Screening test for aqueous solvents used in CO₂ capture: K₂CO₃ used with twelve different rate promoters

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Abstract−K₂CO₃ solution is widely used in the CO₂-capture industry. In particular, it has advantages for treating CO₂ in flue gas under high-temperature and high-pressure conditions. However, it has a lower CO₂-loading capacity and slower absorption kinetics than those of amines, which are its major disadvantages. Thus, in this study, we investigated ten loading-rate promoters, five primary amines and five secondary amines, to develop higher CO₂-loading capacity and faster absorption kinetics. The screening tests of the absorption and desorption processes were conducted at 70 °C and 90 °C, respectively. Based on the results, we concluded that all the amines used improved the CO₂-loading and absorption kinetics compared with the use of K₂CO₃ alone. At a certain value CO₂ loading, the respective performance of the primary and secondary amines was twice and thrice better, respectively, than the neat K₂CO₃ solution. Thus, secondary amines had superior absorption capacity and absorption/desorption rate compared to primary amines. Among the secondary amines, pipecolic acid, sarcosine, and isonipecotic acid were determined as the most effective absorption rate promoters.

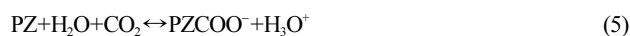Keywords: CO₂ Absorption, Rate Promoter, K₂CO₃ Solution

INTRODUCTION

Carbon dioxide (CO₂), which is known to cause the greenhouse effect, is released in large amounts during fossil-fuel combustion [1]. Consequently, the capture and storage of carbon/CO₂ has been pursued to reduce the amount of atmospheric warming [2]. In general, reagents used for absorbing CO₂ include ammonia, various amines, and K₂CO₃ [3,4]. Amine solutions are widely used because they have high CO₂-absorption rates, good solubility in water, and can be sourced at low cost. However, they not appropriate for use as absorbents in flue gases under high-temperature and high-pressure conditions, because their structures may thermally or chemically degrade, causing the desorption of flue gases [5,6]. However, it is believed that K₂CO₃ solution can be used to treat flue gases under such adverse conditions.

K₂CO₃ solution has different properties to those of amines. It has advantages for efficacious CO₂ removal from high-temperature and high-pressure flue gases, because it does not degrade under such conditions [5-7]. Thus, it is more efficient than neat amine solutions for treating flue gases at high temperatures and pressures. However, neat K₂CO₃ solution does have some disadvantages. First, K₂CO₃ solution can easily precipitate at relatively low temperatures (30 °C). Second, the minimum operating temperature should be greater than 70 °C, below which its rate of CO₂ absorption is low [3-8]. Nevertheless, K₂CO₃ can be advantageously applied to coal-fired thermal power plants [8-11]. The chemical reaction of K₂CO₃ solution with CO₂ is shown in Eq. (1).

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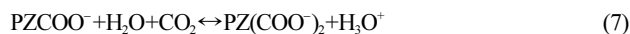$$CO_2 + K_2CO_3 + H_2O \leftrightarrow 2KHCO_3 \tag{1}$$

As previously stated, K₂CO₃ has a slow reaction kinetics and low absorption rate of CO₂ gas [12,13]. Therefore, it might be possible to improve its performance by addressing this deficiency. To do this, rate promoters such as amines can be used to compensate for the low absorption rate of K₂CO₃ [14-17]. For example, alkanolamine and amino acid salts are representative rate promoters [18]. However, alkanolamines are limited in their application because they decompose easily at high temperature or when exposed to SO$_x$ in flue gases. Therefore, they could not be applied to flue gases at high temperatures and pressures. Rate promoters other than amines are required to speed up the CO₂ absorption. Amines could be replaced as rate promoters by amino acid salts. The latter are known to show a high resistance to chemical/heat degradation. In addition, their reactivity toward CO₂ is similar to that of alkanolamine. Furthermore, amino acid salts are characterized by low vapor loss because they form ions in solution. Lastly, amino acid salts are more environmentally benign chemicals than alkanolamines. To date, many researchers have studied the interaction of amino acid salt with K₂CO₃ in solution. Most of these focus on the use of piperazine (PZ), a secondary amine, to increase the CO₂-absorption rate and capacity to more than twice of that possible when only K₂CO₃ is used [6,19-22]. Eqs. (2)-(8) describe the CO₂-absorption process in the presence of PZ.

