Kinetics of Carbon Dioxide Chemical Absorption into Cyclic Amines Solutions

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Chemical reaction kinetics between carbon dioxide with two cyclic amines (pyrrolidine and piperidine) have been studied using a stirred tank reactor with a planar interfacial area. The operational variables considered in this work have been the amine concentration in the liquid phase and the reaction temperature. Specific absorption rates have been determined under different experimental conditions. Results indicate that the absorption process occurs in a pseudo first reaction regime exhibited first-order kinetic with respect carbon dioxide and a second order for both cyclic amines. The reaction-rate constant was determined under the different experimental conditions, and it was correlated depending on the temperature by means of an Arrhenius type equation. © 2010 American Institute of Chemical Engineers *AIChE J,* 57: 2244–2250, 2011
Keywords: absorption, gas purification, mass transfer, reaction kinetics

Introduction

Globally speaking, about one-third of all the anthropogenic CO_2 emissions come from fossil fuels, such as coal and oil, used to generate energy. A variety of industrial processes also emit large amounts of CO_2 from each plant, for example oil refineries, cement works, and iron production.[1] There is a growing political and public concern, supported by consensus among the scientific community, about the global emissions growth, that will soon drive atmospheric CO_2 concentrations to levels never seen before, bringing a growing risk of a fast climate change.

The development of more effective processes for acid gases capture (such as carbon dioxide and sulfur dioxide) have produced an important increase in the research studies based on the development of new systems using chemical absorption between the acid gases and selective liquid phases. Absorption processes represent the most important physicochemical operation to remove CO_2 from gaseous streams. The absorption can be done either by using physical solvents such as water, methanol (Rectisol process), or *N*-methyl-2-pyrrolidone (Lurgi's Purisol process), or by using chemical solvents (reactive absorption), for instance, K_2CO_3 (Benfield process), monoethanolamine (MEA; Girbotol process), Sulfolan + diisopropanolamine + water (Shell's Sulfinol process)[2] or amines blends.[3–5] Reactive absorption is the most preferred option used in the gas-processing industry for CO_2 removal, and the aqueous solutions of alkanolamines remain an industrial important class of compounds, used in the natural gas, petroleum chemical plants, and ammonia industries for the removal of CO_2 and H_2S from gas streams. A wide variety of alkanolamines, such as MEA, diethanolamine, di-isopropanolamine, and *N*-methyldiethanolamine have been used industrially for a number of years.[2]

This study pursues two aims: (1) the development of new alternative amine-based solvents, which are attractive for the enhancement of CO_2 capture, based on the use of cyclic amines and (2) the kinetic characterization of the reaction between carbon dioxide and cyclic primary amines [piperidine (PIP) and pyrrolidine (PYR)].

Experimental Section

Commercial grade CO_2 gas of 99.998% purity, supplied by Carburos Metálicos, (Spain) was used in this work. PIP and PYR of $\geq$99% purity was obtained from Fluka (USA). MEA has been

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Table 1. Experimental Compositions Used in This Work

C_B/mol L^{-1}	C_B/wt %	
	PYR	PIP
0.1	0.71	0.85
0.2	1.43	1.71
0.3	2.14	2.56
0.4	2.85	3.42
0.5	3.57	4.27

supplied by Sigma-Aldrich (USA) with a purity of ≥99%. Aqueous solutions of these cyclic amines were prepared on mass with double distilled water. Table 1 shows the amines concentrations used in this work.

The dynamic viscosity of amines aqueous solutions was determined by the product of kinematic viscosity and density. The kinematic viscosity was determined from the transit time of the liquid meniscus through a capillary viscosimeter supplied by Schott using the procedures described in previous studies.[6] In the measurements, it was used a Schott-Geräte AVS 350 Ubbelohde viscometer. Density was measured with an Anton Paar DSA 5000 vibrating tube densimeter.

