each sample and several samples of water were placed in between the samples measured in this work, obtaining an AARD for water of 0.15%.

An Anton Paar MCR 100 rheometer with a double gap measuring cell (DG-26.7) was used to measure the dynamic viscosity in this work. The setup and the method can be found elsewhere [19].

The N_2O Henry's law constants were measured from 30 to 80ºC. The procedure has been reported previously by [18], [20].

The reactive absorption with unloaded 3 M DEEA + 2M MAPA was carried out in a Double Stirred Cell (DSC) from 30 to 70ºC. The method used in this work was described previously by Jiru and Eimer [21]. Loaded solutions of 3M DEEA + 2 M MAPA were also studied using the DSC from 30 to 70ºC. For these experiences, multiple injections were done in a series basis. Loadings were measured by titration before the first injection and after the last one. Intermediate loadings were calculated based on the Peng-Robinson equation of state. The average absolute difference between calculated final loading and the measured one by titration was 0.01 mol CO_2/mol amine group. The apparatus was initially validated with 5M MEA [22] and a good agreement with literature data was found [23].

3. Method for determination mass transfer and kinetic constants

MAPA is a primary amine that reacts directly with CO_2. Zwitterion mechanism, based on the theory developed by Danckwerts,[24] was used. . The zwitterion reaches a pseudo-equilibrium condition and the reaction rate can be described using equations R1-R2. DEEA, however, cannot react with CO_2 at pH below 13. Instead it promotes the hydrolysis (R3) . The other reactions taking place in the media are given by R4-R6.

$$MAPA + CO_2 \overset{k_2,k_{-2}}{\longleftrightarrow} MAPA^+COO^- \quad \text{(R1)}$$

$$MAPA^+COO^- + B \overset{k_5}{\rightarrow} MAPACOO^- + B^+ \quad \text{(R2)}$$

$$DEEAH^+ + H_2O \leftrightarrow DEEA + H_3O^+ \quad \text{(R3)}$$

$$2H_2O \leftrightarrow H_3O^+ + OH^- \quad \text{(R4)}$$

$$2H_2O + CO_2 \leftrightarrow H_3O^+ + HCO_3^- \quad \text{(R5)}$$

$$H_2O + HCO_3^- \leftrightarrow H_3O^+ + CO_3^{2-} \quad \text{(R6)}$$

Because the reactions R3, R5 and R6 are much slower than R1 and R2, it can be assumed that the absorption of CO_2 would mainly depend on the reaction of CO_2 with MAPA. The formation of the primary and secondary carbamate with MAPA would drive the absorption , as confirmed previously for this system [9].Equation 1 can be used to describe the absorption of CO_2 in the 3 M DEEA + 2M MAPA solution.

$$r_{CO_2} = \frac{k_2[CO_2][MAPA]}{1+\frac{k_{-2}}{k_5[B]}} \quad (1)$$

Since that the reverse in equation R1 is much slower than the loss of the proton of the zwitterion (equation 1), $\frac{k_{-2}}{k_5[B]}$ is considerable smaller than 1 and can be negligible (Equation 1) giving as result the Equation 2.

$$r_{CO_2} = k_2[MAPA]^n[CO_2] = k_{obs}[CO_2] \quad (2)$$

In this work, the zwitterion mechanism was considered together with the two-film theory. The method for the determination of the observed kinetic constant, k_{obs}, is described in the work of Blauwhoff et al. [25] and is included in Equation 3. The calculation is based on the evolution of the partial pressure of CO_2 (Equation 4) and considering that the experiments are carried out under pseudo-first order conditions. Hatta number (Ha) (equation 5) and infinite enhancement factor (E_{inf}) were calculated for each experimental condition to ensure that the experiments were in the

pseudo-first order region (Ha>>5, E_{inf}>>Ha). The diffusivity of the solvent in the solution has been calculated by the theory of Glasscock [6] and the E_{inf} was extracted from the correlation from Van Swaaij and Versteeg [3].

$$\ln P = -\frac{RTA}{V_G H}\sqrt{k_{obs} D}\,t + \ln p_0 \tag{3}$$

$$k_{obs} = \left(\frac{-\frac{d\ln P_{CO_2}}{dt} H V_G}{RTA}\right)^2 \frac{1}{D} \tag{4}$$

$$Ha = \frac{\sqrt{k_{obs} D}}{k_L^0} \tag{5}$$

Based on the two-film theory (Equation 6), the overall mass transfer coefficient K_G can be expressed as the summation of both resistances, from the gas phase (kg) and from the liquid phase (k_L). Moreover, considering that the fast reaction is only taking place in the reaction layer and that there is not gas resistance due to the use of pure CO_2, the k'_G can also be calculated based on the assumption of pseudo-first order regime (Equation 7).

