



Absorption of CO₂ in the aqueous solutions of functionalized ionic liquids and MDEA

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ABSTRACT

Four ionic liquids (ILs)—tetramethylammonium glycinate (=Gly), tetraethylammonium glycinate ([N₂₂₂₂][Gly]), tetramethylammonium lysinate ([N₁₁₁₁][Lys]) and tetraethylammonium lysinate ([N₂₂₂₂][Lys]) were synthesized and mixed with water or N-methyldiethanolamine (MDEA) aqueous solutions to form a new type of solvents for the uptake of CO₂. The solubility or absorption of CO₂ in these IL + MDEA aqueous solutions was investigated over a wide range of IL concentrations (5–100%), temperature (298–318 K) and partial pressure of CO₂ (4–400 kPa). The results indicated that ionic liquid could greatly enhance the absorption and increased the absorption rate of CO₂ in MDEA aqueous solutions. It had been found that the aqueous solutions of 15% IL and 15% MDEA had higher absorption rate and larger uptake than other IL + MDEA solutions of 30% total amines. Temperature (298–318 K) seemed to have some influence on the absorption of CO₂ in IL + MDEA aqueous solutions. Noticeably, due to the two amino groups in a molecular, the mole absorption of the 30% lysine based ionic liquids aqueous solutions was 0.98 ([N₁₁₁₁][Lys]) and 1.21 ([N₂₂₂₂][Lys]) mole CO₂, being about 2–3 times the absorption capacity of MDEA under the same condition. Regeneration under the condition of temperature 353 K, 4 kPa for 240 min showed that aqueous solution of 15% [N₁₁₁₁][Gly] + 15% MDEA had significant regeneration efficiency (over 98%).

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1. Introduction

Chemical absorption of acid gases, such as CO₂, H₂S or SO₂ has been widely used in the industrial process. At present, one of the effective methods for the uptake of CO₂ usually employs aqueous solutions of alkanolamine or their mixtures, including monoethanolamine (MEA), diethanolamine (DEA), and N-methyldiethanolamine (MDEA) [1]. However, the use of alkanolamine presents several concerns: (1) corrosion of amines system limits the concentration of amine in the solution and causes extra cost; (2) amines are prone to degrade under high temperature especially in the regeneration process; (3) oxidative degradation of amines occurs in the presence of oxygen; (4) amines are volatile and lost into the gas stream. The loss of absorbents not only produces extra waste streams but also leads to additional absorbent cost. It should be noted that among the amines usually used, MDEA has lower volatility, thermal stability, less alkaline, higher CO₂ load

(up to 1.0 mol CO₂/mol amine) and less regeneration cost, though the absorption of CO₂ into MDEA is quite slow since MDEA just promotes CO₂ hydrolysis to form bicarbonate. Therefore, in the industrial application, aqueous solution of MDEA is usually mixed with activators of fast reactivity, including MEA, piperazine (PZ), and DEA [2].

Ionic liquids (ILs) are organic salts that form stable liquid below 100 °C or even at room temperature (room temperature ionic liquids, RTILs). For the removal of acidic gases, compared with alkanolamines, RTILs have negligible volatility and significant thermal stability that avoids loss of absorbents. Brennecke's group [3] reported firstly that CO₂ was highly soluble in [bmim][PF₆](1-butyl-3-methylimidazolium hexafluorophosphate), reaching a mole fraction of 0.6 at 8 MPa. Since 0.2 MPa was probably the lowest limit of the Henry's law constant for the physical absorption of CO₂ in ILs at 298 K [4], when pressure of CO₂ was below ambient pressure, the solubility of CO₂ in conventional ILs was only up to 0.035 mol fraction.

By introducing functional groups that could enhance the CO₂ absorption into the anion or cation, functionalized ionic liquids are expected to have significant performance in the uptake of CO₂. Bates et al. [5] synthesized the amine functionalized ionic

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liquid–[pNH₂bim][PF₆], whose molar uptake of CO₂ per mole of IL during 3 h approached 0.5 under the normal pressure and temperature, close to the theoretical maximum for CO₂ sequestration. The IL could be regenerated upon heating (80–100 °C) for several hours under vacuum. In 2005, Ohno's group [6] prepared amino acid based ILs [emim][amino acid] from 20 natural amino acids. Tetraalkylphosphonium amino acid ILs were soon reported in 2006 [7]. Then, five tetrabutylphosphonium amino acid was reported by Zhang's et al. [8]. Due to relatively high viscosities (245–745 mPa s), tetraalkylphosphonium amino acid ILs were loaded on porous silica gel to absorb CO₂. The absorption mechanism of pure amino acid ILs was similar to that of [pNH₂bim][PF₆], i.e., one CO₂ molecule was combined with two amino acid molecules [8].