$$CO_2 + 2H_2O \leftrightarrow HCO_3^- + H_3O^+ \tag{2}$$

$$HCO_3^- + H_2O \leftrightarrow CO_3^{2-} + H_3O^+ \tag{3}$$

$$2H_2O \leftrightarrow H_3O^+ + OH^- \tag{4}$$

$$PZ + H_2O + CO_2 \leftrightarrow PZCOO^- + H_3O^+ \tag{5}$$

$$PZH^+ + H_2O \leftrightarrow PZ + H_3O^+ \tag{6}$$

$$PZCOO^- + H_2O + CO_2 \leftrightarrow PZ(COO^-)_2 + H_3O^+ \tag{7}$$

$$H + PZCOO^- + H_2O \leftrightarrow PZCOO^- + H_3O^+ \tag{8}$$

The Sargas process was developed by Sargas Co. Ltd. to overcome the deficient absorption properties of neat K_2CO_3 solution [23]. The components of Sargas solution have not been disclosed. It has been successfully applied to CO_2 scrubbing of flue gases at high temperatures and pressures. In particular, it has been applied to CO_2 at high partial pressure in a coal-fired power plant. According to Hetland and Christensen, this process has cost advantages over other CO_2-capture processes. It has been industrialized in Europe, the US, and Japan for more than 15 years [23].

PZ has a good CO_2-absorption rate [9]. In addition, glycine and 2/4-amino-2/4-methyl butyric acid have produced similar results. Based on previous studies, adding an amino acid to a solution is generally expected to increase the latter's CO_2-loading capacity and absorption rate. This is because the $-NH_2$ group in amino acid salts imparts a weak basic character to the solution, and thus, it can more easily react with the CO_2 of the acid gas. The reactions of $-NH_2$ in amino acid salts are given in Eqs. (9)-(12). The intermediate amino acid salt structure attacks the amine on CO_2 [6,10,11]. In particular, the intermediate 2 reacts with the product derived from the sterically hindered or bulky side group of the amine-CO_2 complex. Because of these reactions, we expect the amino acid salts to act as efficient absorption rate promoters in K_2CO_3 solutions [8,24,25].

$$H_2O \leftrightarrow H^+ + OH^- \tag{9}$$

$$CO_2 + H_2O \leftrightarrow HCO_3^- + H^+ \tag{10}$$

$$RNH_2 + H^+ \leftrightarrow RNH_3^+ \tag{11}$$

$$RNH_2 + CO_2 \leftrightarrow RNHCOO^- + H^+ \tag{12}$$

Therefore, this research was conducted to study the total CO_2-absorption/desorption capacity and rate by adding various types of amino acid salts. To achieve this, ten candidate amino acid salts were selected as potential rate promoters, each of which included 30 wt% K_2CO_3 and 3 wt% of one of the following primary amino acids: arginine (ARG), 1-aminocyclohexane carboxylic acid (ACCA), histidine (HIS), 2-aminoisobutylic acid (ABA), alanine (ALA), and glycine (GLY), and 3 wt% of one of the following secondary amino acids: sarcosine (SAR), proline (PRO), pipecolic acid (PPA), PZ, diethanolamine (DEA), and isonipecotic acid (IPA). The CO_2-conversion rate was used as the major performance indicator.

EXPERIMENTAL

1. Materials

The selected amino acid salts had chemical structures similar to that PZ and GLY for ease of comparison with previous studies [Table 1]. All chemicals were purchased from Alfa-Aesar at purities greater than 98%. All the rate promoters were used at a concentration of 3 wt% and were added to a 30 wt% K_2CO_3 aqueous solution at room temperature to achieve the desired weight ratio. All the absorbents were used after mixing using a magnetic stirrer for 2 h. However, we cannot disclose the Sargas solvent components because of patent restrictions.

