

Experimental Investigation on the Performance of [APMIm][NTf₂] for Capturing CO₂ from Flue Gas of the Cement Kiln Tail

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Abstract: Facing the global warming trend, humanity has been paying more and more attention to the Carbon Capture, Utilization and Storage. Large amounts of CO₂ is emitted with burning fossil fuel as well as by some special industrial processes like the decomposition of calcium carbonate in a cement plant. The cement industry contributes about 7% of the total worldwide CO₂ emissions and the CO₂ concentration of flue gas of the cement kiln tail even exceeds 30%. Ionic liquid is considered to be an effective and potential material to capture CO₂. In order to investigate the performance of ionic liquids for capturing CO₂ from flue gas of the cement kiln tail, an experiment system was established and an ionic liquid, [APMIm][NTf₂] (1-aminopropyl-3-imidazolium bis(trifluoromethylsulfonyl)imide), was tested using pure CO₂ and simulated gas. The results showed that both physical and chemical absorption play roles while physical absorption dominates in the absorption process. Both the absorption capacity and rate decrease with raising the operating temperature. In the experiment with pure CO₂, the absorption capacity is 0.296 mol_{CO₂}·mol_{IL}⁻¹ at 30°C and 0.067 mol_{CO₂}·mol_{IL}⁻¹ at 70°C. Meanwhile, the ionic liquid can be regenerated for recycling without obvious changes of the absorption capacity. When the ionic liquid is used for flue gas of the cement kiln tail rather than pure CO₂, a sharp decrease of the absorption capacity and rate was observed obviously. The absorption capacity at 30°C dropped even to 0.038 mol_{CO₂}·mol_{IL}⁻¹, 12.8% of that for pure CO₂. Additionally, a natural desorption of CO₂ from the ionic liquid was observed and affected the experimental results of the absorption capacity and the absorption-desorption rate to some extent.

Keywords: ionic liquid, CO₂ absorption, Carbon Capture Utilization and Storage (CCUS), flue gas of the cement kiln tail

1. Introduction

Nowadays, global warming, a global problem has been attracting more and more attention [1]. As one of the most important greenhouse gases, CO₂ coming from

the industry production becomes a threat to sustainable development and even humanity [2, 3]. It is a common and main component in most waste gases emitted. Cement industry contributes about 7% of the total worldwide CO₂ emissions and the CO₂ concentration of

Nomenclature

<i>D</i>	percent deviation
<i>M</i>	molar mass/g·mol ⁻¹
<i>m</i>	mass/g
<i>p</i>	performance
\bar{p}	average performance
<i>q</i>	quantity/mol _{CO₂} ·mol _{IL} ⁻¹
<i>r</i>	absorption or desorption rate/mol _{CO₂} ·mol _{IL} ⁻¹ ·min ⁻¹

τ	time/min
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Abbreviations

IL	ionic liquid
RT	room temperature/°C

Subscript

abs-des	absorption and desorption
<i>i, j</i>	1, 2, 3, ...

flue gas of the cement kiln tail even exceeds 30% [4]. It is efficient to capture CO₂ from flue gas with high CO₂ concentration [5, 6] and this work can deliver substantial environmental, economic and social benefits to the society, and increasingly, to the humanity [7].

Alkanolamine solutions are common CO₂ absorbents, but some disadvantages, such as high re-production cost, serious secondary pollution and serious corrosion, limit their applications [8–10]. In contrast, ionic liquids (ILs) that are a group of organic salts composed of organic cations and a variety of anions have efficient performance for separating CO₂ from mixture gas and can furthermore overcome the problems of the alkanolamine solutions in potential applications [11–13]. A lot of work about materials synthesis, laboratory experiments, molecular simulation and phase equilibrium predictions has been carried out [14]. The absorption capacity of ionic liquid for CO₂ can be improved by inputting a basic group –NH₂, which was verified by comparing the solubility of CO₂ in 6 imidazole ionic liquids before and after amino functionalization [15]. Zhang et al. [16] innovatively put the amino group into the anion of the ionic liquid by a neutralization reaction and pointed out the CO₂ absorption rate of a porous SiO₂ supported ionic liquid is fast and the reaction is reversible. It was also indicated that ionic liquids can absorb equimolar amounts of CO₂ in the presence of water with the concentration of 1 wt%, but absorption mechanism differs from that of anhydrous ionic liquids. Based on the above approach, Zhang et al. [17] prepared a series of 20 dual amino-functionalised phosphonium ionic liquids, (3-aminopropyl)tributylphosphonium amino acid salts and found that the supported absorption of CO₂ reaches equilibrium within 80 min and the chemical absorption of CO₂ approaches 1.0 mol_{CO₂}·mol_{IL}⁻¹. Under certain conditions, Wu et al. [18] tested the solubility of CO₂ in three kinds of ionic liquids, [emim][FAP], [bmim][FAP] and [hmim][FAP], and pointed out that [hmim][FAP] had best absorption performance for CO₂ at 5 MPa and 20°C.