The gas–liquid kinetic experiments were conducted in an experimental setup used previously[7] (see Figure 1), consisting on a stirred cell having a planar interfacial area, working in batches with regard to both phases. Four baffles have been placed in its internal wall to improve the mixing and prevent vortex. The gas phase, pure carbon dioxide, was passed through two humidifiers to prepare the gas phase. This procedure allows the evaluation of the liquid-phase resistance to the gas transfer without other influences upon mass transfer. Water was placed into the "humidifiers." A soap flow-meter was used to determine the carbon dioxide absorption rate accompanied by some chemical reaction, produced by the glucosamine (GA) present in the liquid phase. The absorption rate was measured by analyzing the movement of the soap film (produced by the consumption of carbon dioxide) along the calibrated glass tube.[8] For each, experiment was produced in the gas flowmeter and then, the liquid phase was introduced in the stirred cell. The magnetic stirring began when liquid addition was concluded. At this moment, the experimental data recording was developed by means of software that allows obtain the rate of the soap film in the flowmeter removing errors in the experiments

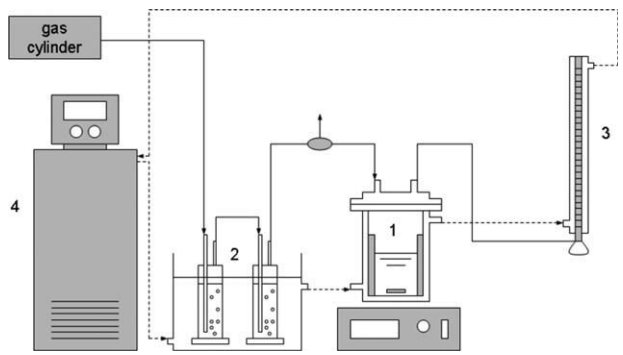


Figure 1. Experimental setup for kinetic studies of gas–liquid systems.

(1) Stirred cell, (2) humidifiers, (3) soap flowmeter, and (4) cryostat-thermostat.

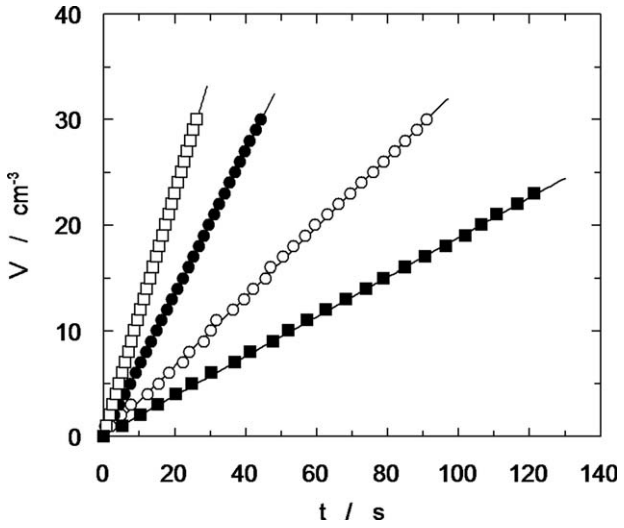


Figure 2. Influence of liquid-phase composition upon carbon dioxide absorption rate in pyrrolidine aqueous solutions.

(■) C_B = 0.1 mol L^{-1}; (○) C_B = 0.3 mol L^{-1}; (●) C_B = 0.4 mol L^{-1}; (□) C_B = 0.5 mol L^{-1}. T = 25°C.

with a high-absorption rate. This procedure was repeated 3 times for each experimental condition, using the mean value for the calculus developed in this work. Absorption processes have been carried out at different temperatures (7–55°C) by means of the connection of the experimental setup to a thermostat-cryostat.

Results and Discussion

The carbon dioxide flow density was determined by means of experimental data corresponding to the absorbed quantity of carbon dioxide along the operation time. Figures 2 and 3

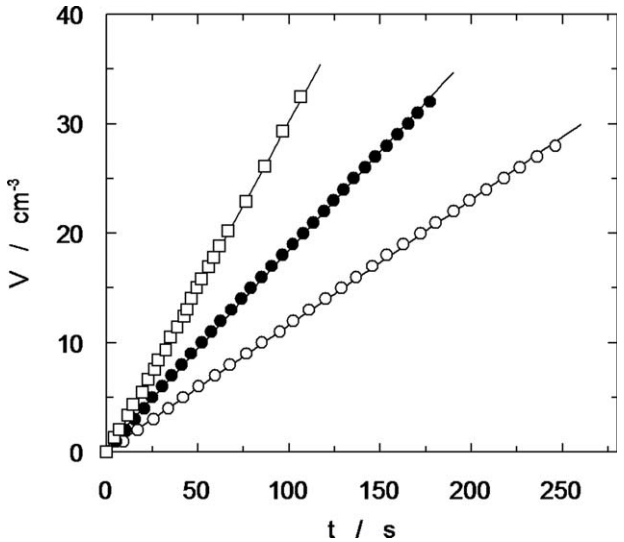


Figure 3. Effect of temperature upon carbon dioxide absorption rate in aqueous solutions of pyrrolidine.

