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Absorption characteristics of new solvent based on a blend of AMP and 1,8-diamino-*p*-menthane for CO_2 absorption

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Abstract

Aqueous 1,8-diamino-*p*-menthane (KIER-C3) and commercially available amine solutions were tested for CO_2 absorption. A 2-amino-2-methyl-1-propanol (AMP) solution with an addition of KIER-C3 showed 9.3% and 31.6% higher absorption rate for CO_2 than the AMP solution with an addition of monoethanolamine (MEA) and ammonia (NH_3), respectively. The reaction rate constant for CO_2 absorption by the AMP/KIER-C3 solution was determined by the following equation: $k_{2,AMP/C3} = 7.702\times10^6 \exp(-2248.03/T)$. A CO_2 loading ratio of the AMP/KIER-C3 solution was also 2 and 3.4-times higher than that of the AMP/NH_3 solution and the AMP/MEA solution, respectively. Based on the experimental results, KIER-C3 may be used as an excellent additive to increase CO_2 absorption capability of AMP.

Key words: carbon dioxide; absorption rate and capacity; 1,8-diamino-*p*-menthane; 2-amino-2-methyl-1-propanol

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Introduction

Global warming has become one of the most complicated issues in the world. Carbon dioxide (CO_2) is recognized as a major greenhouse gas generated in large quantities from various emission sources including fossil-fuel electric power plants, synthetic ammonia industries, steel production works, etc. (Gadalla et al., 2005). Although a number of techniques have been tested for the separation of CO_2 from fossil fuel fired flue gas, an economically viable process is not currently available. Therefore, a more efficient CO_2 recovery process still needs to be developed.

Many studies have been conducted to develop more efficient alkanolamine absorbents for CO_2. A zwitterion mechanism was proposed for the CO_2 absorption mechanism of the alkanolamine absorbent by Caplow (1968) and Danckwerts (1979). Different from monoethanolamine (MEA), sterically hindered amines form unstable carbamates due to the bulkiness of the carbon groups attached to the amino group. The hydrolysis of carbamates then forms bicarbonate and restores the free amines which can react with additional carbon dioxide (Sartori and Savage, 1983). A representative sterically hindered amine, 2-amino-2-methyl-1-propanol (AMP) has therefore a higher CO_2 loading capacity than MEA (Dawodu and Meisen, 1994).

Using a single amine can cause relatively fast corrosion and degradation reactions (Choi et al., 2009). Hence more effective and consistent absorption for CO_2 is expected by using a blend of two or more amines (Horng and Li, 2002). A number of studies related to the kinetics of CO_2 absorption into a blend of aqueous amine solutions containing AMP have been conducted (Lee et al., 2008; Aroonwilas and Veawab, 2004; Xu et al., 1991). The blended amine solutions containing showed significant enhancement in the absorption capacity and absorption rate for CO_2 maintaining the characteristics of sterically hindered amines (Li and Chang, 1994).

In this study, KIER-C3 was examined for its possibility as an additive to AMP for CO_2 absorption using a lab-scale reactor. Reactivity of KIER-C3 with CO_2 was evaluated comparing with other additives such as MEA and NH_3 in terms of CO_2 absorption rate, reaction kinetics and CO_2 loading. Its performance was also tested under various operating conditions.

1 Materials and methods

1.1 Materials

AMP, MEA, NH_3 and 1,8-diamino-*p*-menthane (KIER-

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C3) were used in this study as CO_2 absorbents. We obtained 95% AMP and 70% KIER-C3 from Sigma Aldrich (USA), 99% MEA from Yakuri Pure Chemicals (Japan), and 30% NH_3 from Junsei Chemical (Japan). Each solution was diluted with distilled water to a certain concentration. The purity of CO_2, N_2 and N_2O gases in each cylinder was 99.9%.