Table 1. Selected rate promoters of amino acid salts and its chemical structure for screening test

Chemicals	Chemical structure
Arginine	
1-Aminocyclohexane carboxylic acid	
Histidine	
2-Aminoisobutyric acid	
Alanine	
Glycine	
Sarcosine	
Proline	
Pipecolic acid	
Piperazine	
Diethanolamine	
Isonipecotic acid	

2. Experimental Setup and Procedure

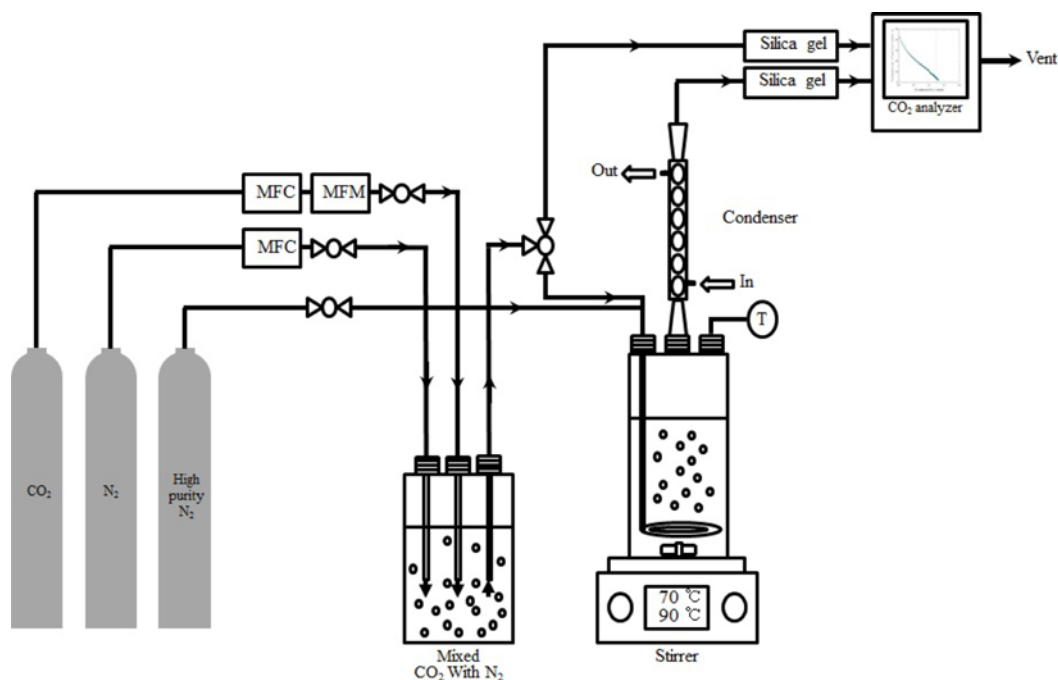This study was performed to estimate the CO_2-absorption capac-

Fig. 1. Schematic diagram for screening test with 3 wt% rate promoter+30 wt% K_2CO_3 solution.

ity and absorption/desorption rate of a K_2CO_3 solution when amino acid salts were added. The experimental results are shown in Fig. 1. The experiments are similar to those used by Song et al. (2012). The reproducibility of our results was verified to enable valid comparison with results of other studies for 30 wt% MEA solutions in a designed apparatus. Before the experiment, we synthesized a simulated flue gas of 15 vol% CO_2 with N_2 gas. The simulated gas was designed to approximate the flue gas of a coal-fired power plant, and it was controlled by a mass flow rate controller (MFC). The CO_2-flow rate was continuously monitored using an IR CO_2 analyzer (Gas Master, Woori System) with the measurement range 0-25% and a resolution of 0.01 vol% CO_2. The CO_2 and N_2 gas-flow rates were measured and controlled at 0.25 and 1.78 mL·min^{-1}, respectively.

The CO_2-absorption test consisted of the following steps. First, 400-mL solution of 30 wt% K_2CO_3 and 3 wt% amino acid salt was fed into a 500-mL Pyrex glass reactor, 7.5 cm in diameter and 16 cm in height. After feeding the absorbent into the reactor, high-purity (99.999%) N_2 gas was injected into the reactor for 20 min through a ¼-inch Teflon tube to remove the residual CO_2. With the help of continuous monitoring, we verified that CO_2 was undetectable. In the experiment, we used two glass reactors. The first reactor was used to mix the simulated gas, and it became a CO_2-saturated reactor. The second reactor was used to absorb CO_2. The conditions in each reactor were as follows. The absorption reaction temperature was set to 70 °C using a thermostatic water circulator, and the condenser was used to trap water vapor at −4 °C. After these preliminary steps, the simulated gas (15 vol% CO_2) was injected into the CO_2-saturated reactor, which was controlled by a water bath at 70 °C. Generally, the CO_2-absorption process using K_2CO_3 solution is operated at above 70 °C, because the CO_2-absorption rate of K_2CO_3 is higher. However, the purpose of our study was to study the effects of adding various rate promoters in a K_2CO_3 absorption solution. Thus, we