(○) T = 7°C; (●) T = 25°C; (□) T = 55°C. C_B = 0.1 mol L^{-1}.

Table 2. Viscosity of Aqueous Solutions of Pyrrolidine (PYR), Piperidine (PIP), Glucosamine (GA), and Monoethanolamine (MEA) at 20°C and Absorption Density for Each Amine System

	PYR		PIP		GA		MEA	
C_B (mol L^{-1})	η (mPa s)	$N_A \times 10^{-4}$ (mol m^2 s^{-1})	η (mPa s)	$N_A \cdot \times 10^{-4}$ (mol m^2 s^{-1})	η (mPa s)	$N_A \cdot \times 10^{-4}$ (mol m^2 s^{-1})	η (mPa s)	$N_A \cdot \times 10^{-4}$ (mol m^2 s^{-1})
0	0.989	9.7	0.989	9.7	0.989	9.7	0.989	9.7
0.1	1.023	22.4	1.016	22.4	1.046	16.50[7]	1.013	12.9
0.2	1.058	35.2	1.044	34.8	1.114	21.40[7]	1.034	16.8
0.3	1.094	56.1	1.073	55.2	1.168	25.10[7]	1.055	20.0
0.4	1.131	83.0	1.105	80.9	1.227	27.30[7]	1.076	23.9
0.5	1.168	117.2	1.137	113.7	1.282	–	1.096	28.8

show the experimental results obtained in relation to the carbon dioxide flux density, determined for the different liquid phases and experimental conditions used in this study. In these figures, the experimental data show a linear trend in all the cases, and this behavior allows the use of these experimental data for kinetic determination. The slope of the linear plot, previously noted, allows the calculation of the absorption volumetric flux at different operation conditions. Results from Figure 2 indicate that there is an influence of the initial PYR concentration in the liquid phase upon the carbon dioxide absorption rate or upon the slope of linear fits. The presence of PYR in the aqueous solution increase the liquid phase viscosity (see Table 2) (this effect tend to reduce mass-transfer rate in physical absorption), so the obtained behavior confirms the existence of a chemical reaction in the liquid phase between the carbon dioxide and the PYR, because an increase in the volumetric flux (related to the increase in the linear fit slope) is observed when the amine concentration increases in the liquid phase (see Table 2). The same behavior has been obtained when different concentrations of PIP were used.

We have developed the analysis of the influence of amines concentration in the liquid phase upon the carbon dioxide absorption rate, and the results allow us to conclude that there is a chemical reaction between the amines and the carbon dioxide absorbed in the liquid phase. Figure 3 shows the effect of temperature upon the rate that the chemical absorption is produced. The experimental data show that an increase in temperature to develop the absorption experiments also produces an increase in the slope of the plot between the carbon dioxide absorbed flow rate and the operation time. This way, the absorption rate increases as well. Figure 3 includes experimental data corresponding to aqueous solutions of PYR, but a similar behavior was observed for the same study using PIP as an amine. The obtained behavior, in relation to the influence of temperature upon the absorption rate, is similar to the corresponding ones using other kind of amines for carbon dioxide chemical absorption.[8]

The cyclic amines used in this work only include an amino group (NH), so the possible influence of the hydroxyl group (common in other amines used for the carbon dioxide capture)[9] upon the kinetic study must not be taken into account.

Using the experimental data of carbon dioxide absorbed volume along the operation time, the gas flow density has been determined under the different experimental conditions. Figures 4 and 5 show the results calculated for both experimental systems used in this work. It is also possible to observe in these figures the influence of each amine concentration in the liquid phase, as well as the effect caused by temperature upon the carbon dioxide absorption rate. The behavior observed for both amines is quite similar to a positive effect upon the absorption rate when the amines concentration increases in the liquid phase. The influence of temperature was also the same, producing an increase in the global gas mass-transfer rate.