1.2 Apparatus and procedure

A schematic diagram of the experimental set up is presented in Fig. 1. Absorption experiments were carried out using an agitated glass reactor with an internal diameter of 7.3 cm and a height of 15.1 cm. Each solution of 150 mL was located inside the reactor at 313 K and agitated at 50 r/min. N_2 and CO_2 gases were premixed in the mixing chamber and then injected into the solution. The flow rates of N_2 and CO_2 gases were controlled using mass flow controllers (5850E, Brooks Instruments, USA, accuracy: ± 1% of full scale) to maintain the CO_2 concentration of 15%. The flow rate of effluent gas from the reactor was determined using a bubble meter. The CO_2 absorption rate was then determined from the difference between the flow rates of the influent and the effluent gas. Each experiment was repeated three times.

The molar flux of CO_2 into each solution j (N_j kmol/(m^2·sec)) was determined from the initial absorption rate, $V(t_1)/t_1$ using the following equation:

$$N_j = \frac{P_T - P_W^0}{SRT}\frac{V(t_1)}{t_1} \quad (1)$$

where, P_T (kPa) is the atmospheric pressure, P_W^0 (kPa) is the vapor pressure, S (cm^2) is the gas-liquid contact area, R ((L·atm)/(mol·K)) is the gas constant, and T (K) is the temperature.

Another experimental system was constructed to test the amount of CO_2 absorption into each solution at equilibrium as shown in Fig. 2. A reactor with a height of 160 mm was located in a temperature-controlled vessel. Four baffles with 5 mm width were installed inside the reactor. A two-blade impeller (70 mm × 20 mm) was also installed in the middle of the liquid level. The temperature inside the reactor was measured with a K-type thermocouple with an accuracy of ± 0.1 K. The stirring speed was limited at no more than 50 r/min to keep the gas-liquid interface planar and smooth. Each solution of 300 mL was located in the reactor, and its absorption capacity was determined with a CO_2 partial pressure in the range of 0–600 kPa at 313 and 383 K, respectively. The CO_2 partial pressure in the reactor was recorded using a pressure data logging system (PR2000, Madgetech, USA). The CO_2 loading of each solution was calculated according to the following Eqs. (2)–(4):

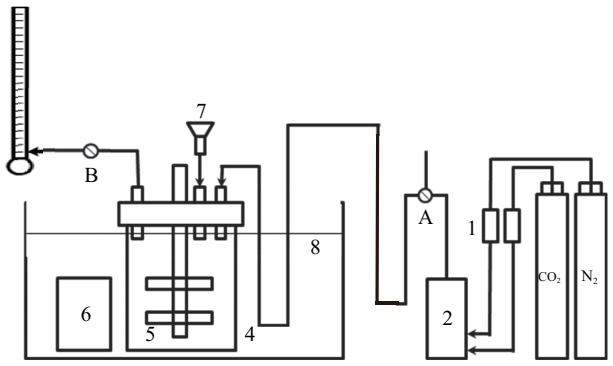

Fig. 1 Schematic diagram of the agitated vessel. (1) mass flow controller; (2) mixing chamber; (3) soap bubble meter; (4) reactor; (5) impeller; (6) liquid bottle; (7) funnel; (8) water bath.

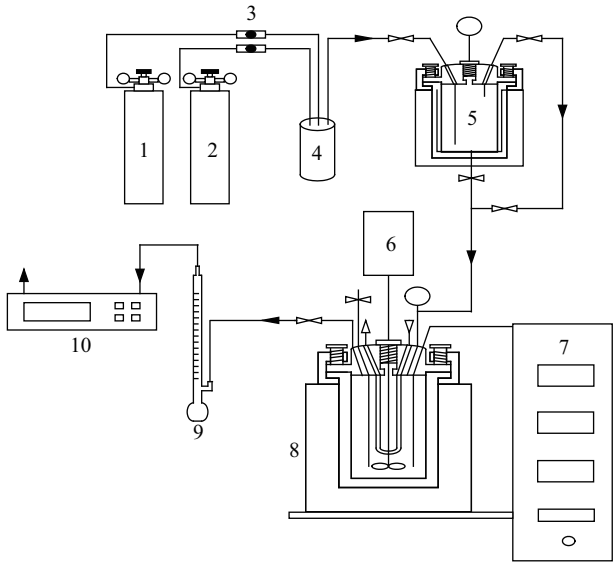

Fig. 2 Schematic diagram of experimental system used to determine CO_2 adsorption capacity. (1) N_2 cylinder; (2) CO_2 cylinder; (3) mass flow controller; (4) mixing chamber; (5) saturator; (6) magnetic drive; (7) controller of temperature and rpm; (8) reactor in a vessel; (9) soap bubble meter; (10) analyzer.