High viscosity, which is a disadvantage of the ionic liquids for capturing CO₂, causes high power consumption and harms the diffusion and solution of CO₂.

In order to decrease the energy consumption and increase the absorption rate, it is necessary to synthesize ionic liquids with low viscosity besides using supported absorption. Li et al. [19] carried out experimental study on the CO₂ absorption performance of three low viscous protic ionic liquids. Xiao et al. [20] gave the variation of the viscosity of four ionic liquids with temperature. Kaisy et al. [21] synthesized several ammonium based ionic liquids containing carboxylic anions. Yang et al. [22] paid attention to the effect of temperature, pressure and water concentration on the absorption capacity of [APMIm]Br and claimed that an obvious improvement was observed in the absorption capacity with the mass concentration of water exceeding 60.84%. Additionally, the temperature and pressure showed a negative and positive relation with the absorption capacity, respectively. Maiti [23] proposed a theoretical approach to evaluate the absorption performance of the ionic liquid and thought [NTf₂]⁻ has a high efficiency in dissolving CO₂.

Many researchers contributed to the absorption performance of amounts of ionic liquids by using pure CO₂. However, in actual applications, the gas is usually a mixture and the aim is to fix the mixture or separate them. Therefore, it is significant to study the absorption performance of ionic liquids with flue gas mixtures. Mohammadali et al. [24] investigated the solubility of CO₂, H₂S and their mixture in an ionic liquid by experiments. Barati-Harooni et al. [25] developed four models to predict the solubility of the CO₂ and H₂S in ionic liquids. Fu et al. [26] pointed out that the CO₂ partial pressure and then the concentration of the insoluble gas influence the absorption capacity and rate. Althuluth et al. [27] studied the permselectivity of CO₂/CH₄ using supported ionic liquid membrane. Ramdin et al. [28] also gave the solubility of CO₂/CH₄ gas mixture in ionic liquids. Shiflett et al. [29, 30] paid attention to the separation of N₂O and CO₂ using room-temperature ionic liquids. Yang et al. [31] considered the effects of SO₂ and O₂ on the CO₂ capture using the absorbent of a mixed ionic liquid and amine solution.

The previous studies focused on the absorption

performance with pure CO₂ or simple mixtures. There are little studies that concern the CO₂ separating performance of ionic liquids for the real mixture flue gas. In view of the high CO₂ concentration of the cement kiln tail and the potential application of ionic liquids, in this article, a detailed experimental research was conducted on the absorption performance of an ionic liquid, [APMIm][NTf₂] (1-aminopropyl-3-imidazolium bis(trifluoromethylsulfonyl)imide), including the absorption capacity and rate with both pure CO₂ and a flue gas of the cement kiln tail. The effect of reusing ionic liquid on the absorption performance was another important topic in the experimental study.

2. Methodology

2.1 Experimental system

The components of the experimental system are a vacuum drying oven, a sliding vane vacuum pump, three gas cylinders, three mass flow controllers, an oil bath, a test tube and an analytic balance, as shown in Fig. 1.

The molecular formula of [APMIm][NTf₂] is C₉H₁₄N₄O₄S₂F₆. Its molecular weight is 420.35. It is yellow viscous liquid whose decomposition temperature is 381°C. The purity of the ionic liquid that used in the experiment is 98%. Some moisture and CO₂ may be

absorbed by the ionic liquid before the test, which affects the experimental result of the absorption performance of CO₂. A vacuum drying oven is used to produce a warm and vacuum environment to take out the absorbed water and CO₂ in the preparing process. In order to simulate the flue gas of the cement kiln tail, a simple gas system composed of three gas cylinders and three mass flow controllers is used to produce an adjustable mixture gas with CO₂, O₂ and N₂ as components. The absorption and desorption reactions occur in a test tube which is located in an oil bath. An analytic balance is used to test the mass variation of the reacted ionic liquid. The detailed information of the instruments and the devices can be found in Table 1.