The viscosities of these amino acid ILs (AAILs) were all over 200 mPa s [9]. In general, the diffusivity of CO₂ is determined by the viscosity of the solvent, i.e., the lower viscosity of solvent, the higher CO₂ diffusivity in the solvent and thus the faster absorption. Jiang et al. [9] in our research group successfully synthesized nine tetraalkylammonium based amino acid ionic liquids which could rapidly absorb CO₂ (the absorption equilibrium could all be reached within 60 min). Among them, viscosities of [N₂₂₂₂][L-Ala] and [N₂₂₂₂][β-Ala] were 81 and 132 mPa s, respectively, lower than the reported amino acid based ionic liquids.

The disadvantages of all ILs, particularly the functionalized ILs, are the high viscosity and cost, which seriously hinder their application in the separation of sour gases. In the light of methods and theories of the mixed amines, mixing functionalized ILs with MDEA solution is a good way to improve the usage of ILs in the absorption of CO₂ [10]. In general, the amino acid ILs with very high solubility in water, lead to a very high concentration of IL. In a US patent [11], ionic liquid [bmim][BF₄] (1-butyl-3-methylimidazolium tetrafluoroborate) and [bmim][acetate] aqueous solutions, and the hybrid of ionic liquid and MDEA (MEA) were used for the removal of CO₂. However, the absorption of CO₂ in these solvents was limited under high equilibrium partial pressure of CO₂ since [bmim][BF₄] and [bmim][acetate] were not very good for the absorption of CO₂.

Table 1
Physical parameters of ILs (298 K).

IL	Molecular weight	ρ (g cm ⁻³)	η (mPa s)
[N ₁₁₁₁][Gly] ^a	148.18	1.089	304
[N ₂₂₂₂][Gly] ^a	204.35	1.064	129
[N ₁₁₁₁][Lys] ^b	219.11	1.071	569
[N ₂₂₂₂][Lys] ^a	275.28	1.050	672

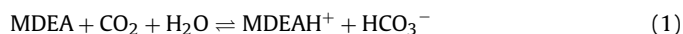
^a Data from Ref. [9,10].

^b Measured at 323 K.

two amine groups in one lysine molecule, probably leading to more CO₂ load. Glycinate has been reported as good absorbents of CO₂ [12,13]. Thus four ionic liquids—tetramethylammonium glycinate ([N₁₁₁₁][Gly]), tetraethylammonium glycinate ([N₂₂₂₂][Gly]), tetramethylammonium lysinate ([N₁₁₁₁][Lys]) and tetraethylammonium lysinate ([N₂₂₂₂][Lys]) could be synthesized and used for CO₂ absorption. The density and viscosity of these ILs are listed in Table 1. The aqueous solutions of functionalized ILs and MDEA would take advantages of IL's stability and fast absorbing rate, and the high CO₂ load of MDEA. The absorption performance and physical properties of these ILs and their mixture with MDEA aqueous solutions will be investigated in detail.

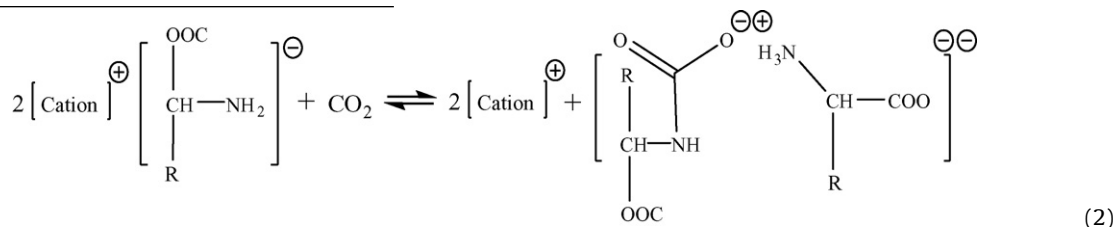
2. Reaction mechanism

The mechanism for the reaction of MDEA (tertiary amines) and CO₂ was proposed by Donaldson and Nguyen [14]:



Since this reaction is essentially a base-catalyzed CO₂ hydrolysis, i.e., MDEA does not combine with CO₂, the absorption is very slow.