proposed that such a system would not require such a high temperature to absorb CO_2. Cullinane and Rochelle also performed a similar experiment at 70 °C [26]. A point of CO_2-absorption/desorption capacity and rates, therefore, seem to have enough to make a results. The simulated feed gas generated bubbles through a diffuser to ease the CO_2 absorption in the absorbent. The CO_2 concentration in the vent gas was measured at 30-s intervals by using a CO_2 analyzer. Finally, we assumed that CO_2-absorption reaction was complete when the venting CO_2 concentration was 15 vol%. After absorption, we rapidly changed the water bath temperature from 70 °C to 90 °C to initiate desorption. During desorption, we removed the residual CO_2 gas using high-purity (99.999%) N_2 gas. The desorption experiment used the same N_2-gas flow rate (1.780 mL·min^{-1}) as that used in the absorption experiment. Meanwhile, the CO_2 concentration was continuously measured using an IR CO_2 analyzer. We assumed that the desorption experiment was complete when the CO_2 concentration in the vent gas was 0%. In each experiment, high-purity N_2 gas was used to remove the residual CO_2 gas at each stage.

RESULTS AND DISCUSSION

1. 30 wt% MEA and K_2CO_3 Solution

The results of our study were compared with those of other studies to verify the comparability of our experimental apparatus. The outlet CO_2 concentration was 15 vol% as measured by the CO_2 analyzer. The input-CO_2 flow rate was fixed to 250 mL·min^{-1}, and the N_2-gas flow rate was controlled by an MFC until it matched the total CO_2 concentration of 15 vol% at the CO_2 analyzer. Therefore, we believe that the concentration was not 15 vol% when it was calculated. Fig. 2 shows a comparison of our results with those of other studies. We used 30 wt% solutions of both MEA and K_2CO_3. The latter was used as a control because many other studies also used 30 wt% MEA solution for CO_2 capture. Therefore, we obtained CO_2-
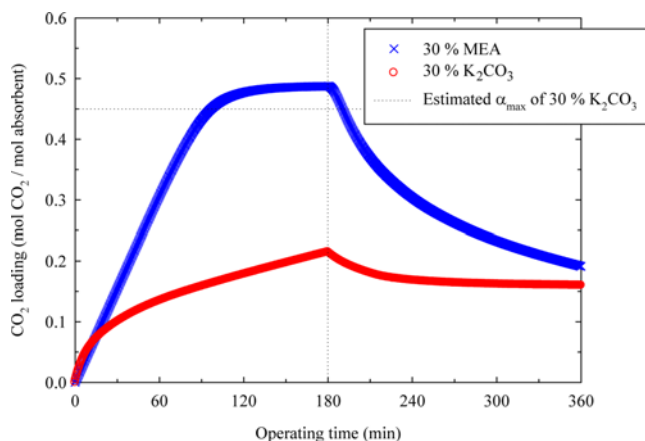
Fig. 2. Verification of experiment apparatus of 30 wt% K₂CO₃ and MEA solution.