$$\frac{1}{K_G} = \frac{1}{k_g} + \frac{H_{CO_2}}{E\,k_L^0} = \frac{1}{k_g} + \frac{1}{k'_G} \tag{6}$$

$$k'_G = \frac{\sqrt{k_{obs} D}}{H} = \frac{-\frac{d\ln P_{CO_2}}{dt} V_G}{RTA} \tag{7}$$

The partial pressure CO_2 can affect the mass transfer [26]. In this work, in order to obtain comparable results, all the experiments were carried out at similar initial partial pressure of CO_2. Furthermore, the used partial pressure is similar to those of used in the literature ([27], [26], [28], [29],[30], [31]). he objective was to inject enough pure CO_2 to be able to monitor the decrease of the normal logarithm of P_{CO_2} with the time, but low enough so that short injection time (4 seconds) could be used. Additionally, this also ensures that the loading of the solution will be small, as seen by checking with titration. The absorption rate can be calculated from the mass transfer coefficient k'_G and the partial pressure of CO_2 (Equation 8).

$$R_{CO_2} = k_G \cdot P_{CO_2} \tag{8}$$

For the calculation of diffusivity of CO_2 in the solutions studied, the N_2O analogy was used (equation 9). The viscosity of water was extracted from the work of Korson et al. [32], while the viscosities of the amine solutions were measured in this work. The diffusivity of CO_2 in water was interpolated from Versteeg and Swaajj [33]. The diffusivity of CO_2 in 5M MEA solutions found in the literature [34] was used in this work.

For the CO_2 solubility in unloaded and loaded 3M DEEA+ 2M MAPA solutions, the N_2O analogy included in the work of Versteeg and Swaaij [33] was used (equation 10) together with their R_H correlation. The solubility of N_2O in the solutions were measured in this work. The CO_2 solubility in 5M MEA solutions were extracted from literature [15].

$$D_{Co2-solution} = D_{Co2-water}\left(\frac{\mu_{water}}{\mu_{solution}}\right)^{0.8} \tag{9}$$

$$H_{CO_2-sol} = \frac{H_{N_2O-sol}}{R_H} \quad (10)$$

4. Results and discussion

4.1 Viscosity, density and Henry's law constant

Viscosities of unloaded and loaded solutions of 3M DEEA + 2M MAPA were measured from 25 to 80ºC (Figure 1). The standard S60 was used to validate the equipment, at 40 and 80ºC, obtaining an AARD of 0.8 and 0.75% respectively. Results of measured viscosities of unloaded solutions of 3M DEEA + 2M MAPA show a good agreement with the literature [9]. As seen in the Figure 1, the viscosity increases with the loading and decrease with the temperature.

Densities of unloaded and loaded solutions of 3M DEEA + 2M MAPA were measured from 25 to 80ºC (Figure 1). In addition to the validation with water, mentioned previously, the results of the unloaded solution are in agreement with the literature [9] as seen from Figure 1, The densities as function of loading and temperature was done modelled using Equations 11 and 12. The model shows a good agreement with the experimental values obtained in this work, with an AARD of 0.6%. As observed in the Figure 1, the density decreases with increases on the temperature and increases with increases on the CO_2 loading.