For pure amino acid based ILs, one CO₂ molecule is combined with two IL molecules [8], thus the theoretical maximum of CO₂ load is 0.5 mol per IL mol:



In the present work, as shown in Fig. 1, [N₁₁₁₁]⁺ and [N₂₂₂₂]⁺ were chosen as cation, and lysine and glycine as anion. There are

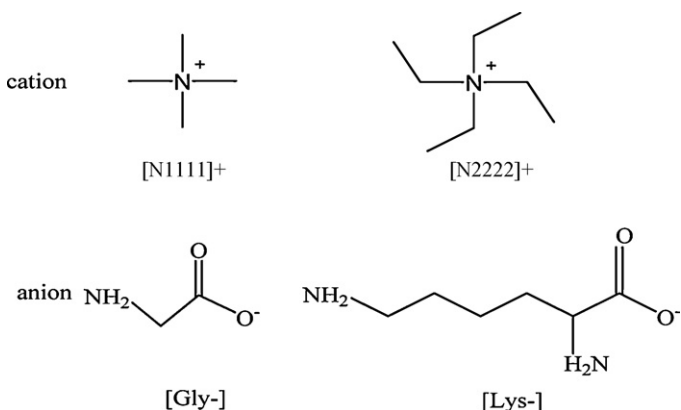
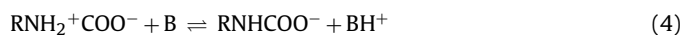


Fig. 1. Cation and anion of amino acid ionic liquids.

Tetraalkylammonium amino acid IL completely dissociates into hydrated cations and [H₂N–CHR–COO]⁻ anion in aqueous solution. Since the amino acid ions react with CO₂ similarly to the primary alkanolamines, [H₂N–CHR–COO]⁻ is represented as RNH₂ in the following. The zwitterion mechanism is generally applied to model the carbon dioxide absorption in amino acid solutions [15]. The zwitterion is produced initially through the reaction of CO₂ with amino acid:

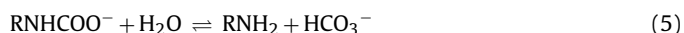


Then, the zwitterion is deprotonated by the bases (denoted by B) in the solution, including RNH₂, H₂O, OH⁻, and MDEA.



In the present aqueous solution systems, the amine group of the amino acid can react quickly with CO₂ to form zwitterions which will transfer protons to MDEA, the absorption rate of CO₂ is therefore increased greatly, compared with the MDEA aqueous solution. It was noteworthy that precipitation of the products was observed at high concentration of amino acid salt and high loading, due to the form of the amino acid (RNH₃⁺) [12,16]. In this work, RNH₃⁺

was expected to be eliminated by plenty of MDEA. The equilibrium reactions in the liquid phase are suggested as follows:



Through Eqs. (5)–(10), the concentration of species could be calculated.

3. Experimental

The amino acid ILs were prepared in the same way as described by Jiang et al. [9]. The densities of the ILs and their aqueous solutions were measured using Anton Paar DMA 5000, with a precision of $0.000001 \text{ g cm}^{-3}$. The viscosities of the liquids were measured on a HAAKE Rheostress 600 viscometer, and the pressure of the system is recorded every 10 s using a pressure transducer WIDEPLUS-8 manufactured by Fujian WIDEPLUS Precision Instruments Co., Ltd.

The absorption of CO_2 was carried out with the set-up shown in Fig. 2. The experimental procedure is as follows: absorbent of a known weight (about 4 g) is placed in the absorption vessel (57.3 ml) and degassed by vacuum pumping; close valve 1 and valve 4, then open valve 2 and transfer gases into the storage vessel (176.3 ml) until the pressure reaches a scheduled value, P_0 ; close valve 2 and valve 3, then open valve 4 to transfer gas from storage vessel to absorption vessel where the absorbent is stirred magnetically. Since the pressure (P) in the absorption vessel decrease as the gas being absorbed, amount of the gas absorbed will be calculated by measuring the variation of the pressure in the absorption vessel.

In the experiments, the temperature of the storage vessel and absorption vessel is maintained with a certain value with a thermostatic water bath. The absorption could be performed under several temperature values to reveal the influence of temperature on the absorption of CO_2 in the solutions. Besides pure CO_2 , N_2 was used to explore the influence of the inert gas on the CO_2 absorption. Three kinds of liquids were prepared as absorbents: (1) pure ionic liquids; (2) ionic liquid aqueous solutions with the weight fraction ranging 10–80%; (3) IL+MDEA aqueous solutions with 30% total amines (amino acid IL was regarded as amines as well as MDEA). After the fully absorption of CO_2 in the solutions at $P_0 = 97 \text{ kPa}$ and