loading values in both the MEA and K_2CO_3 solutions [Fig. 2]. Our results were similar to those of other studies: 0.76 mol CO_2/mol MEA [5], 0.483 mol CO_2/mol MEA [6], 15.74 mol CO_2/kg MEA [7], and 0.15 mol CO_2/mol K_2CO_3 [27]. Consequently, we believe that our experimental setup was appropriate for testing the selected amino
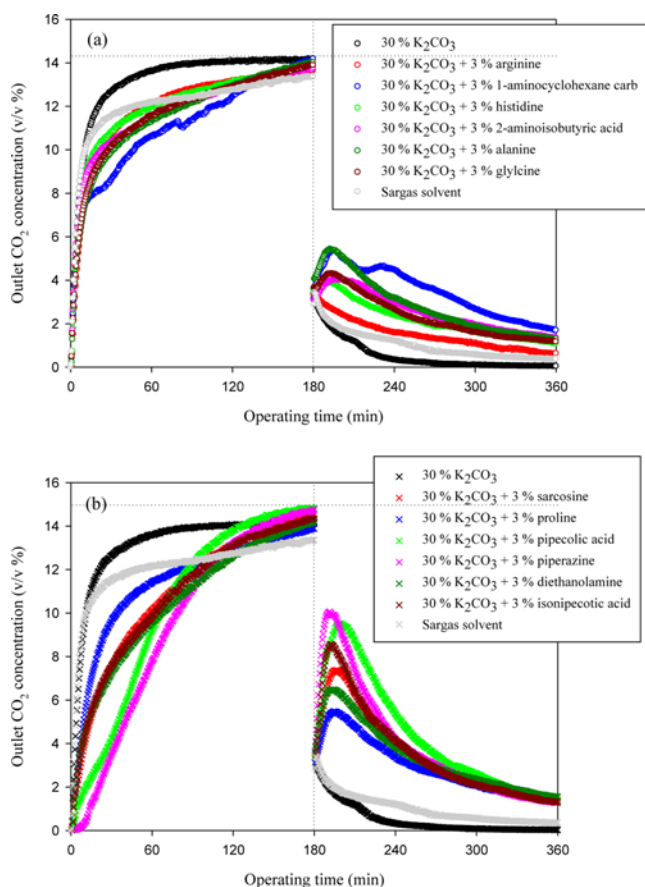

Fig. 3. The results of outlet CO₂ concentration (v/v%) at absorption/desorption process. (a) is indicated with primary amino acid salts; (b) is secondary amino acid salts. The results of K₂CO₃ and sargas solvent were included to comparing with each selected rate promoters.
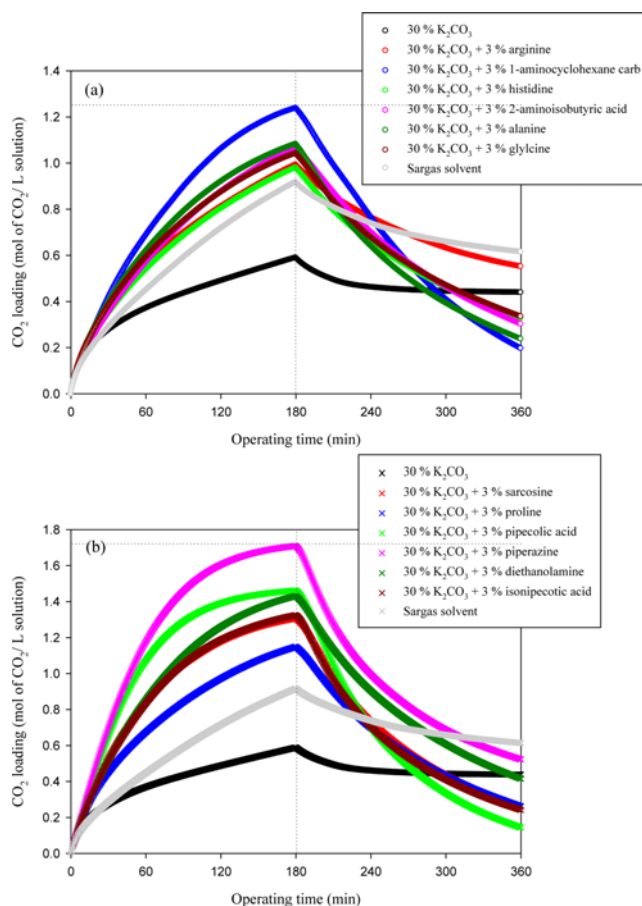
acid salts as CO_2 absorbents.

2. CO₂ Loading of K₂CO₃+Rate Promoter Solution

After verifying the experimental technique, we performed the experiments using amino acid salts. As previously stated, the absorbents were made with 30 wt% K_2CO_3 and 3 wt% rate promoters. All studies of the CO_2-absorption process required approximately 3 h until the CO_2 analyzer showed 15 vol% CO_2 concentration. The desorption process also required 3 h to balance the absorption experiment. All CO_2-absorption/desorption results are shown in Fig. 3.