$$n_{\text{object gas}} = [(P_{N_2+\text{object gas}} - P_{eq})V]/RT \quad (2)$$

$$P_{\text{object gas}} = (P_{eq} - P_{N_2}) \quad (3)$$

$$\alpha = n_{\text{object gas}}/\text{mole of amine} \quad (4)$$

where, $n_{\text{object gas}}$ is the amount of object gas, $P_{\text{object gas}}$ is the partial pressure of object gas, P_{eq} is the equilibrium pressure, P_{N_2} is initial pressure and α is the CO_2 loading ratio.

2 Results and discussion

2.1 Solubility

CO_2 is physically soluble in an aqueous alkanolamine solution without chemical reaction. The physical solubility of CO_2 was determined from the solubility of N_2O in each solution because CO_2 reacts with the amine solution. Each KIER-C3 solution with different concentrations of 0.05, 0.10, 0.15 and 0.20 kmol/m^3 was added into a 2 kmol/m^3 AMP solution. Henry's constant (H) for N_2O on each AMP solution with an addition of KIER-C3 was determined at various temperatures of 293, 303, 313 and 323 K and then converted to the constant for CO_2 in the solution using Eq. (5).

$$(H_{CO_2})_{\text{amine}} = (H_{N_2O})_{\text{amine}} \times (H_{CO_2}/H_{N_2O})_{\text{water}} \quad (5)$$

As shown in Fig. 3, Henry's constant decreased with

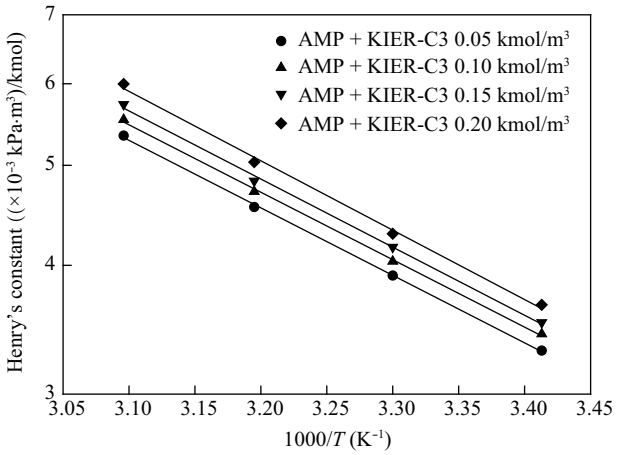

Fig. 3 Henry's constants for CO_2 in AMP/KIEP-C3 solution with different KIER-C3 contents as a fuction of temperature.

increasing temperature and the concentration of KIER-C3 in the AMP/KIER-C3 solution. Therefore, the solubility of CO_2 in the AMP/KIER-C3 solution was found to increase with an increase in the concentration of KIER-C3.

2.2 Diffusivity

Diffusivity (D_A) of CO_2 in the AMP/KIER-C3 solution was also obtained from determining diffusivity of N_2O in the solution at the temperatures of 293, 303, 313 and 323 K by using Eq. (6).

$$(D_{CO_2})_{amine} = (D_{N_2O})_{amine} \times (D_{CO_2}/D_{N_2O})_{water} \quad (6)$$

Detailed information on the wetted wall column used to determine the diffusivity is found in our previous publication (Choi et al., 2009). As shown in Fig. 4, diffusivity of CO_2 in the AMP/KIER-C3 solution decreased with increasing KIER-C3 content for all tested temperatures. This may be attributed to the increase in the viscosity of the solution with increasing KIER-C3 content.