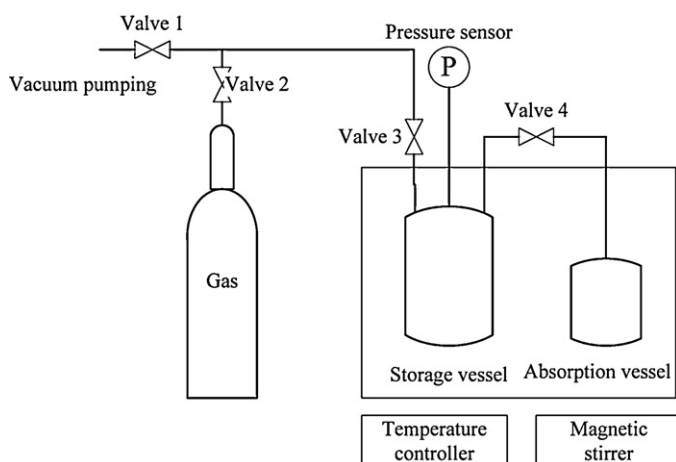


Fig. 2. Schematic diagram of gas absorption apparatus.

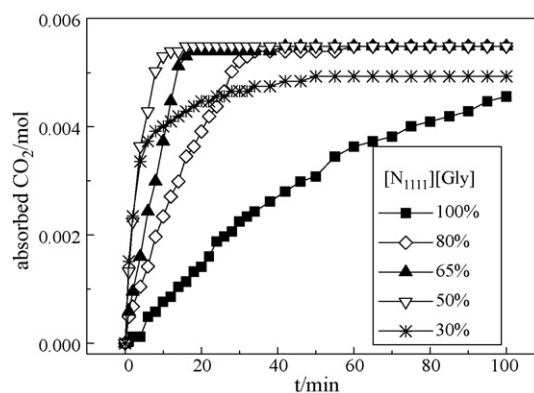


Fig. 3. Absorption amount of CO_2 in aqueous solution of $[\text{N}_{1111}][\text{Gly}]$.

$T = 298 \text{ K}$, the regeneration of the absorbent in the present work was carried out under the condition of 353 K , 4 kPa for 240 min. Appropriate amount of water was added to keep the weight of the solution and the second absorption was performed. Then the absorbent was regenerated under the same condition as the first regeneration, followed by another absorption. The regeneration efficiency was calculated from the ratio of the second absorption amount and the first absorption amount.

4. Results and discussion

4.1. Absorption of CO_2 in various solutions

The amount of absorbed CO_2 can be calculated by the following equation:

$$n_{\text{CO}_2} = \frac{P_0 V_S - P(V_S + V_A - V_L) + P_v V_L}{RT} \quad (11)$$

where V_S and V_A are denoted to volumes of storage vessel and absorption vessel, respectively. V_L represents volume of liquid and P_v the saturated vapor pressure of liquid.

Uptake of CO_2 into aqueous solutions of $[\text{N}_{1111}][\text{Gly}]$ at $P_0 = 97 \text{ kPa}$ and $T = 298 \text{ K}$, is illustrated in Fig. 3. It can be seen that the absorption of CO_2 in the pure IL was rather slow due to the very high viscosity of the liquid. With the addition of water, the absorption of CO_2 was greatly accelerated and the absorption equilibriums can be reached in less than 45 min. In the four aqueous solutions of IL (30%, 50%, 65% and 80%), the 50% IL aqueous solution had a rapid absorption rate and more absorption capacity of CO_2 than the aqueous solutions of 65% IL and 80% IL, while the amounts of absorbed CO_2 in the aqueous solutions of IL (50%, 65% and 80%) were almost the same. This could be explained from Eq. (4): for the 80% IL solution, the zwitterion was deprotonated basically by RNH_2 since there were larger amount of RNH_2 in the solution; for the 65% and 50% IL aqueous solution, some water participated in the deprotonation of zwitterion, thus a considerable amount of fresh RNH_2 could react with CO_2 , inducing a relatively high absorption amount. Moreover, according to Eq. (5), the carbamate could hydrolyze to bring fresh RNH_2 when the concentration of water is high. Due to the lower viscosity, the gas absorption in the aqueous solution of 30% IL was very fast in the initial stage, though the uptake amount of CO_2 was eventually a little less than other aqueous solutions due to the less molecules of IL in the liquid. The absorption capacity of the solutions was compared in Table 2, indicating that the CO_2 load was $0.61 \text{ mol CO}_2/\text{mol IL}$ for 30% $[\text{N}_{1111}][\text{Gly}]$ aqueous solution larger than those of 65% and 80% IL aqueous solutions. Moreover, as shown in Fig. 3, the absorption rate of CO_2 in 30% $[\text{N}_{1111}][\text{Gly}]$ aqueous solution was also very high, especially in the first 10 min.