In relation to the use of different amines for the carbon dioxide capture, a comparison between the absorption rate data for carbon dioxide using aqueous solutions of ethanolamine (MEA) and GA[7] has been carried out. MEA is commonly used for carbon dioxide capture, whereas the use of aqueous solutions of GA for carbon dioxide removal has been suggested in the last years, contributing to improvements in operational safety in comparison with more common processes. Figure 6 shows this comparison between the behavior of these amines, and the experimental results indicate that cyclic amines (PYR and PIP) allow carbon dioxide capture at a higher absorption rate than aqueous solutions of MEA and GA. Figure 6 shows that the difference observed regarding absorption rate in the different amine-based systems is important, and it indicates that these cyclic amines improve significantly the carbon dioxide capture process.

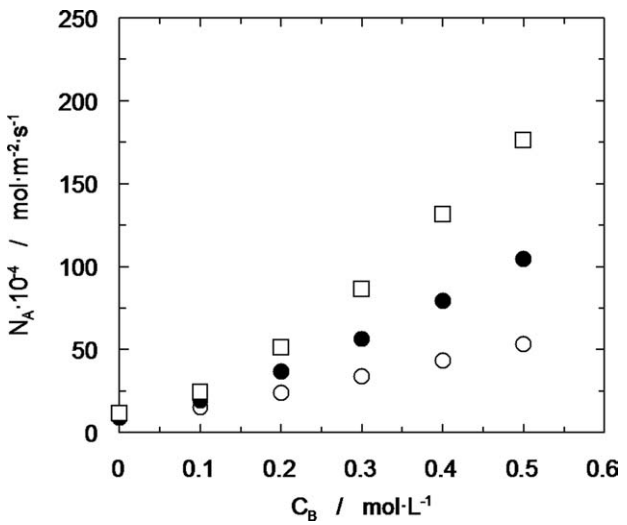


Figure 4. Influence of piperidine concentration upon the carbon dioxide absorbed flow.

(○) $T = 7$°C; (●) $T = 25$°C; (□) $T = 45$°C.

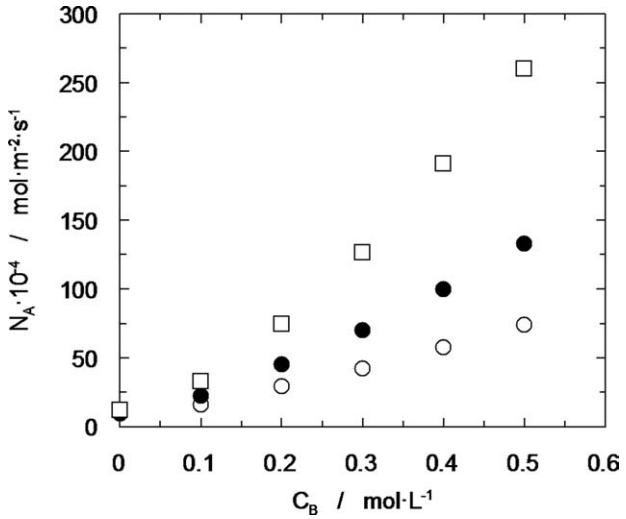


Figure 5. Influence of pyrrolidine concentration upon the carbon dioxide flow.

(○) $T = 15$°C; (●) $T = 35$°C; (□) $T = 55$°C.

The reaction between the carbon dioxide and the amine involves different parallel chemical reactions, such as the reaction between this gas and hydroxyl ions (bicarbonate formation) and water (carbonic acid formation). However, the influence of these reactions under the conditions employed in this work could be considered negligible. The carbonic acid formation and reaction is very slow and it must be considered of no influence on the system studied. Regarding the other parallel reaction, it could be negligible due to the low-hydroxyl ions concentration in the system, the way different studies have proved.[10–12]

The kinetic has been analyzed in the basis of conclusions reached in different studies (i.e., Astarita et al.[13]), which

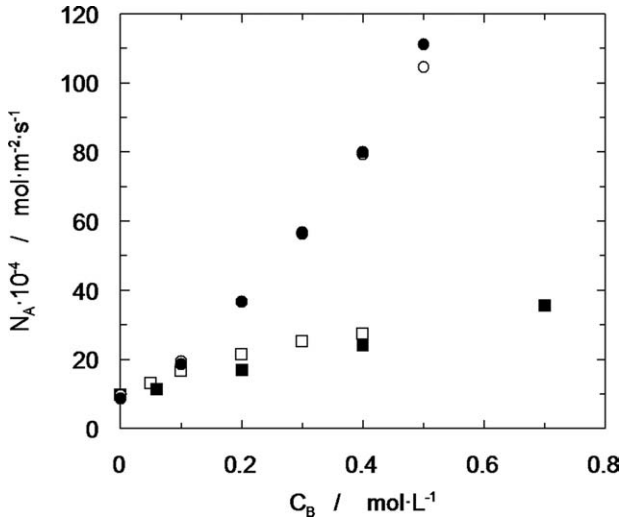


Figure 6. Comparison between the absorption-flux density corresponding to cyclic amine systems and the corresponding ones for other primary amines.