2.3 Absorption rate

The CO_2 absorption rate was determined for AMP, MEA, NH_3 and KIER-C3 with various concentrations of 0.5, 1.0, 1.5 and 2.0 kmol/m^3 at the temperature of 313 K and

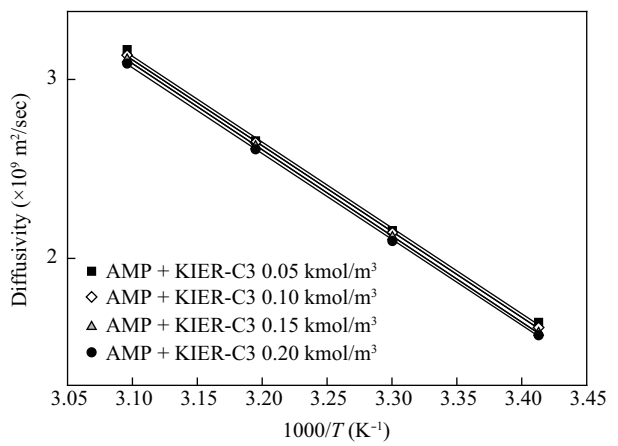

Fig. 4 Diffusivity of CO_2 in AMP/KIER-C3 solutions with different KIER-C3 contents at 2 kmol/m^3 AMP as a function of temperature.

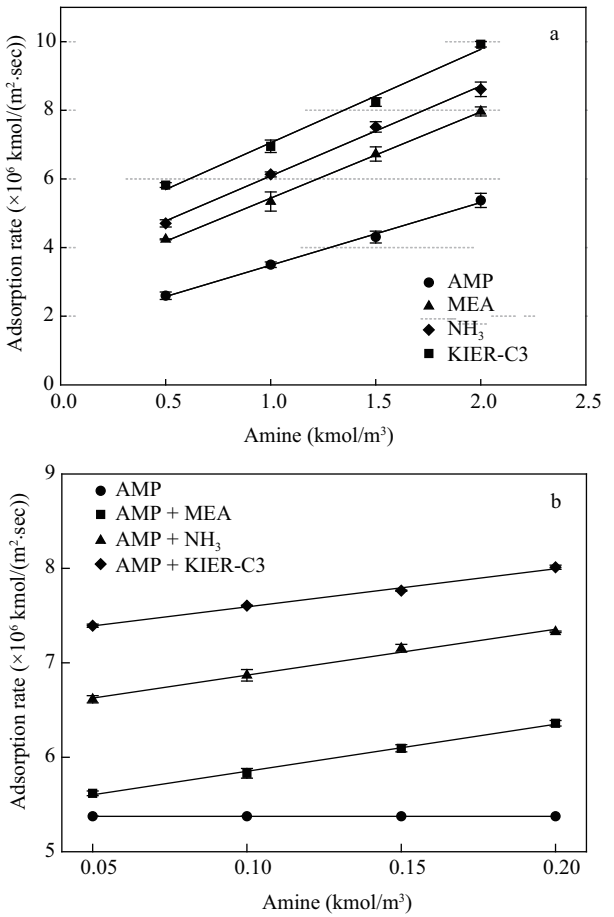

Fig. 5 Absorption rates of the CO_2 into (a) aqueous AMP, MEA, NH_3 and KIER-C3 and (b) AMP/amine solutions as a function of the concentration of each amine solution at 313 K.

the CO_2 partial pressure of 15 kPa. Figure 5a shows the absorption rate as a function of the concentration of each solution. The absorption rate increased with increasing concentration of each solution. This is caused by enhanced mass transfer in the gas-liquid interfacial area with increasing concentration. The absorption rate of CO_2 into 2 kmol/m^3 KIER-C3 solution was found to be 84.7%, 24.6% and 15.2% higher than that into 2 kmol/m^3 AMP, MEA and NH_3, respectively. As hydrolysis of carbamate, which is created via reaction of KIER-C3 and CO_2, is accelerated, and CO_2 within carbamate is converted to bicarbonate, the concentration of carbamate gets lower. However, since concentration of the reactant amine increases, those will be involved in absorption reaction of CO_2, and stimulates absorption to adjust lower concentration of carbamate and higher amine to equilibrium concentration, which leads to a higher reaction rate.