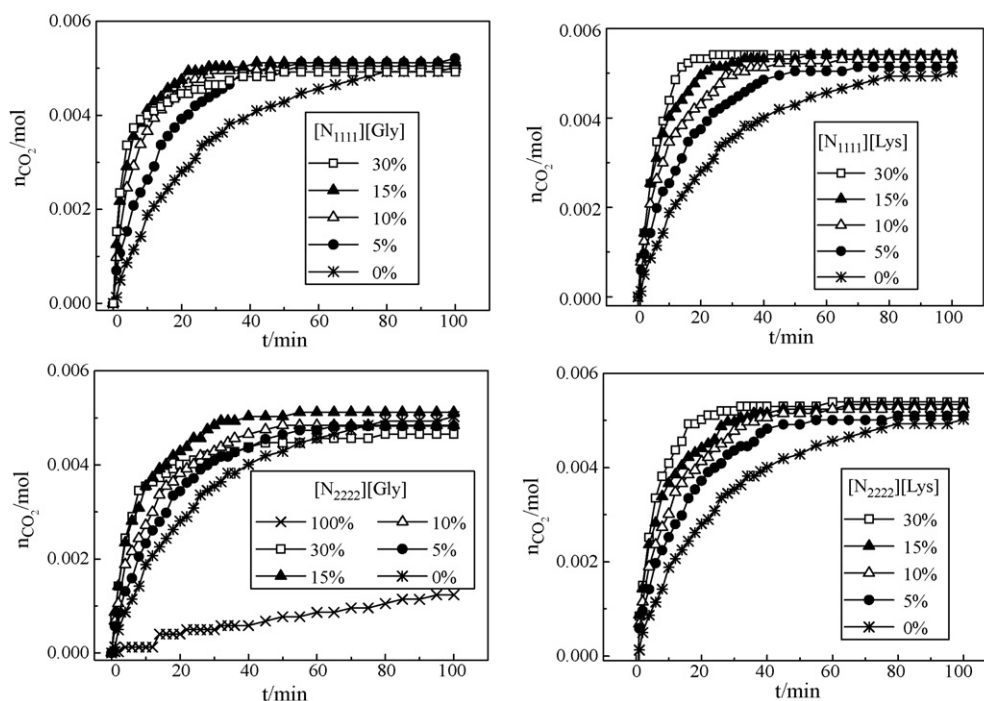


Fig. 4. Absorption amount of CO₂ in aqueous solution of IL + MDEA with 30% total amines.

Table 2
Absorption amount of aqueous solutions of [N₁₁₁₁][Gly].

IL (wt%)	Weight (g)	ρ (g cm ⁻³)	n_{IL} (mol)	n_{CO_2} (mol)	α (mol _{CO₂} mol _{IL} ⁻¹)
100	4.0162	1.08618	0.0271	0.00458	0.169
80	3.9994	1.07469	0.0216	0.00546	0.253
65	4.0238	1.06001	0.0177	0.00548	0.311
50	4.0265	1.04338	0.0136	0.00546	0.402
30	4.0515	1.02282	0.0082	0.00492	0.601

The absorption of CO₂ in MDEA-IL aqueous solutions of total 30% amines (MDEA + amino acid IL) was performed also at $P_0 = 97$ kPa, $T = 298$ K. As shown in Fig. 4, adding the ILs greatly accelerated the absorption of CO₂ in MDEA aqueous solution. Noticeably, for [N₁₁₁₁][Gly] and [N₂₂₂₂][Gly], the solutions with 15% IL and 15% MDEA had higher absorption rate and larger uptake capacity than other IL + MDEA solutions of 30% total amines. Due to the high viscosity, the absorption of CO₂ in pure [N₂₂₂₂][Gly] was very slow and insufficient in 100 min. For [N₁₁₁₁][Lys] and [N₂₂₂₂][Lys], it seemed that the outstanding absorbents were also the aqueous solutions of 15% IL and 15% MDEA, though the absorption into the solution having 30% IL was more rapid in the first 30 min. The full absorption capacities of four 15% IL + 15% MDEA solutions are compared in Table 3, indicating that lysine based ILs could absorb more CO₂ than glycine based ILs. The amount of absorbed CO₂ in aqueous solution of 15% MEA + 15% MDEA, was similar to that of 15% [N₁₁₁₁][Lys] + 15% MDEA, while the absorption load was 0.363. The absorption of CO₂ by glycine salt has been investigated in detail, exploring that the absorption performances of glycine salts was better than those of MEA [12,17].