Total CO_2 loading (mol/L)

$$= \left\{ \frac{\sum \text{feed } CO_2 \text{(mol/s)} \times \left[\dfrac{\begin{array}{c}\text{setup } CO_2 \text{ gas concentration (\%)} \\ -\text{vent } CO_2 \text{ gas concentration (\%)}\end{array}}{15\%} \right] \times \text{time (s)}}{\text{volume of solvent (L)}} \right\} \quad (13)$$

The results of the calculation are indicated in Fig. 4(a), (b). The

Fig. 4. The results of CO₂ loading curves with 3 wt% rate promoter+30 wt% K₂CO₃ solution. (a) is primary amino acid salts and (b) is secondary amino acid salts.

rich/lean CO_2-loading values are different. However, the net cyclic capacity allows a comparison of the rate promoters with each other. In particular, the secondary amino acid salts perform better than the primary amino acid salts, with the exception of 3 wt% proline. It is possible that the superior performance of the latter is caused by its chemical structure [6]. This is because PRO has the lowest CO_2-absorption capacity (1.149 mol $CO_2 \cdot L^{-1}$ solution) among the selected secondary amines. Similarly, the secondary amines are generally better than the primary amines except for ACCA. Among the secondary amines, 3 wt% added PPA has the highest CO_2-absorption capacity over the same reaction time.

According to Fig. 4(a), the absorption mixtures with added primary amino acid salts were approximately twice of those using K_2CO_3 only. It means that primary amino acid could increase the CO_2-loading capacity to promote CO_2 absorption. Thus, amino acid salts have significant potential as absorption-rate promoters. As shown in Fig. 4(a), CO_2 loading values (mol $CO_2 \cdot L^{-1}$ solution) followed the order: ACCA>ALA>ABA>GLY>ARG>HIS. Among the primary amino acids, ACCA has the largest CO_2-absorption capacity in terms of its molar carbon dioxide concentration. ALA>ABA>GLY>ARG>HIS. In particular, the CO_2-absorption capacity of GLY (1.038 mol of

CO_2/L solution) of the patented solution is better than that of K_2CO_3 (0.586 mol of CO_2/L solution) and that of Sargas solvent (0.914 mol of CO_2/L solution), but is less than those of ACCA (1.2373 mol of CO_2/L solution) and ALA (1.082 mol of CO_2/L solution).

In addition, ACCA shows the highest CO_2-desorption capacity compared with the other primary amino acids under the same conditions of CO_2 desorption. However, in the K_2CO_3 control case, the CO_2-desorption capacity is not significantly different compared with those where a rate promoter is added. This was because of a low temperature (90 °C) during desorption, whereas the general K_2CO_3-desorption temperature is more than 120 °C [28]. However, we suppose that our process has lower energy consumption than the general K_2CO_3-desorption process when total respective CO_2 capacities, as well as their respective absorption and desorption rates, are compared.

The absorption performances of most of the used secondary amino acid salts were far superior to those of the primary amino acid salts tested. As shown in Fig. 4(a), the CO_2 loadings of the secondary amino acid salts followed the order: PZ>PPA>DEA>IPA>SAR>PRO. In particular, the addition of 3 wt% PZ solution to 30 wt% K_2CO_3 solution achieved a CO_2 loading of 1.706 mol of CO_2/L solution. The CO_2 loading of K_2CO_3/PZ solution was more than three times of that of the K_2CO_3 control solution. PZ also had a high desorption capacity. However, PZ has been applied by many researchers and has limited accessibility in patents. Therefore, among the
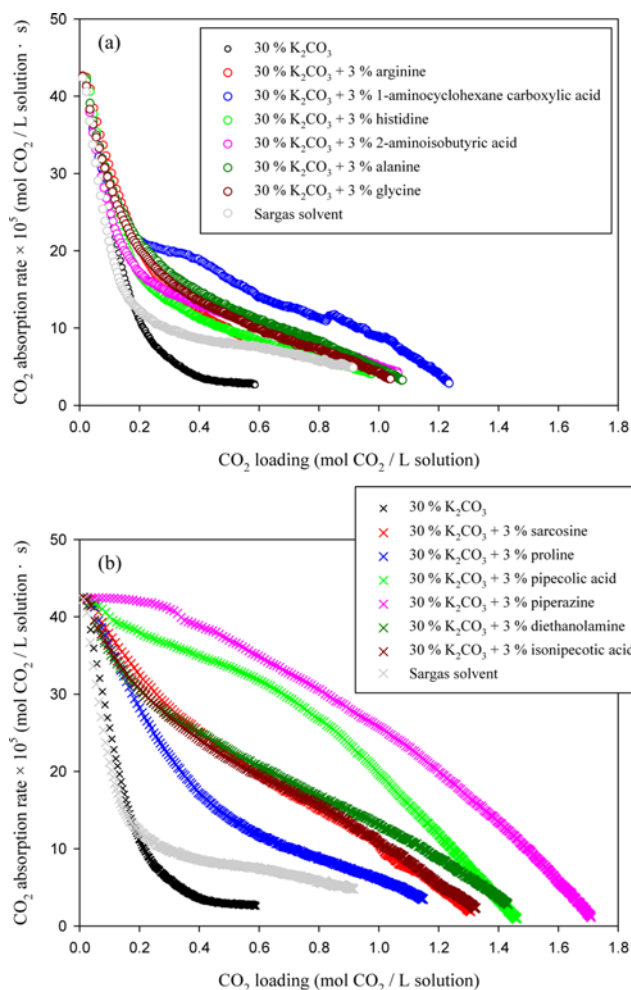