Figure 5b shows the CO_2 absorption rates of 2 kmol/m^3 AMP with different concentrations of additives at 313 K. The absorption rates were found in the range of 5.62×10^{-6} to 8.01×10^{-6} kmol/(m^2·sec). The absorption rate of the solution with an addition of KIER-C3 was 9.3%–31.6% higher than the absorption rates of other solutions with MEA and NH_3. Therefore, KIER-C3 showed the highest absorption rate for CO_2 and the best performance as an additive into AMP out of the absorbents tested in this study.

2.4 Effect of the KIER-C3 concentration on CO_2 absorption

The adsorption of CO_2 in AMP solution with an addition of KIER-C3 was tested at different temperatures. In addition to the total absorption rate of the AMP/KIER-C3 solution, the absorption rates of the AMP portion and the KIER-C3 portion in the AMP/KIER-C3 solution were determined respectively as presented in Fig. 6.

It was observed from Fig. 6 that the absorption rates into the KIER-C3 portion ($N_{KIER\text{-}C3}$) increased from 1.28×10^{-6} to 4.12×10^{-6} kmol/(m^2·sec) and 1.96×10^{-6} to 6.52×10^{-6} kmol/(m^2·sec) with increasing KIER-C3 concentration at the temperature of 293 and 323 K, respectively. But the absorption rate into the AMP portion (N_{AMP}) decreased with increasing KIER-C3 concentration. As a result, $N_{KIER\text{-}C3}$ was as much as 55.7% of the total absorption rate (N_{total}) at the KIER-C3 concentration of 0.2 kmol/m^3.

2.5 Reaction kinetics

Reaction rate constants (k_2) for the absorption of CO_2 into AMP and AMP/KIER-C3 were determined by plotting overall reaction rate constant (k_{ov}, m^3/(kmol·sec)) with the absorbent concentration at 293, 303, 313 and 323 K. By using Eq. (7), k_{ov} can be calculated as follows:

$$k_{ov} = k_{mn} = (N_A H_A^{(m+1)/2} / P_A^{(m+1)/2} / D_A^{1/2})^2 \quad (7)$$

where, N_A (m^3/(kmol·sec)) is the overall absorption rate, H_A ((kPa·m^3)/kmol) is the Henry's constant, P_A (kPa) is the partial pressure, D_A (m^2/sec) is the diffusivity of CO_2, m is the order of reaction with respect to species A, and n is the order of reaction with respect to CO_2.

The reaction of CO_2 with the AMP/KIER-C3 solution can be classified as a fast pseudo first-order reaction. We confirmed the reaction order by plotting Eq. (7) with the concentration of KIER-C3 in the solution. Figure 7 shows straight lines with a slope of 1. In addition, the k_{ov} value increased linearly with increasing the temperature and the concentration of KIER-C3 in the solution. This may be attributed to the increased diffusion at the gas-liquid interface with increasing the temperature and concentration.

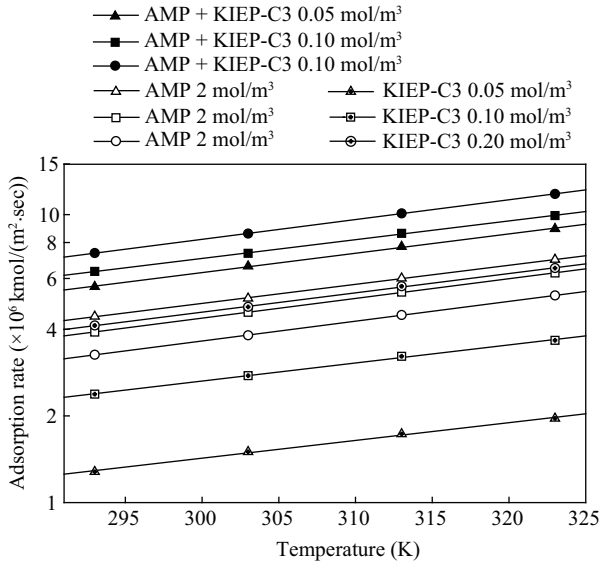

Fig. 6 Absorption rate of CO_2 into the AMP/KIER-C3 solution at different temperatures.