Table 3
Absorption capacity of aqueous solutions of 15%IL + 15%MDEA.

Absorbent	m (g)	μ (mPas)	ρ (g cm ⁻³)	n_{CO_2} (mol)	α (mol _{CO₂} mol _{amines} ⁻¹)
15%[N ₁₁₁₁][Gly] + 15%MDEA	4.0144	2.90	1.0247	0.00512	0.562
15%[N ₂₂₂₂][Gly] + 15%MDEA	3.9963	2.87	1.0225	0.00511	0.643
15%[N ₁₁₁₁][Lys] + 15%MDEA	4.0158	3.15	1.0266	0.00541	0.694
15%[N ₂₂₂₂][Lys] + 15%MDEA	3.9970	3.15	1.0230	0.00534	0.740
15%MEA + 15%MDEA	4.0176	3.27	1.0181	0.00542	0.363

According to the Damping-Film Theory [18], for the isothermal absorption of gas, the relationship between partial pressure of the gas versus the time could be described as follows:

$$\ln \frac{P_0 - P_e}{P - P_e} = ut \quad (12)$$

where P represents the partial pressure of CO₂ at time t . P_0 and P_e were denoted to the partial pressure at the beginning and the absorption equilibrium, respectively. u stands for the apparent absorption rate constant. With the help of state equation of ideal gas, Eq. (12) will be written in the form of n , the amount of CO₂:

$$\ln \frac{n_0 - n_e}{n - n_e} = Kt \quad (13)$$

where K was also the apparent absorption rate constant as well as u . In Fig. 5, the slopes of the curves stood for the apparent absorption rate constants for 15% IL + 15% MDEA aqueous solutions. Obviously, the apparent absorption rates of IL and MDEA aqueous solutions were much higher than those of MDEA aqueous solution, revealing the reinforcement of the ILs on the absorption of CO₂ in MDEA aque-

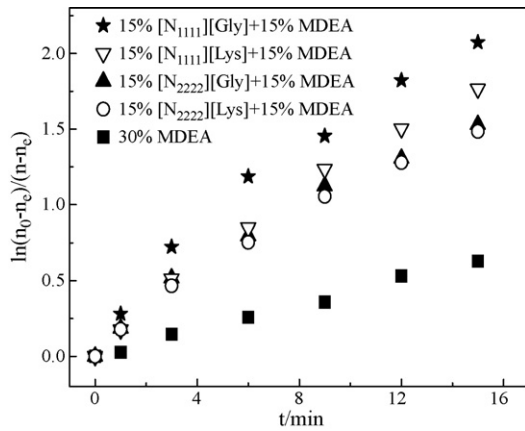


Fig. 5. Apparent absorption rates of the absorbents.

ous solution. The absorption rates of $[N_{1111}][AA] + MDEA$ aqueous solutions were higher than those of $[N_{2222}][AA] + MDEA$ aqueous solutions, probably due to the larger molar concentration of $[N_{1111}][AA]$. In the same way, since the molar concentration of $[cation][Gly]$ was larger than that of $[cation][Lys]$ under the same weight, the absorption rate of $[cation][Gly]$ was higher than $[cation][Lys]$.

4.2. CO_2 solubility in aqueous solutions of IL and MDEA

The solubility of CO_2 in aqueous solutions of IL + MDEA is illustrated in Fig. 6, showing that under low equilibrium pressure (<25 kPa), the absorption capacity of the four solutions were very close to each other. Due to the smallest molecule weigh, there were more $[N_{1111}][Gly]$ molecules in the solution than other ILs of same weight, thus $[N_{1111}][Gly]$ could absorb more CO_2 under the low pressure of the sweep gas. The absorption capacity of lysine based IL + MDEA aqueous solutions was much larger than that of glycine based IL + MDEA aqueous solutions when the equilibrium pressure was over 25 kPa, resulting from the two amine groups in one lysine

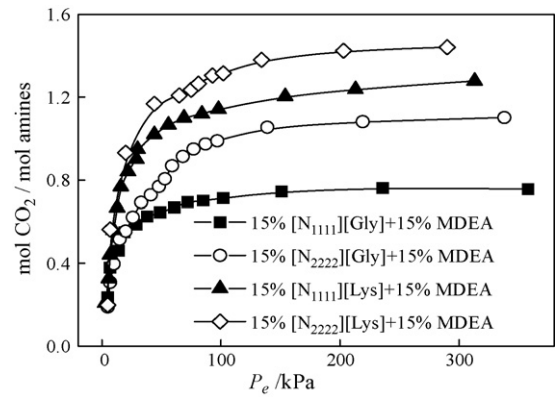


Fig. 6. Solubility of CO_2 in aqueous solutions of IL and MDEA at 298 K.