Fig. 5. The results of CO_2 absorption rate (mol of CO_2/L solution·s) vs CO_2 loading values. (a) is primary amino acid salts and (b) is secondary amino acid salts.
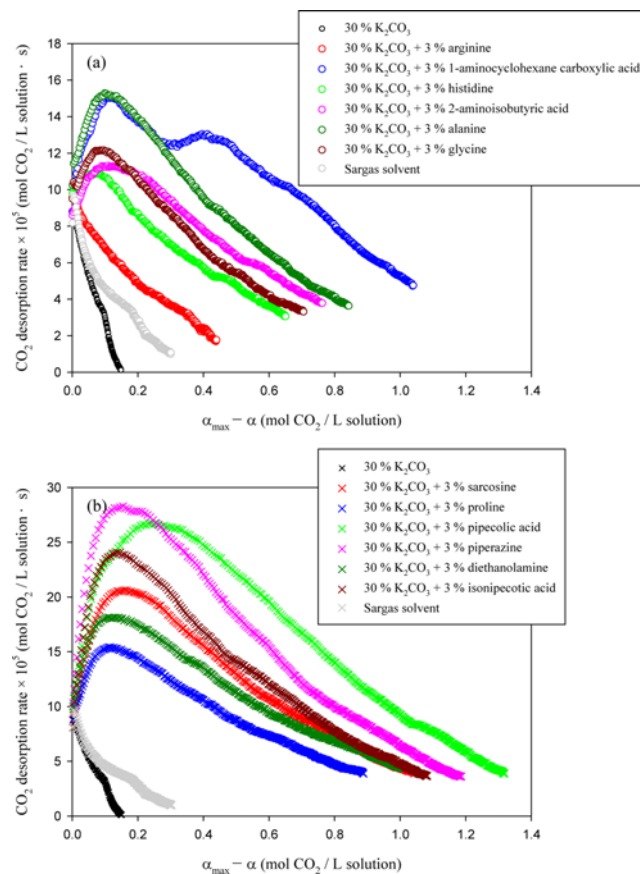

Fig. 6. The results of CO_2 desorption rate (mol of CO_2/L solution·s) vs $\Delta\alpha$ (mol of CO_2/L solution). (a) is primary amino acid salts and (b) is secondary amino acid salts.

remaining secondary amino acid salts, we find that PPA provides the best absorption performance on the basis of its CO_2 loading.

3. Rate of CO_2 Absorption and Desorption of K_2CO_3+Rate Promoter Solution

In Figs. 5 and 6, various CO_2-absorption/desorption rates (mol of CO_2/L solution·s^{-1}) are presented. It has a similar form. Each CO_2-absorption/desorption loading is divided by the reaction time (s) and multiplied by a factor of 10^5 because of its relatively low value. Consequently, similar trends can be seen in the CO_2-loading results. In the case of primary amino acids, ACCA has the biggest CO_2-absorption loading, which is more than twice that of the K_2CO_3 control when assumed to operate over the same time. The CO_2 loading of Sargas solvent is different from those of the primary amino acid salts. Nevertheless, it still shows good absorption efficiency and the CO_2-absorption rate is not significantly different from those of the primary amino acid salts.