(○) Piperidine; (●) pyrrolidine; (□) glucosamine[7]; and (■) monoethanolamine. $T = 25$°C.

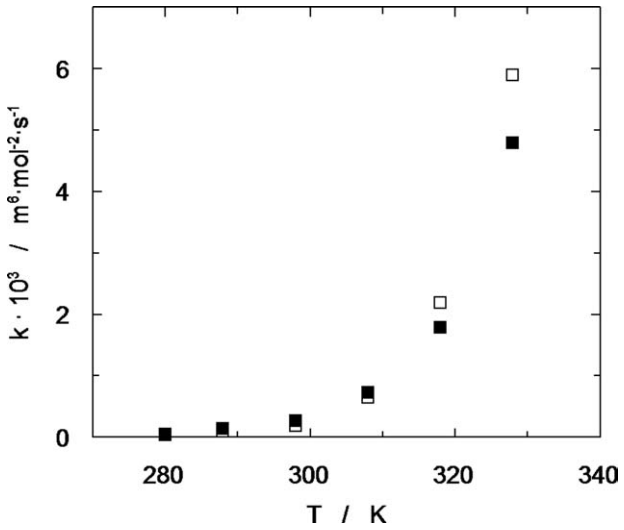


Figure 7. Effect of temperature upon the kinetic constant value corresponding to the chemical reaction between carbon dioxide and cyclic amines.

(□) Carbon dioxide—pyrrolidine system; (■) carbon dioxide—piperidine system.

indicate that the carbamic reaction is the mechanism of reaction between carbon dioxide and amines when the carbonation relationship (moles of carbon dioxide/moles of amine) is less than 0.5. Under these conditions, three possible regimes could tale place: physical absorption, fast-reaction, and instantaneous regime. Because the amine concentration is high in the liquid phase and this implies values of $C_{Bo}/C_{Ao} \gg 1$, the physical absorption regime must be ruled out.

An instantaneous regime for the absorption, accompanied by a chemical reaction, relates the carbon dioxide flow density to the initial amine concentration, where a linear trend is detected. Figures 4 and 5 show this relation and the obtained behaviors indicate that this regime does not govern the overall reaction mechanism.

The last regime, fast-reaction, allows the calculation of the carbon dioxide flow density by means of Eq. 1, taking into account that the reaction order for the carbon dioxide, when reacting to aqueous solutions of amines, is commonly one.

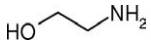
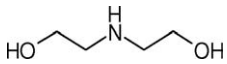
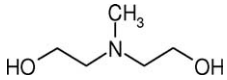
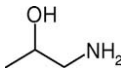
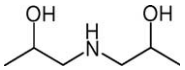
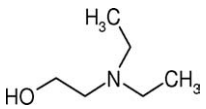
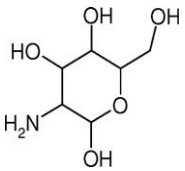
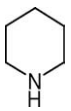
$$N_A = C_{Ao}\sqrt{D_A \cdot k_{1,n} \cdot C_B^n} \tag{1}$$

where C_{Ao} and C_{Bo} are the initial concentrations of carbon dioxide and GA in the liquid phase, D_A is the gas diffusivity in GA aqueous solutions and $k_{1,n}$ is the overall reaction rate constant. The diffusion coefficient corresponding to carbon dioxide in aqueous solutions of different cyclic amines was calculated using the expression shown in Eq. 2.[14]

$$D_A = D_{A,w} \cdot \left(\frac{\eta_w}{\eta}\right)^{0.8} \tag{2}$$

where $D_{A,w}$ is the diffusivity of carbon dioxide in pure water, η_w and η are the viscosity of pure water and the aqueous solutions of each amine, respectively. The value of carbon

Table 3. Activation Energy for the Chemicals Reaction Between Carbon Dioxide and Several Amines