$$\rho_l = \rho_u + T^{1.02}(0.0009\alpha + 0.00001) \quad (11)$$

$$\rho_u = 1.2267 - 0.0009T \quad (12)$$

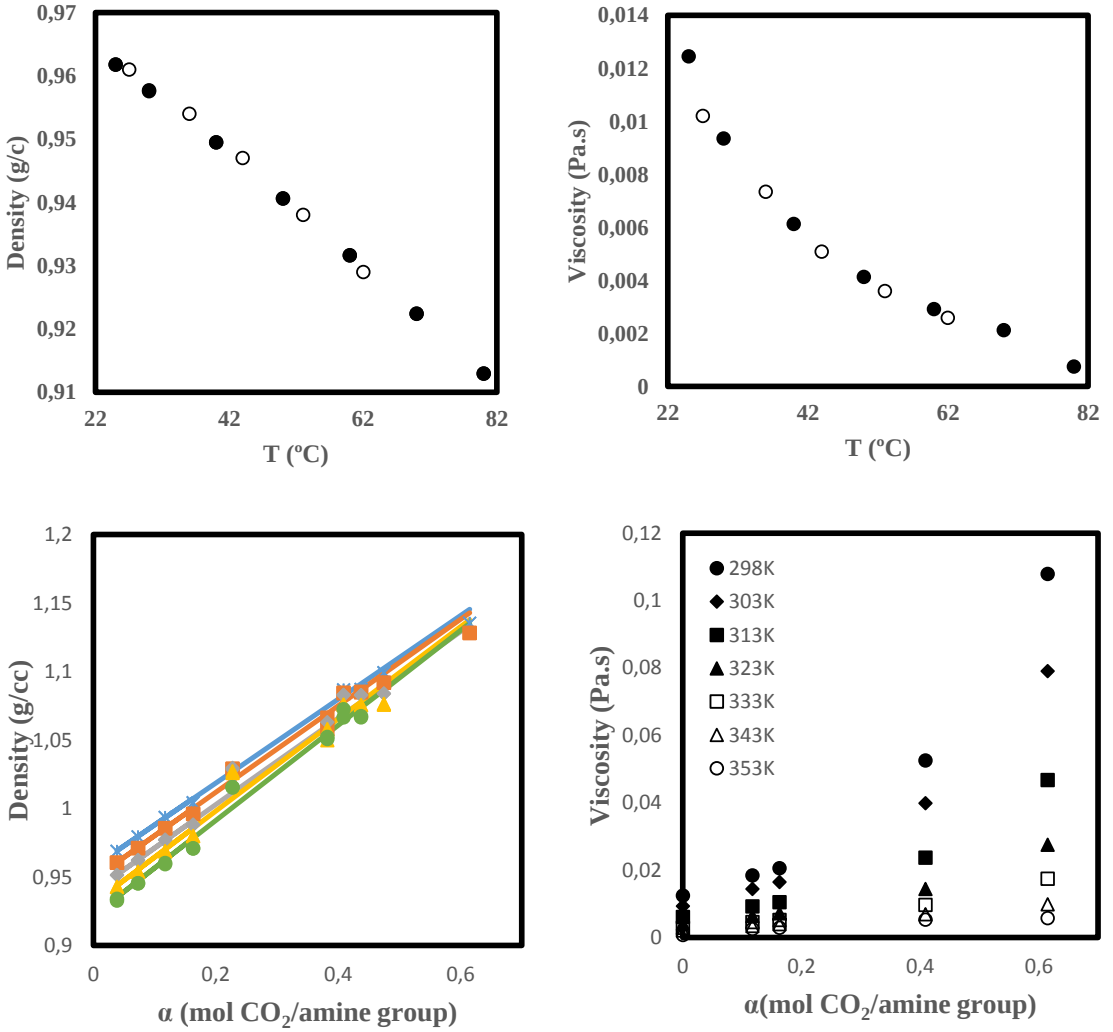

Figure 1(Top, left) Density of unloaded 3M DEEA + 2M MAPA measured in this work (filled black circles) and from Monteiro et al. (empty circles)[9]; (Top, right) Viscosity of unloaded 3M DEEA + 2M MAPA measured in this work (filled black circles) and from Monteiro et al. (empty circles)[9] (Bottom, left) Density of loaded 3M DEEA + 2M MAPA measured in this work, from 30 to 80 ºC, and mathematical model (lines). Colours: blue (30ºC), orange (40ºC), grey (50ºC), yellow (60ºC) and green (70C); (Bottom. right) Viscosity of 3M DEEA + 2M MAPA measured in this work, from 25 to 80ºC.

The unloaded solution of 3M DEEA+ 2M MAPA was used to ensure correct operation of the N_2O solubility apparatus used in this work. As observed in Figure 2, the results agree with the literature [9]. The N_2O solubility of loaded

solutions was measured from 30 to 80ºC, considering loadings up to 1.76 (mol CO_2/mol MAPA). The results (Figure 2) shows that the apparent Henry´s law constant increases with increase of loading or/and temperature. This difference is higher at higher temperatures.
At higher loading, the solubility of N_2O in the solution is lower. Consequently, the solubility of CO_2 in the solution is lower at higher loadings and temperature.