The secondary amino acid salts also show similar results to those of the primary amines. In particular, there is a significant contrast between the respective CO_2-absorption rates of K_2CO_3 and Sargas solvent. Although its CO_2 loading is less than that of PZ, PPA possesses sufficient advantages as a rate promoter, because its absorption rate decelerates with time. Although DEA has CO_2 capacity similar to that of PPA, its CO_2-absorption rate rapidly decreases early in the reaction. Thus, SAR is better than DEA in terms of its optimal CO_2 loading and absorption rate.

Absorption/Desorption rate (mol of CO_2/L solution·s)

$$= \frac{CO_2 \text{ loading (mol/L)}}{\text{time (s)}} \times 10^5 \qquad (14)$$

In terms of the desorption rate, ACCA showed the best performance compared with the other primary amino acid salts. In addition, K_2CO_3, Sargas solvent, and ARG showed rapid absorption rates in comparison with the total CO_2-loading values. These absorption/desorption rates were relatively lower than those of the others. Apart from K_2CO_3, Sargas solvent, and ARG, the primary amino acid salts also showed similar results. Therefore, ACCA and ALA were highly efficient with regard to their respective desorption rates, $\Delta\alpha$.

The secondary amino acids showed different performance than that of the primary amino acid salts. ARG, a primary amino acid, showed a similar result to that of K_2CO_3. However, the secondary amino acids were not similar in performance to the K_2CO_3 solution. All of the secondary amino acid salts tested desorbed CO_2 early in the desorption process. In particular, PZ and PPA performed well in terms of their respective absorption/desorption conversion rates as their desorption rates were high. Consequently, we find that secondary amino acid salts, when added in small amounts as rate promoters to a K_2CO_3 absorption solution, are superior to primary amino acids, both in terms of their respective CO_2 capacities and their absorption/desorption rates.

CONCLUSION

We performed a screening test of various amino acid salts as CO_2-absorption rate promoters in K_2CO_3 solutions. The latter, when used without rate promoters, are known to have low CO_2-loading values and low absorption/desorption rates. Based on our results, various amino acid salts were effective in increasing the CO_2-absorption rate. Some of the rate promoters used in this study also enhanced the CO_2-loading value of the K_2CO_3 solutions. However, the amino acid salts were only effective at temperatures greater than 70 ºC. Among the amino salts, the secondary amino acid salts were distinguished by imparting excellent CO_2 loadings and significantly higher absorption/desorption rates to K_2CO_3 solutions than those achieved by the addition of primary amino acid salts. As expected, the performance of PZ was better than that of the other selected amino acid salts. However, we thought that PPA was better as a rate promoter than PZ, because the latter has two NH groups in its chemical structure, whereas the former has only one [Table 1]. Therefore, we supposed that PPA would perform better than the other rate promoters under the same operating conditions. Thus, we proposed that the performance of the optimum rate promoters followed the order: PPA> SAR>IPA. Finally, the use of a small amount of a rate promoter

Table 2. CO_2 loading values of each 3 wt% rate promoter in 30 wt% K_2CO_3 solution

Absorbents		Rich CO_2 loading (mol of CO_2/L solution)	Lean CO_2 loading (mol of CO_2/L solution)	Net cyclic capacity (mol of CO_2/L solution)
K_2CO_3		0.589	0.438	0.151
Sargas solvent		0.917	0.614	0.303
Primary amino acid salts	ARG	0.992	0.551	0.441
	ACCA	1.237	0.196	1.041
	HIS	0.979	0.327	0.652
	ABA	1.066	0.302	0.764
	ALA	1.082	0.237	0.845
	GLY	1.041	0.334	0.707
Secondary amino acid salts	SAR	1.307	0.26	1.047
	PRO	1.149	0.262	0.887
	PPA	1.462	0.144	1.318
	PZ	1.709	0.526	1.183
	DEA	1.432	0.415	1.017
	IPA	1.325	0.242	1.083

such as an amino acid salt in K$_2$CO$_3$ solution is highly effective in assisting the CO$_2$ absorption.

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