2.2 Experimental method

Absorption experiment of CO₂ using pure CO₂ and a mixture gas was performed at the standard pressure (one atmospheric pressure) and different temperature. The ionic liquid [APMIm][NTf₂] was located in a bubble reactor with 1.0 g for the pure CO₂. In the experiment, it is found that the absorption capacity for the mixture gas is much less than that for the pure CO₂. In order to achieve satisfying measurement accuracy, 4.0 g of the ionic liquid is used for the simulated gas. The testing tube is placed in an oil bath which works at 30°C, 50°C and

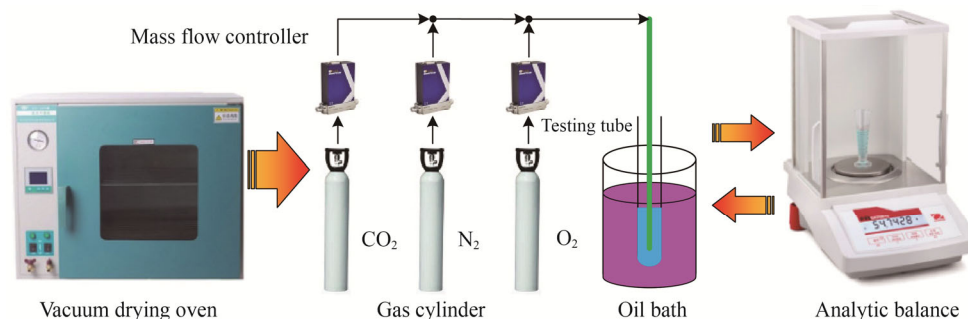


Fig. 1 Schematic diagram of the experimental system

Table 1 Information of the devices in the system

Item	Information
Vacuum drying oven	The model number is DZF-6050. Its temperature range is RT+10°C to 200°C with a resolution of 0.1°C. The vacuum degree is 133 Pa. The operating temperature range is 5°C to 40°C.
Sliding vane vacuum pump	The model number is TWX. The maximum flow rate is 60 L·min ⁻¹ and the ultimate vacuum is 2 Pa.
Gas cylinders	They supply CO ₂ , O ₂ and N ₂ with the purity of 99.99%.
Mass flow controller	The model number is CS200A. The accuracy is ±0.35% of the full span. The operating temperature is 5°C to 45°C and the maximum operating pressure should be lower than 3 MPa.
Oil bath	The maximum temperature is 300°C and the accuracy is ±1.0°C.
Analytic balance	The model is FR224CN. The upper limit of the range is 220 g and the minimum graduation is 0.0001 g. The typical settling time is 4 s.
Nuclear magnetic resonance spectrometer	It is a product from Bruker. In addition, the nuclear magnetic tubes with diameter of 5 mm from Wilmad LabGlass are used to store the sample dissolved with deuterium reagent during the nuclear magnetic resonance detection.

70°C, respectively. The mass variation of the ionic liquid is monitored by an analytic balance at regular intervals. For both the pure CO₂ and the mixture gas, the gas flow rate through the ionic liquid is kept as about 100 mL·min⁻¹. The component fraction of the simulated gas can be adjusted by the mass flow controllers. The target volume ratio for the mixture CO₂/O₂/N₂ is 0.337/0.045/0.618 which was measured data in a cement plant.