molecule. As shown in Fig. 6, under the same weight, glycine based ILs and lysine based ILs were suitable for the absorption of CO_2 under low pressure and high pressure, respectively. The solubility of CO_2 in aqueous solution of amino acid salt or mixed amines had been theoretically investigated [12,16,19,20]. For the aqueous solution of IL and MDEA, the CO_2 load and species concentrations could be calculated from the equilibrium constants of Eqs. (5)–(10), mass balance of CO_2 and amine, and charges balance. Since some important parameters, including Henry coefficient, binary interaction parameters were not available, the model for the solubility and kinetics of CO_2 uptake was not reported in the present work.

4.3. Effect of temperature and insoluble gas on the absorption

Portugal et al. [15] had revealed that temperature ranging 293–313 K had little influence on the absorption of CO_2 in aqueous solutions of potassium glycinate. As shown in Fig. 7, temperature (298–318 K) could slightly affected the absorption of CO_2 in ILs + MDEA solutions. For aqueous solutions of glycine ILs + MDEA, the higher temperature led to the larger reaction rate and less viscosity (higher diffusivity), thus the absorption rate increased with

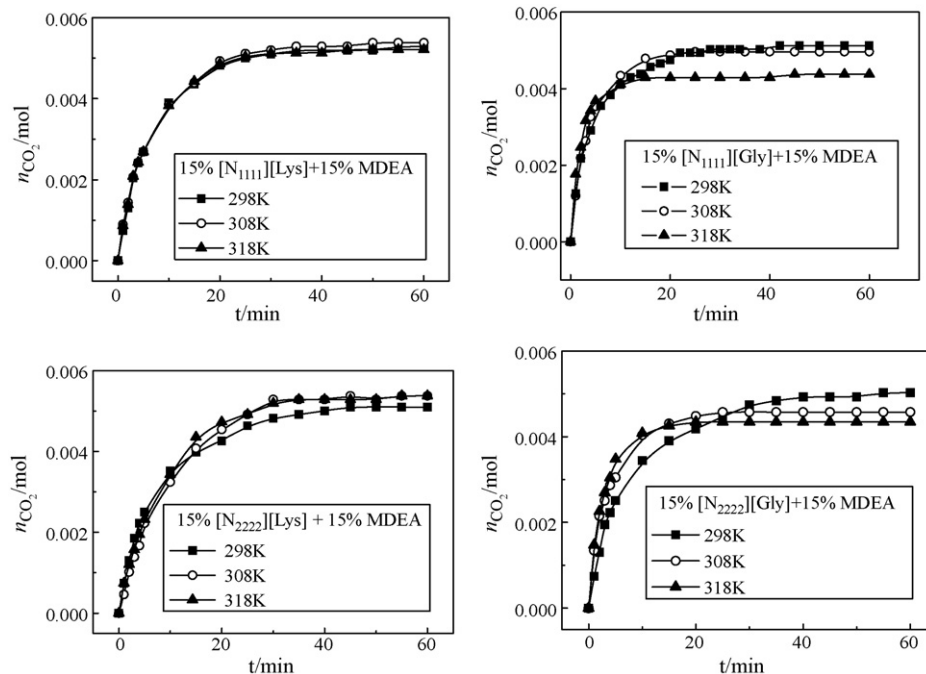


Fig. 7. Effect of temperature on the absorption of CO_2 into 15% IL + 15% MDEA aqueous solutions.

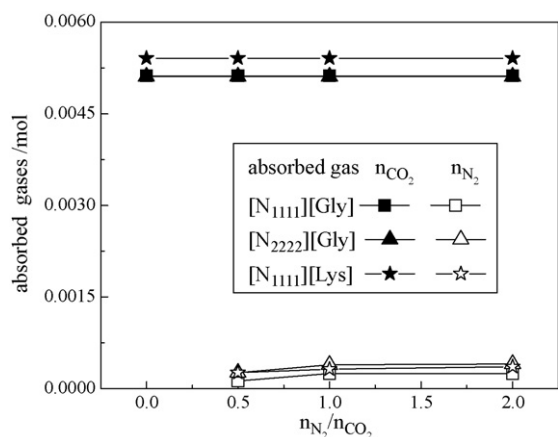


Fig. 8. Absorption of N_2 into 15% IL + 15% MDEA aqueous solutions.

a rise in temperature in the first 20 min. With a further increase of the absorption time, the higher temperature led to less absorbed amount of CO_2 .