The reaction rate constants (k_2) were then determined for the absorption of CO_2 into AMP and AMP/KIER-C3 at different temperatures and shown in Table 1. The values of k_2 were found to be 530.98–1323.81 m^3/(kmol·sec) for AMP and 3939.53–7347.65 m^3/(kmol·sec) for AMP/KIER-C3. Hence the k_2 values for a blend of AMP and KIER-C3 were five to six times higher than that for AMP without KIER-C3. This result shows that the reactivity of AMP is significantly enhanced with an addition of KIER-C3. Therefore, KIER-C3 is considered to be an effective activator in the AMP solution. This may be attributed that KIER-C3 has two amine groups which have characteristic of sterically hindered amine. While primary amine forms stable carbamate, sterically hindered amine forms more instable cabarmate. The reduction in stability promotes hydrolysis of carbamate and CO_2 absorption. Because KIER-C3 has two sterically hindered amine groups, KIER-C3 may act as an effective activator in the AMP solution.

Figure 8 shows the correlation between reaction rate constant (k_2) and temperature using the results in Table 1. From a linear regression analysis ($r^2 = 0.99$), the following equations were obtained for the relationship between k_2 and temperature.

$$k_{2,AMP} = 1.079\times10^7 \exp(-2909.16/T) \quad (8)$$

$$k_{2,AMP/C3} = 7.702\times10^6 \exp(-2248.03/T) \quad (9)$$

From the exponents in the Arrhenius equations, activation energies for the reaction of CO_2 with AMP and

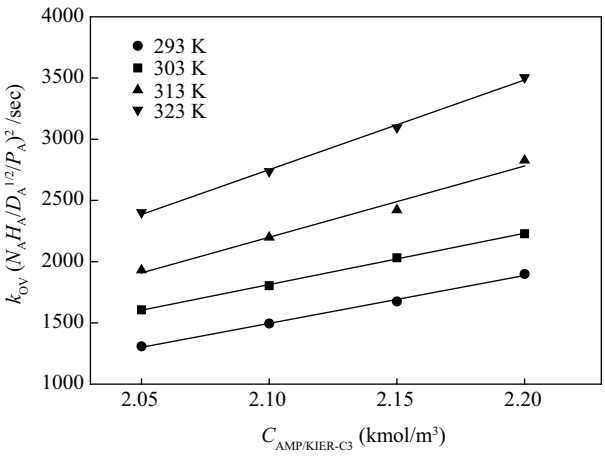

Fig. 7 Pseudo first-order overall reaction rate constant for the reaction of CO_2 with AMP/KIER-C3 solution at different temperatures.

Table 1 The second-order reaction rate constant calculated for the reaction of CO_2 into aqueous AMP and 2 kmol/m^3 AMP/KIER-C3 solutions as a function of temperature

Temperature (K)	AMP (m^3/(kmol·sec))	AMP + KIER-C3 (m^3/(kmol·sec))
293	530.98	3939.53
303	730.53	4146.09
313	992.78	5976.13
323	1323.81	7347.65

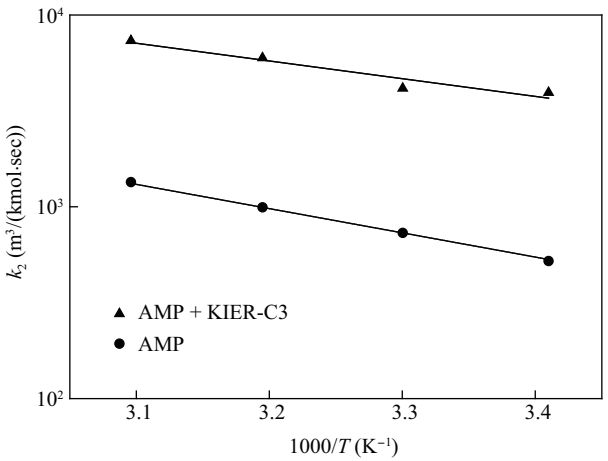

Fig. 8 Arrhenius plot for the correlation between reaction rate constant and temperature.