In the desorption process, a stream of pure N₂ flows through the ionic liquid and reduces the partial pressure of the dissolved CO₂, desorption occurring simultaneously. The flow rate of the pure N₂ and temperature of the ionic liquid are kept equal to that in the absorption process. The desorbing rate is obtained by measuring the mass variation at regular intervals. In addition, heating and vacuum can take the captured CO₂ out from the ionic liquid and be used in practical application. The absorbing quantity is defined as the ratio of the absorbing moles of CO₂ to the moles of the ionic liquid. The definition of desorbing quantity is similar as the absorbing quantity. Therefore, the expression of the absorbing or desorbing quantity is obtained as Eq. (1). By dividing the change of the absorbing or desorbing quantity by the time interval, the absorbing or desorbing rate can be obtained, as shown in Eq. (2).

$$q_{\text{abs-des}} = \frac{m_{\text{CO}_2, \text{abs-des}}/M_{\text{CO}_2}}{m_{\text{IL}}/M_{\text{IL}}} \quad (1)$$

$$r_{\text{abs-des}} = \frac{|q_{\text{abs-des}, i+1} - q_{\text{abs-des}, i}|}{\tau_{i+1} - \tau_i} \quad (2)$$

The weighting accuracy is 0.0001 g, so the standard uncertainty of mass of the ionic liquid is 0.0001/√3 g. According to the above equations and molecular weight of CO₂ (44.01 g/mol) and [APMIm][NTf₂] (420.35 g/mol), the standard uncertainty of absorption quantity can be obtained. If initial mass of the ionic liquid is 1.0 g, the standard uncertainty of absorption quantity is equal to 5.5×10⁻⁴ g. The value is 1.4×10⁻⁴ g for 4.0 g of the ionic liquid.

In order to study if there is an obvious change of the absorption performance in reusing the ionic liquids, the percent deviation of the absorption capacity, the maximum absorption rate and the maximum desorption rate are defined for several series of experiment. The percent deviation is the ratio of the performance difference between each value and the average value to the average value. It can be found in Eq. (3).

$$D_j = \left[\frac{|p_j - \bar{p}|}{\bar{p}} \right] \times 100\% \quad (3)$$

3. Results and Discussion

The absorption and desorption of CO₂ in ionic liquids are affected by the operating temperature and pressure. It

should be noted that the partial pressure can be decreased by mixing other gases into CO₂.

3.1 Effect of the operating temperature

In order to reveal the effect of the operating temperature on the absorption and desorption characteristic, a series of experiments were performed with pure CO₂ under one atmospheric pressure and at 30°C, 50°C and 70°C, respectively.

As shown in Fig. 2, the absorption quantity of CO₂ increases as time goes on and finally reaches a maximum in the absorption process. The absorption capacity of [APMIm][NTf₂] decreases with raising the operating temperature. The absorption capacity at 70°C is only 0.067 mol_{CO₂}·mol_{IL}⁻¹, 22.6% of that at 30°C, 0.296 mol_{CO₂}·mol_{IL}⁻¹. It can clearly be seen that the operating temperature has a large impact on the absorption capacity greatly. The absorption capacity at 50°C is 0.197 mol_{CO₂}·mol_{IL}⁻¹. The maximum absorption rate appears at the beginning of each experiment, the reason of which is that the concentration of the active components is much higher at the beginning than that in any other time. Additionally, the viscosity of the ionic liquid increases with absorbing CO₂ and then harms the diffusion of CO₂ in it. The maximum appears at 26 min, 14 min and 8 min for 30°C, 50°C and 70°C, respectively. However, that is not to say that the absorption rate increases with increasing the operating temperature. In contrast, the maximum absorption rate decreases with raising the operating temperature. The values are 0.062, 0.047 and 0.026 mol_{CO₂}·mol_{IL}⁻¹·min⁻¹ for 30°C, 50°C and 70°C, respectively. The ionic liquid at higher temperature has lower absorption capacity and it is easier to reach the absorption capacity. The absorption rate represents the absorption quantity per minute. It is one of the most important parameters for capturing CO₂. High absorption rate means smaller absorption reactor in the practical application. In addition, higher absorption capacity effects lower power consumption of the circulating pump. In a word, high absorption capacity and absorption rate should be pursued to get excellent performance in capturing CO₂ in the practical application. There is another important property named viscosity. It also impacts the power consumption and decreases with increasing the temperature of the ionic liquid [20]. Low viscosity means low power consumption of the circulating pump and compact system. Therefore, on the premise of absorption performance, low viscosity should be also pursued for selecting ionic fluid.