Amine	Structure	E_a/kJ mol^{-1}	References
Monoethanolamine (MEA)		41.2	21
Diethanolamine (DEA)		32.3	22
Methyldiethanolamine (MDEA)		19.8	12
3-Amino-1-propanol (AP)		43.5	23
2-Amino-2-methyl-1-propanol (AMP)		68.0	8
1-Amino-2-propanol (MIPA)		41.8	24
Di-isopropanolamine (DIPA)		59.9	19
N,*N*-diethylethanolamine (DEEA)		95.4	25
Glucosamine (GA)		47.6	7
Piperidine (PIP)		75.9	This work
Pyrrolidine (PYR)		79.9	This work

dioxide diffusivity in pure water was determined using the expressions proposed by different studies.[15] On the other hand, the water viscosity and cyclic amines aqueous solutions have been obtained from literature.[6]

It has been shown in the literature[16] that the partial order with respect to CO_2 is always 1, as we obtained in this work based on the experimental data, but the partial order regarding the amine can vary between 1 and 2, depending on the chosen amine.[17]

The initial carbon dioxide concentration in the liquid phase coincides with the concentration in equilibrium in the gas phase, and this parameter could be replaced in Eq. 1 using Henry's law. These considerations allow us to obtain the linearized expression shown in Eq. 3 to fit experimental data and to calculate the reaction order corresponding to amines:

$$\log\left(\frac{N_A^2 \cdot He^2}{P_A^2 \cdot D_A}\right) = \log(k_{1,n}) + n \cdot \log(C_B) \qquad (3)$$

where He is Henry's constant and P_A is the carbon dioxide partial pressure.

Fitting the experimental values corresponding to the carbon dioxide flow density vs. the initial amines concentration,

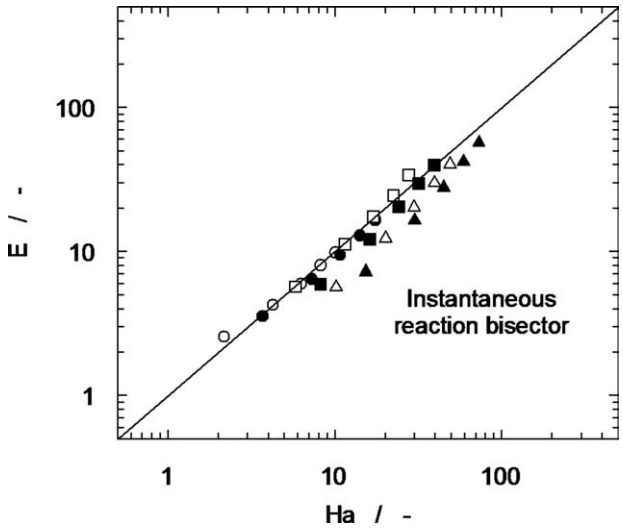


Figure 8. Relation between enhancement factor and Hatta number for carbon dioxide—aqueous solutions of pyrrolidine.

(○) $T = 7°C$; (●) $T = 15°C$; (□) $T = 25°C$; (■) $T = 35°C$; (△) $T = 45°C$; (▲) $T = 55°C$.

the fit agreement is satisfactory and then, kinetic parameters were determined. In relation to the corresponding reaction order for each amine, it was determined for all temperatures and both systems show average values of 2.2 and 2.1 for PIP and PYR, respectively. To a large extent of carbon dioxide—aqueous solutions of amines systems the corresponding reaction order is one,[18] but in certain systems[19,20] the same reaction order as in this work was obtained.

The value of the kinetic constant corresponding to each experimental system has also been determined in this work, using Eq. 3. The calculated values for this parameter and the influence of temperature upon this value are shown in Figure 7. For low temperature, both systems take similar values for the kinetic constant, but when temperature increases, the carbon dioxide—PYR system takes high values for this parameter in relation to the other system.

The influence of temperature upon the value of the kinetic constant for the reaction studied in this work has been analyzed. Experimental values have been fitted using an Arrhenius type equation to calculate the pre-exponential factor and the activation energy. Equations 4 and 5 show the calculated values for both parameters.

$$\text{Piperidine}: \qquad \ln\, k = 36.3 - \frac{9144}{T} \tag{4}$$

$$\text{Pyrrolidine}: \qquad \ln\, k = 37.9 - \frac{9620}{T} \tag{5}$$

The values determined for the activation energy were 75.9 and 79.9 kJ mol^{-1} for PIP and PYR, respectively. The calculated values for this parameter are higher than the corresponding ones for the reaction of carbon dioxide and other amines, such as MEA, MIPA, AP,[8] and GA.[7]

Taking into account the previously commented kinetic parameters, we can conclude that the value of the pre-exponential parameter for the cyclic amines is higher than the corresponding one for other amines previously commented, and these cyclic amine systems also take higher values for the activation energy (see Table 3).