AMP/KIER-C3 were determined to be 24.2 and 18.7 kJ/mol, respectively. The lower activation energy for AMP/KIER-C3 also indicates that the reactivity of AMP for CO_2 is enhanced with an addition of KIER-C3.

2.6 Absorption equilibrium

The reaction of CO_2 with the primary amino group can produce three possible reactions: the formation of carbamate and bicarbonate, the reversion of carbamate to bicarbonate, or the formation of the carbonate ion (Yih et al., 1988).

$$CO_2(g) + 2RNH_2 \longleftrightarrow RNH_3^+(aq) + RNHCOO^-(aq) \quad (10)$$

$$RNHCOO^- + H_2O \longleftrightarrow RNH_2 + HCO_3^- \quad (11)$$

The equilibrium loading capacities of primary and secondary amines are limited by stoichiometry (0.5 mol of CO_2/mol of amine) of Eq. (10). The zwitterions mechanism originally proposed by Caplow (1968) and reintroduced by Danckwerts (1979) is generally accepted as the reaction mechanism for Eq. (10) (Blauwhoff et al., 1983).

$$CO_2 + RNH_2 \longleftrightarrow RNH_2^+ COO^- \quad (12)$$

$$RNH_2^+COO^- + B' \longleftrightarrow RNHCOO^- + B'H^+ \quad (13)$$

where, B′ is amine, OH^- or H_2O. The carbamates formed from primary and secondary amines are quite stable. If the carbamate ion is unstable, as in the case of a hindered amine, it undergoes the formation of bicarbonate ion as in Eq. (11). This reaction means that 1 mol of hindered amine allows loading of CO_2 up to 1 mol.

The CO_2 loading ratio is the most basic factor to evaluate the performance of absorbents. CO_2 loading in each 2 kmol/m^3 solutions of AMP, MEA, NH_3 and KIER-C3 was determined respectively with increasing CO_2 partial pressure at 313 K. The highest CO_2 loading in the AMP, MEA, NH_3 and KIER-C3 solution was 0.72, 0.82, 0.93 and 2.88 mol CO_2/mol absorbent, respectively, in the range of CO_2 partial pressure as shown in Fig. 9a. Hence KIER-C3 was found to have three to four times higher CO_2 loading than other solutions. The higher CO_2 loading in KIER-C3 may be attributed that KIER-C3 has two nitrogen compounds which have characteristics of sterically hindered amine. In addition, the CO_2 loading of more than 2 mol CO_2/mol absorbent indicates that intermediate products formed during the reaction act as the absorbents for CO_2.

In the full-scale CO_2 separation process using alkanolamine solutions, CO_2 absorption and stripping systems may be operated at the temperature range of 310–320 K and 380–400 K, respectively. To examine CO_2 loadings in blended amines at the absorption and stripping temperatures, each AMP solution with different additives was tested at 313 and 383 K. As shown in Fig. 9b, AMP/KIER-C3 shows much higher CO_2 loading at 313 K and similar CO_2 loading as the other absorbents at 383 K. Hence, AMP/KIER-C3 shows 2 and 3.4-times higher difference in CO_2 loading compared to AMP/NH_3 and AMP/MEA,