When the ionic liquid is saturated with CO₂, a stream of pure N₂ is led into the ionic liquid to take out the dissolved CO₂. The flow rate of the pure N₂ is about 100 mL·min⁻¹. The absorption quantity decreases towards zero with leading pure N₂ into [APMIm][NTf₂]

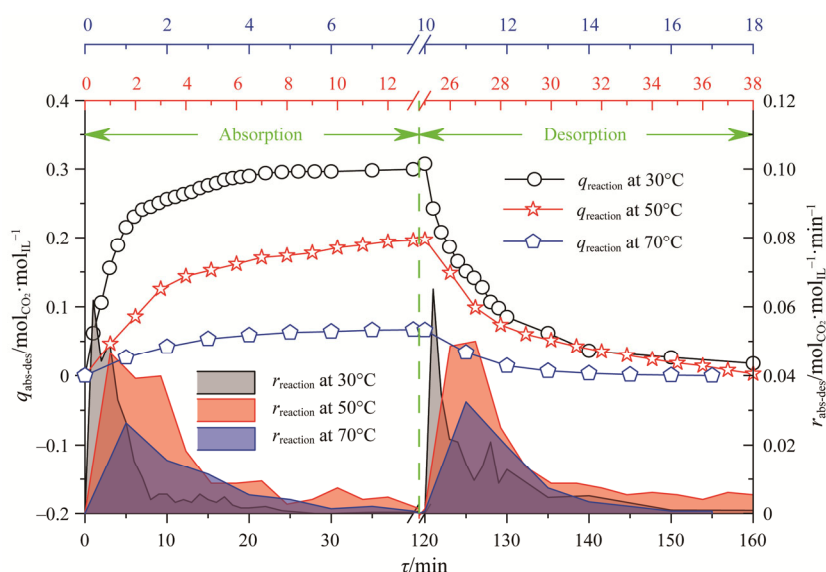


Fig. 2 The absorption and desorption characteristic with pure CO₂ at different temperature

continuously. In other words, the ionic liquid is regenerated for capturing CO₂ again. As similar with the absorption process, the maximum desorption rate appears at the beginning of the desorption process and decreases with increasing the operating temperature. The values are 0.065, 0.049 and 0.032 mol_{CO₂}·mol_{IL}⁻¹·min⁻¹ for 30°C, 50°C and 70°C, respectively. It is worth noting that the shorter the time interval is, the more accurate the absorption-desorption rate is. That means the mass of the bubble reactor should be measured as frequently as possible to get a very accurate absorption-desorption rate. In this experimental investigation, the time interval is specified as one minute.

The chemical absorption and physical absorption are two absorption processes for dissolving CO₂ using ionic liquid. The difference between them is whether CO₂ reacts with the dissolvent during the absorption process. For the chemical absorption, the absorbent can be regenerated from rich CO₂ solution in some cases and cannot in other cases depending on the property of the absorbent. In order to know if there is a chemical absorption of CO₂ in reaction, the nuclear magnetic resonance (NMR) carbon spectrums were obtained by a superconducting NMR spectrometer at 400 MHz for the initial [APMIm][NTf₂] and a CO₂-saturated sample got at 20°C. The analysis of the product was performed by 13-C NMR spectroscopy (Bruker Avance III 400 HD). To monitor the reaction, 200 μL of the mixture and 400 μL of Dimethyl sulfoxide-d₆ (DMSO-d₆) were added together and was directly configured into 13-C NMR samples for analysis.

The NMR carbon spectrums can characterize the change of the sample structure. As shown in Fig. 3, compared with the initial ionic liquid, a new resonance is observed at 158.11×10⁻⁶ for the CO₂-saturated sample,