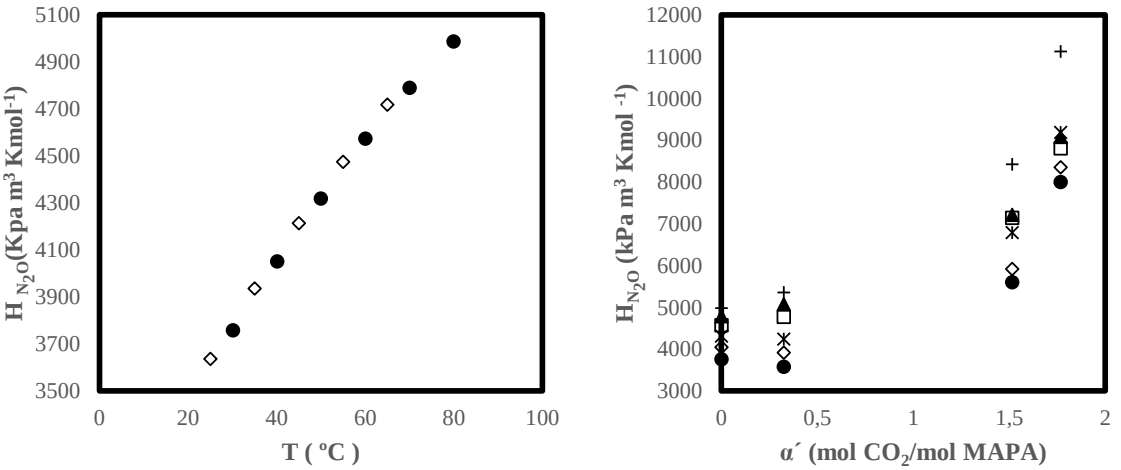

Figure 2 (Left) Apparent Henry´s law constant of unloaded 3M DEEA + 2M MAPA, from 25 to 80ºC, measured in this work (filled circles) and from literature (empty diamonds) [9]; (Right) Apparent Henry´s law constant of 3M DEEA + 2M MAPA solutions, from loadings of 0 to 1.76 mol CO_2/mol MAPA): measured in this work at 30 (circles), 40 (diamonds), 50 (stars), 60 (squares), 70 (triangles) and 80ºC (crosses) ((Right)Apparent Henry´s law constant of 3M DEEA + 2M MAPA solutions, from loadings of 0 to 1.76 mol CO_2/mol MAPA): measured in this work at 30 (circles), 40 (diamonds), 50 (stars), 60 (squares), 70 (triangles) and 80ºC (crosses)

4.2 Kinetics

The results of mass transfer coefficients measured with the DSC for unloaded 3M DEEA+ 2M MAPA solutions are included in the Figure 3.

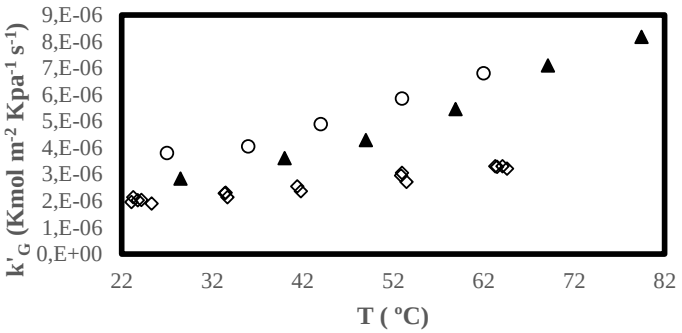

Figure 3 Mass transfer coefficient measured in this work for 3M DEEA+ 2M DEEA solutions from 25 to 80ºC (triangles); measured by Monteiro et al. [9] (empty circles); and for 5M MEA solutions from 25 to 62ºC from the work of Luo et al. [23]

Triangles show the measured values for 3M DEEA + 2M MAPA.. Circles represents data from Monteiro et al. [9] The reported values from this work are lower than the previously reported in the work of Monteiro et al. [9] from 25 to 50ºC. It can be due to the difference of partial pressure of CO_2 used. While in this work the measurements were carried out with a partial pressure of CO_2 of 10-15 kPa in a DSC, Monteiro et al. [9] used partial pressures of CO_2 lower than 0.4 kPa in a String of Discs Contactor (SDC).