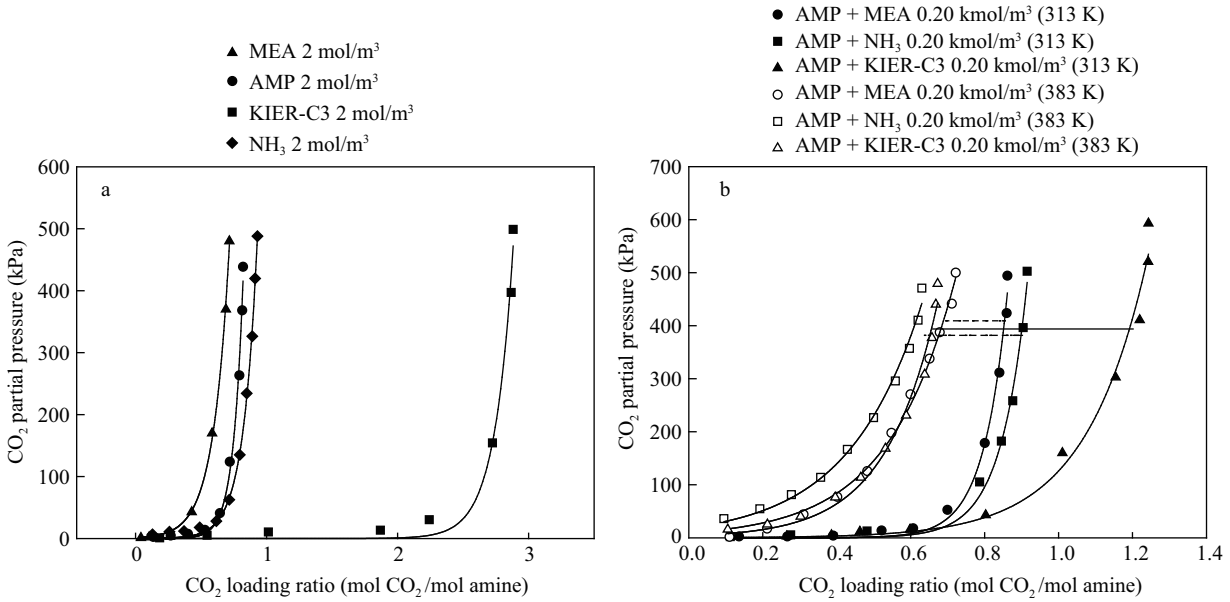

Fig. 9 CO_2 loading in AMP, MEA, NH_3 and KIER-C3 with increasing CO_2 partial pressure at 313 K (a) and in AMP with different additives at 313 and 383 K and AMP 2 kmol/m^3 (b).

respectively. This suggests that KIER-C3 can be used as an excellent additive to AMP in the CO_2 separation process.

3 Conclusions

Absorption of CO_2 into AMP/KIER-C3 was tested using a stirred batch tank reactor with different concentrations and reaction temperatures. The absorption rate into the aqueous KIER-C3 solution were 84.7%, 24.6% and 15.2% higher than that into aqueous AMP, MEA and NH_3 solution at 313 K, respectively. Also, the CO_2 absorption rate on the addition of KIER-C3 into 2 kmol/m^3 AMP solutions increased by 9.3% to 31.6% compared to the addition of MEA and NH_3 solution, respectively. This may be ascribed to the increase in diffusion and contact between the CO_2 and KIER-C3. KIER-C3 showed the best performance as an additive to increase CO_2 absorption rate of AMP. The calculated reaction rate constant for the reaction of CO_2 into aqueous AMP and AMP/KIER-C3 solution was determined to be $k_{2,\mathrm{AMP}} = 1.079\times10^7\exp(-2909.16/T)$ and $k_{2,\mathrm{AMP/C3}} = 7.702\times10^6\exp(-2248.03/T)$, respectively. In addition, activation energies of AMP and AMP/KIER-C3 were found to be 24.16 and 18.62 kJ/mol, respectively. The reactivity of AMP was enhanced with an addition of KIER-C3. CO_2 loading in a KIER-C3 solution was three to four times higher than other solutions such as AMP, MEA and NH_3. Also, CO_2 loading in the AMP/KIER-C3 solution was 2 and 3.4-times higher than that of the AMP/NH_3 solution and the AMP/MEA solution, respectively. Therefore, KIER-C3 may be used as an excellent absorbent or additive to AMP for the separation of CO_2 from flue gases.

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