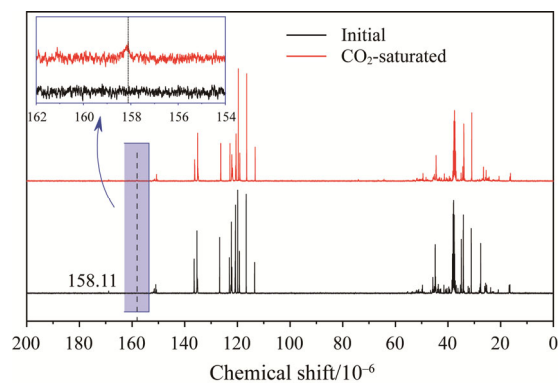
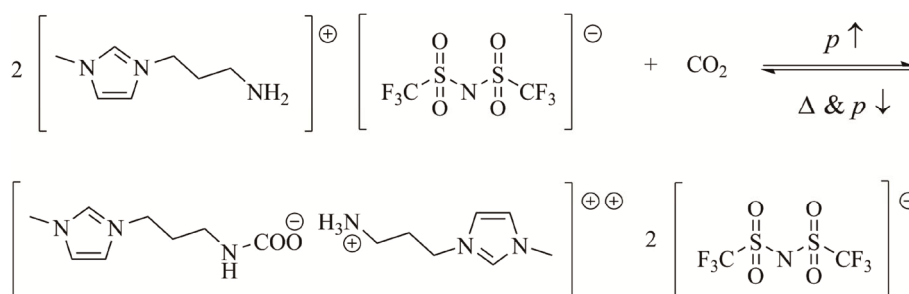
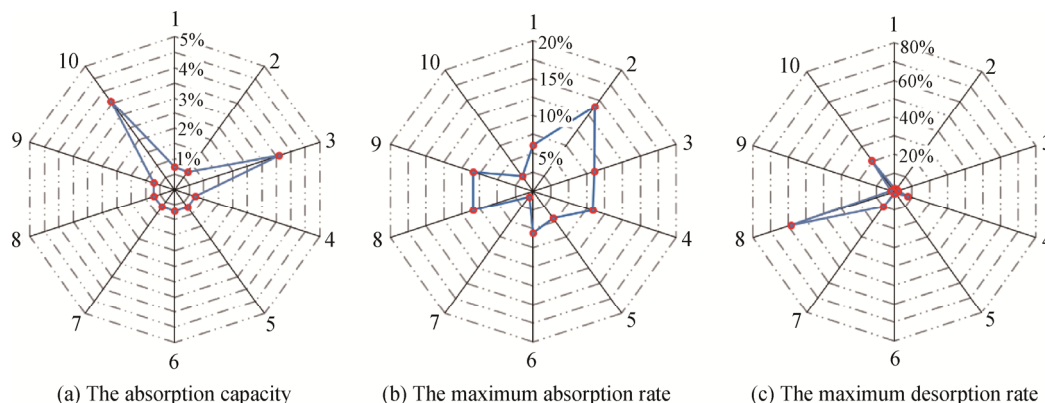


Fig. 3 The NMR carbon spectrum of the initial and a CO₂-saturated [APMIm][NTf₂]

attributable to a carbamate carbonyl carbon [32]. The new carbamate carbonyl carbon is generated from the reaction of -NH₂ and CO₂. However, it should be noted that the new peak is very low, which indicates that the chemical absorption is weak whereas the physical absorption plays a more important role. The mechanism of the chemical absorption of CO₂ is shown in Fig. 4.

3.2 Absorption performance of the regenerated absorbent

As discussed above, some absorbents can be regenerated and contribute to reduce the operating expense. The ionic liquid [APMIm][NTf₂] can be regenerated by reducing the partial pressure of CO₂. In this section, the absorption and desorption performance were studied by comparing the experimental data from a series of experiments. Though ionic liquid at low temperature showed better absorbing performance than that at high temperature, 70°C is similar as the actual condition of the flue gas of the cement kiln tail.


Fig. 4 The mechanism of the chemical absorption

Fig. 5 The percent deviation of the absorption and desorption performance of the regenerated ionic liquid

Therefore, the operating temperature of the regeneration experiments for the ionic liquid is specified as 70°C. As shown in Fig. 5(a), the absorption capacity shows very slightly variation in the ten experiments. The maximum percent deviation of the absorption capacity is about 3.6% appearing in two experiments and the value in the other experiments is about 0.7%. The two larger percent deviation may be caused by experimental error. The CO₂ absorption capacity stays almost constant with reusing the ionic liquid for ten times.

As shown in Fig. 5(b) and Fig. 5(c), the percent deviation of the maximum absorption rate and desorption rate is much larger than that of the absorption capacity. The percent deviation of the maximum absorption rate is smaller than 15% and that of the maximum desorption rate isn't larger than 10% except two values of 20.7% and 58.5%. This phenomenon shows not only the general stability of the absorption-desorption rate but also the large error in calculating the absorption-desorption rate in some conditions. If more accurate value of the absorption-desorption rate is needed, the time interval for measuring the solution mass should be reduced and the total mass of the ionic liquid should be raised. These two measures are restricted on this experimental system.