Diamonds represents the mass transfer coefficients of 5M MEA, from Luo et al. [23]. As observed in the Figure 3, the unloaded solutions of 3 M DEEA+ 2M MAPA shows higher values of k'_G than the traditional solvent, 5M MEA. This difference is much smaller at low temperature, 25-30 ºC, and reaches its maximum at high temperature, 80ºC. As shown in the Figure 3, the slope of the trend of mass transfer coefficient of 5M MEA vs the temperature is not as high as it of 3 M DEEA + 2 M MAPA.

Mass transfer coefficients were measured from 30 to 70ºC with 3M DEEA+ 2M MAPA with loadings between 0 and 1.2 mol CO_2/mol MAPA, based on the zwitterion mechanism and the two film theory. The results are compared with 5M MEA solutions from literature [23], where the measurements were done with a String of Discs contactor (SDC) and a Wetted Wall Column (WWC). It has been noticed that the results from the WWC were lower than these from the SDC. As observed in the Figure 4, the mass transfer coefficient in 3M DEEA + 2M MAPA solutions decrease with the CO_2 content and increase with temperature. It is noticed that the effect of loading of CO_2 is stronger than changes on the temperature. As seen in Figure 4, the values of loaded solutions at 60-70 ºC are similar than the 5M MEA solutions. However, at lower temperature (30-50ºC), the blend 3M DEEA+ 2M MAPA shows higher values of k'_G. Additionally, the mass transfer coefficient of the absorption of CO_2 in 5M MEA drastically decrease at loading 0.4 mol CO_2/ mol amine. This is because MEA solution is close to maximum loading of 0.5mol CO_2/ mol amine. For the blend 3M DEEA+ 2M MAPA, however, the value of mass transfer continues on the same trend for higher loading.

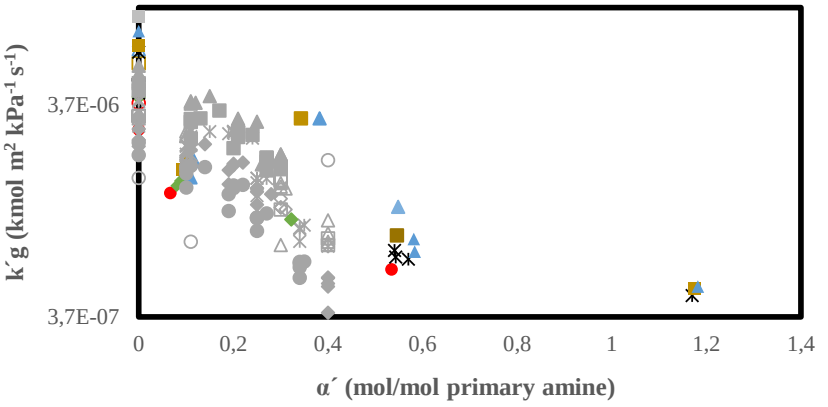

Figure 4 Mass transfer coefficient for 3M DEEA+ 2M MAPA solutions measured in this work; and 5 M MEA solutions from Luo et al. [23] (grey empty figures are measurements from the WWC while grey filled figures are measurements from the SDC). For the temperatures: 30 ºC (circles); 40 ºC (diamonds); 50 ºC (stars); 60 ºC (squares); 70 ºC (triangles)

The observed kinetic coefficients were extracted from 30 to 70ºC at loadings between 0 and 1.16 mol CO_2/mol MAPA (Equation 4). As extracted from the Figure 5, the blend 3M DEEA+ 2M MAPA shows higher values of observed kinetic coefficient than the ones of 5M MEA solutions. As observed in the mass transfer coefficients, these of 5 MEA solutions dramatically decrease at loading 0.4 mol CO_2/mol MEA and this is not observed in the studied blend 3M DEEA + 2M MAPA. On the contrary, the slope of the kinetic coefficients over the loading is maintained until 1.16 mol CO_2/mol MAPA.