The pre-exponential factor is proportional to the global number of collisions which lead to reaction, whereas the other part of the equation, that involves the activation energy, is the fraction of collisions that results in reaction (effective collisions).

Bearing these comments in mind, we can conclude that the reaction between carbon dioxide absorbed and the cyclic amines (PYR and PIP) present in the liquid phase, requires a higher activation energy (higher energy barrier) than the corresponding reaction of this gas with other common amines. This behavior is due to the reaction with cyclic amines, which suffer a high-steric hindrance that difficult the effective collisions between both molecules. On the other hand, these cyclic amines provide a higher electronic density regarding other amines (i.e., MEA and GA) that favor the chemical reaction and then increase the reaction rate (shown in Figure 7).

The Hatta number (Ha) and enhancement factor (E) have been calculated with the aim of confirming the reaction regime supposed at the beginning of the results and discussion section. Equation 6 has been used to determine the Hatta number,

$$Ha = \sqrt{\frac{k \cdot C_{\mathrm{Bo}} \cdot D_{\mathrm{A}}}{k_{\mathrm{L}}^2}} \tag{6}$$

where k_{L} is the liquid-phase mass-transfer coefficient.

Equation 7 has been used to calculate the value of this enhancement factor, based on the value of carbon dioxide flux density.

$$E = \frac{N_{\mathrm{A}}}{k_{\mathrm{L}} \cdot C_{\mathrm{A}}^*} \tag{7}$$

Equations 6 and 7 include in their expressions the value of mass-transfer coefficient corresponding to the liquid phase, k_{L}. This parameter has been determined using the same aqueous solutions of cyclic amines, but the pH was modified adding HCl to produce an acidic medium and then inhibit the chemical reaction between carbon dioxide and the amines.

In addition, the instantaneous enhancement factor has been calculated using Eq. 8.

$$E_{\mathrm{i}} = 1 + \frac{D_{\mathrm{B}}}{D_{\mathrm{A}}} \cdot \frac{C_{\mathrm{Bo}}}{C_{\mathrm{A}}} \tag{8}$$

The diffusion coefficient for each cyclic amine (D_{B}) in aqueous solution has been calculated by means of the Wilke-Chang equation[26] modified by Hayduk and Laudie[27] (Eq. 9).

$$D_{\mathrm{B}} = \frac{7.4 \cdot 10^{-8} \cdot \sqrt{\varphi_{\mathrm{w}} \cdot M_{\mathrm{w}}} \cdot T}{\eta_{\mathrm{w}} \cdot V_{\mathrm{B}}^{0.6}} \tag{9}$$

where M_{w} is molecular weight of water, T is temperature, η_{w} is the viscosity of water, V_{B} is the molar volume of each amine, and φ_{w} is the solvent (water) association factor with a value of 2.26.

Using the previously commented equations, we can observe that the values of Hatta number take values in all the cases into the interval established by Eq. 10 that match the characteristics of an instantaneous and pseudofirst reaction order.

$$3 < Ha < \frac{10 \cdot E_i}{2} \quad (10)$$

Figure 8 shows the relation between the enhancement factor and Hatta number in logarithmic coordinates. This plot shows that the enhancement factor experimental data takes values lower than the bisector. Therefore, the instantaneous reaction regime for the absorption accompanied by a chemical reaction between carbon dioxide and PYR is confirmed. The reactive system formed by carbon dioxide and PIP aqueous solutions shows the same behavior previously described for aqueous solutions of PYR.

Conclusions

Two new amines (PYR and PIP) have been used to capture carbon dioxide by chemical absorption, and the reaction kinetic for both systems has been determined. The absorption rate experimental data and the chemical absorption theory have determined the existence of an instantaneous and pseudofirst reaction regime between the carbon dioxide and both cyclic amines. The reaction order corresponding to carbon dioxide was 1 and 2 for the amines. The influence of the temperature upon the rate constant has been analyzed, and the activation energy was calculated and compared with the value obtained for other carbon dioxide–amine systems. The conclusion is that the amines used in this study show a higher steric hindrance, but also have high-electron density, involved in the high-reaction rate shown in these experimental systems.

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