3.3 Absorption performance for the flue gas of the cement kiln tail

Based on the above analysis, it is obvious that the decrease of the partial pressure of CO₂ in the ionic liquid

causes that CO₂ comes out from the CO₂-absorbed ionic liquid. In fact, the partial pressure of CO₂ also decreases by using mixture gas like the real industrial gas. Therefore, in the actual application, the absorption capacity and absorption rate for capturing CO₂ are affected by the composition of the industrial gas. In this section, the attention is paid to the effect of a real flue gas, the flue gas of the cement kiln tail on the CO₂ absorption properties of [APMIm][NTf₂]. Same as the above experiments, this series of reactions also operate under the standard pressure and at 30°C, 50°C and 70°C.

As shown in Fig. 6, the trends of the curves for the absorption quantity and the desorption quantity are similar with that using pure CO₂ while a huge decrease is observed for the absorption capacity compared with the above results. For example, the absorption capacity at 30°C is 0.038 mol_{CO₂}·mol_{IL}⁻¹, only 12.8% of that with pure CO₂ at the same operating temperature. As shown in Fig. 4, increasing the operating pressure is helpful for enhancing the absorption. On the other hand, the reaction equation goes in the opposite direction with decreasing the operating pressure. The O₂ and N₂ in the simulated gas cannot dissolve in the ionic liquid, so the CO₂ partial pressure is decreased by mixing these two components. Correspondingly, the absorption-desorption rate is much lower than that using pure CO₂. Therefore, O₂ and N₂ in the gas play a great role in decreasing CO₂ partial pressure and further weakening the CO₂ absorption. Compared with conditions using pure CO₂, the partial

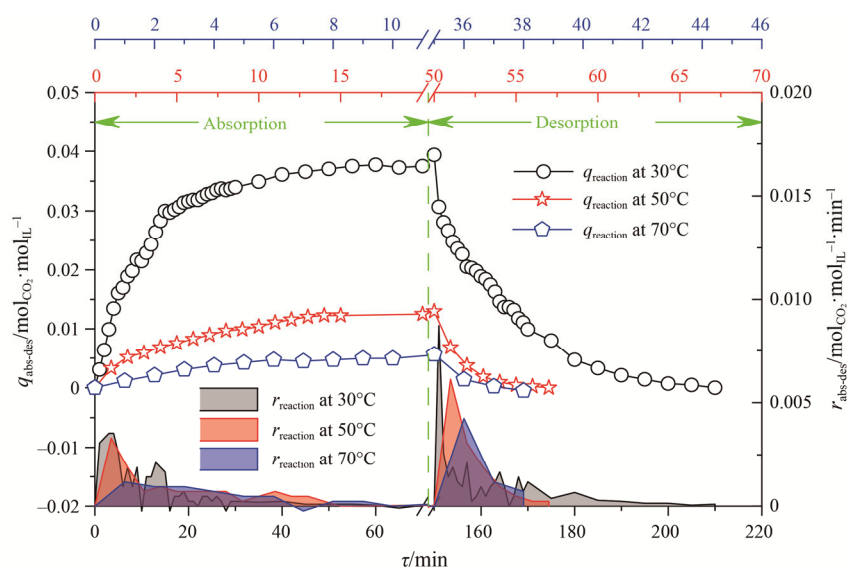


Fig. 6 The absorption and desorption characteristic with flue gas of the cement kiln tail at different temperature