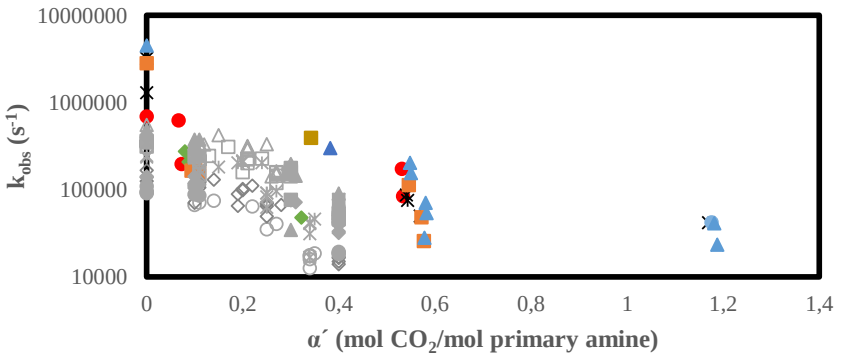

Figure 5 Observed kinetic coefficients for 3M DEEA+ 2M MAPA solutions measured in this work (filled figures); and 5 M MEA solutions from Luo et al. [23] (grey empty figures from the SDC and filled figures from the WWC). For the temperatures: 30 ºC (circles); 40 ºC (diamonds); 50 ºC (stars); 60 ºC (squares); 70 ºC (triangles)

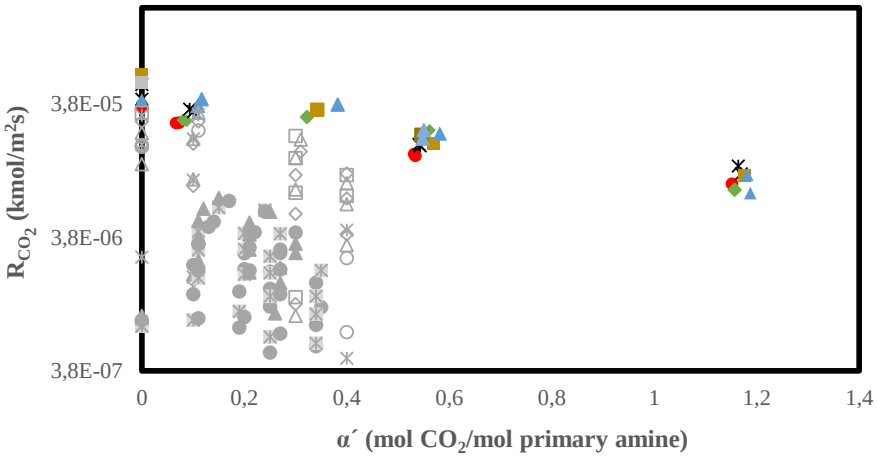

Figure 6 Absorption rate for 3M DEEA+ 2M MAPA solutions measured in this work ; and 5 M MEA solutions from Luo et al. [23] (grey empty figures from the WWC and filled figures from the SDC). For the temperatures: 30 ºC (circles); 40 ºC (diamonds); 50 ºC (stars); 60 ºC (squares); 70 ºC (triangles)

The Figure 6 includes the absorption rate of the loaded solutions of 3M DEEA+ 2M MAPA and its comparison with 5M MEA [23]. As observed in the Figure 6, the absorption rate of 3M DEEA+2MAPA is higher than this of the 5M MEA. At the lowest loading, approximately 0.09 mol/mol primary amine, the 5M MEA solution shows a similar absorption rate than the 3M DEEA+2M MAPA and it decreases at 0.4 mol/mol MEA due to, as mentioned before, the proximity to the its maximum loading. In the case of the 3M DEEA+2M MAPA solutions, still at 1.16 mol/mol MAPA, the absorption rate is considerable.

5. Conclusions

The mass transfer coefficients and observed kinetic constants of the absorption of CO_2 in loaded 3M DEEA and 2 M MAPA were measured with a double stirred cell, from 30 to 70ºC. The zwitterion mechanism and two film theory were used in the calculations and the measurements were based on the pseudo-first order regime. Unloaded 5M MEA and 3M DEEA + 2M MAPA solutions were used for the validation of the apparatus and methodology. The results extracted in this work show a good agreement with the values from the literature.

Physical properties (density, viscosity and N_2O solubility) were measured from 25 to 80ºC and N_2O analogy was used for the calculation of CO_2 solubility and CO_2 diffusion in the amine solutions.

The results included in this work show that the loaded 3M DEEA and 2M MAPA aqueous solution has higher mass transfer and kinetic coefficients of CO_2 absorption than this in 5M MEA. Only at 60-70 ºC, the mass transfer coefficient of 3M DEEA+ 2M MAPA seems to be similar to 5M MEA. Additionally, due to the CO_2 absorption capacity of MAPA, it is possible to reach higher values of CO_2 loading in 3M DEEA + 2M MAPA in comparison to 5M MEA. Even at the loading 1.16 mol CO_2/mol MAPA, the absorption rate does not decrease drastically.

Acknowledgments

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