In the industrial process, CO_2 was usually mixed with N_2 , thus the selective absorption of gas mixture into the absorbents was very important. By keeping the partial pressure of CO_2 as 97 kPa and $T = 298$ K, as displayed in Fig. 8, the absorbed N_2 (inert gas) into the aqueous solutions of IL + MDEA was very small.

4.4. Regeneration of absorbent solutions

The regeneration efficiencies of the solutions are compared in Fig. 9, showing that the regeneration performance was ranked in the following order: $[N_{1111}][Gly] > [N_{1111}][Lys] > [N_{2222}][Gly] > [N_{2222}][Lys]$ for the IL + MDEA aqueous solutions. It should be noted that aqueous solution of 15% $[N_{1111}][Gly]$ + 15% MDEA had significant regeneration efficiency (over 98%). Considering the incomplete desorption of CO_2 under 4 kPa, the aqueous solution of 15% $[N_{1111}][Gly]$ + 15% MDEA could be used repeatedly as a good absorbent. For the ILs with same anion, the regeneration efficiency of the solutions with $[N_{1111}]^+$ was higher than that of the solutions with $[N_{2222}]^+$ [10].

The properties and behaviors of the amino acid ILs in water were similar to those of amino acid salt with the metal cations, such as potassium, and sodium. For the aqueous solution of amino acid salt, although CO_2 reacts directly with amino acid anions, the salt cation may have some influence on the absorption or desorption

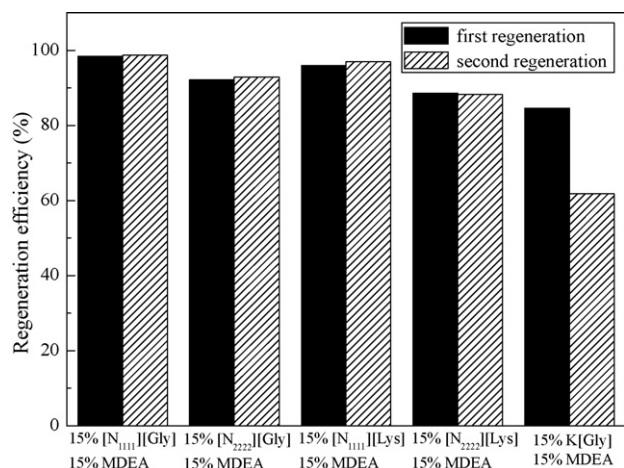


Fig. 9. Regeneration efficiencies of 15% IL + 15% MDEA aqueous solutions.

process at high solution concentration (about 3.0 mol dm^{-3}), since the short-range interactions of salt cation with other ions were different [12]. In the present work, the IL + MDEA aqueous solutions kept as solution in the absorption or regeneration. For potassium glycinate + MDEA aqueous solutions, the precipitation was found in the regeneration, resulting in low regeneration efficiency as shown in Fig. 9.

5. Conclusion

In this work four functionalized amino acid ILs: tetramethylammonium glycinate ($[N_{1111}][Gly]$), tetraethylammonium glycinate ($[N_{2222}][Gly]$), tetramethylammonium lysinate ($[N_{1111}][Lys]$) and tetraethylammonium lysinate ($[N_{2222}][Lys]$) were mixed with aqueous solution of MDEA to form a new type of absorbent for the CO_2 capture. It had been found that adding amino acid IL greatly reinforced the CO_2 absorption of MDEA aqueous solution. Aqueous solution with 15% IL + 15% MDEA had higher absorption rate and larger uptake capacity than other IL + MDEA aqueous solutions of total 30% amines. Moreover, the absorption of N_2 in the 15% IL + 15% MDEA aqueous solutions was very small.

Under low pressure (< 25 kPa), the absorption capacity of the four solutions with same weight was very close. When the pressure was over 25 kPa, the absorption capacity of lysine based IL + MDEA was much higher than that of glycine based IL + MDEA. Moreover, the regeneration performances of the IL + MDEA aqueous solutions were ranked in the following order: $[N_{1111}][Gly] > [N_{1111}][Lys] > [N_{2222}][Gly] > [N_{2222}][Lys] > [K][Gly]$. It should be noted that aqueous solution of 15% $[N_{1111}][Gly]$ + 15% MDEA has significant regeneration efficiency (over 98%).

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