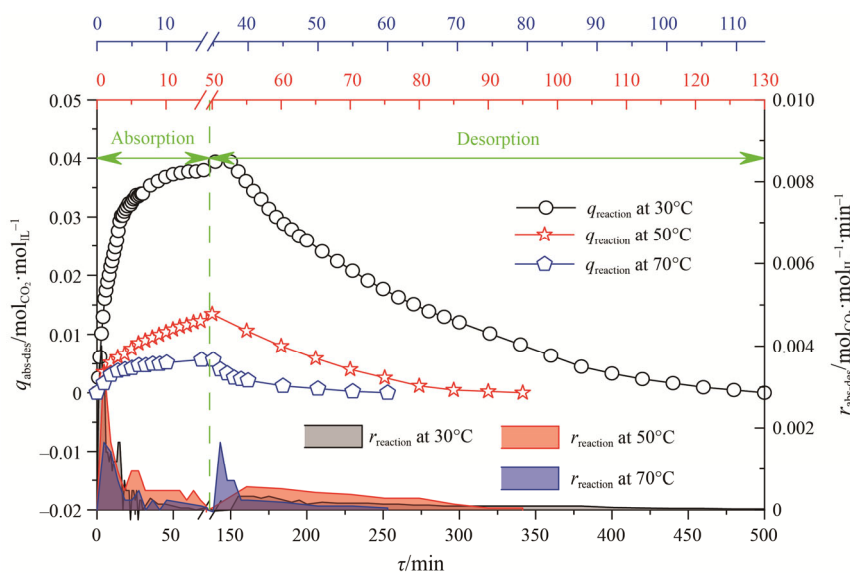


Fig. 7 The comparison between the absorption process and the natural desorption process

pressure of CO_2 in the gas stream decreases by 66.3%. A lot of research showed that the ionic fluids have good absorption performance for CO_2 . However, in the literature, pure CO_2 was used rather than the real or simulated flue gas. In addition, the operating pressure was much higher than the standard pressure in many experiments. The operating conditions are much different with the practical application. Because there is a sharp drop of the absorption performance with the real operating pressure and standard pressure, it is necessary to carry out extra experiment for the real gas.

Because the absorption capacity is very low with the flue gas of the cement kiln tail, a natural desorption phenomenon was observed. It is also indicated that a natural desorption exists during the interval time for

measuring the mass of the ionic liquid and may contribute to the desorption rate in the above experiments. Therefore, the natural desorption affects the experimental results of the absorption and desorption. Then, the natural desorption process was monitored in this section with the same parameter settings for the absorption process.

As shown in Fig. 7, the results for the absorption process are almost the same as that in the previous experiments with the simulated gas while the natural desorption for the CO_2 -saturated ionic liquid takes about twice the time for regenerating the solvent. The natural desorption may be considered as an instability of the CO_2 -absorbed ionic liquid and this instability is mainly due to the physical absorption rather than the chemical absorption. It is not a good thing for CO_2 capturing

process, because it requires that the CO₂-absorbed ionic liquid has to be transferred to the CO₂ desorbing place as soon as possible. From another perspective, the natural desorption provides an opportunity for regenerating the solvent without energy consumption. However, the desorption takes a longer time and the time may extend under high pressure. It is worth noting that the natural desorption may exist in all the series of experiments and is not obvious in conditions with high absorption capacity.

4. Conclusions

Focusing on capturing CO₂ from a CO₂-rich flue gas of the cement kiln tail, an experimental investigation was carried out on the absorption and desorption properties of CO₂ by an ionic liquid, [APMIm][NTf₂].

(1) For both pure CO₂ and mixture gas, the absorption capacity of CO₂ by the ionic liquid decreases with raising the operating temperature. The absorption-desorption rate, including the absorption rate and the desorption rate, usually shows a peak at the beginning of each process. Though the reaction at higher temperature takes shorter time, the absorption-desorption rate has a negative relation with the operating temperature.

(2) The ionic liquid, [APMIm][NTf₂], can be regenerated and reused with little change of the absorption capacity.

(3) Low partial pressure is helpful for reducing the solubility and can be used to regenerate the ionic liquid. However, this directly causes a huge decrease of the CO₂ absorption capacity for the simulated gas compared with pure CO₂. The value of the absorption capacity decreases by 87.2% from 0.296 mol_{CO₂}·mol_{IL}⁻¹ to 0.038 mol_{CO₂}·mol_{IL}⁻¹.

(4) A natural desorption of CO₂ from the ionic liquid was observed and affected the experimental results of the absorption capacity and the absorption-desorption rate. On the one hand, the natural desorption has an adverse effect on increasing the CO₂ absorption capacity and requires a quick transfer of the CO₂-absorbed ionic liquid from the absorbing place to the desorbing place. On the other hand, the natural desorption provides an opportunity for regenerating the solvent without energy consumption, though it takes a long time especially under high pressure.

Acknowledgments

Project 2016YFB0601504 supported by National Key R&D Program of China is gratefully acknowledged. The authors are also grateful for the help about the NMR test from Dr. WAN Qiang in Institute of Chemistry, Chinese Academy of Sciences.

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