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Experimental study of ionic liquids as solvent for CO_2 absorption process

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Post combustion carbon capture process is considered for the removal of CO_2 using chemical absorption method. The commercial CO_2 absorption process is being carried out with the help of Monoethanolamine (MEA) as solvent. However, MEA has a high vapour pressure and is corrosive, thus it's critical to find a different solvent, like ionic liquids. In this study, ionic liquids were utilized as a solvent for CO_2 absorption process. The ionic liquids namely Tetrabutyl ammonium acetate (TBA OAC) and Tri Butyl (Methyl) Ammonium Dicyanamide (TBMA DCA) were used in CO_2 absorption process. Additionally, utilizing ionic liquids and MEA, physicochemical parameters were determined experimentally both before and after the CO_2 absorption process. Then the effect of diffusivity and viscosity of carbon loaded solutions with various temperatures were measured experimentally. Finally, the performance of ionic liquids was compared with that of MEA and the results showed that there is no significant rise in viscosity of IL even after absorption process. From the results it revealed that due to the presence of low viscosity during CO_2 absorption, there is a chance to reduce energy demand during solvent regeneration.

Keywords: Carbon loading, Chemical absorption, CO_2 absorption, Diffusivity, Ionic liquid, Solvent regeneration

Introduction

The anthropogenic emission of CO_2 causes global warming. Rising levels of greenhouse gases in the atmosphere have a disastrous impact on the ecosystem and human well-being. Carbon dioxide (CO_2) is a major greenhouse gas, and its releases from power plants that use fossil fuels have been reported to be the primary cause to climate change[1]. According to Intergovernmental Panel on Climate Change (IPCC) of about 79% of CO_2 emissions from fossil fuels are being used for electricity generation. Coal power plants emitted 60% CO_2 and play a major contribution to the global warming[2]. CO_2 capturing and eventual storage in the ground might be a potential way to substantially decrease the release of greenhouse gasses from their sources. As a result, establishing commercially feasible CO_2 capture and separation methods is an important to diminish CO_2. Various CO_2 mitigation methods have been researched such as chemical and physical absorption, chemical looping and membrane separation[3]. The most common CO_2 separation technique is chemical absorption of acid gases with amine-based solvents. Among these, monoethanolamine serves as a best solvent in the CO_2 elimination but the use of alkanolamines having the drawbacks as high vapour loss, more energy require to regenerate the solvent and equipment damage[4]. To overcome these drawbacks an alternate potential solvent was screened to mitigate the CO_2 emissions. Nowadays current research focused on CO_2 capture using Ionic liquids due to low vapour pressure, less volatile, high chemical and thermal resistance. Because of their massive cations and anions, ionic liquids have versatile characteristics that are captivating[5]. The physical and chemical attributes of solvents vary when CO_2 is absorbed. The change in physicochemical properties affects the CO_2 absorption kinetics in the absorber thereby reducing the CO_2 absorption capacity[6]. Some of the current literatures that are available for CO_2 solubility and physicochemical properties of the cation and anion of ionic liquids (ILs) has been discussed below.

Generally, CO_2 solubility exhibits a modest rise with the increase of alkyl chain at low pressures, followed by a significant rise at elevated pressures. Aki *et al.* [7] examined the influence of cation on CO_2 dissolution by augmenting the alkyl chain length of the cation, which yielded a moderate enhancement in dissolution at high pressures. The extension of the alkyl chain leads to a larger space available for CO_2 interaction.

The solubility of CO_2 in various ILs increases with pressure and decreases with temperature. Studies on

pyridinium- and ammonium-based ILs show the longer alkyl chains enhance CO_2 solubility by strengthening cation–CO_2 interactions and weakening cation–anion interactions[8]. Fluorinated anions further improve solubility due to strong CO_2–fluoroalkyl interactions. Similar trends were observed in aqueous TBAOH, where CO_2 uptake occurs mainly through van der Waals interactions and occupation of free volume within the ionic liquid[9-10].

Wappel *et al.*[11] screened the potential solvent for solvent based CO_2 capture process. Various concentrations of aqueous IL were tested. It was found that 60 wt% IL in water performed better absorption performance compared to 30wt% MEA, pure IL and other IL concentrations (28 wt%, 43 wt%, 48 wt% and 75 wt%) because the viscosity of pure IL was very high it will affect the absorption kinetics. The viscosity of IL was reduced by adding water to the IL to improve the CO_2 absorption and solvent regeneration performance.

The density, viscosity, and surface tension of various ammonium-based ionic liquids decrease with increasing temperature due to thermal expansion[12-14]. Ethanolammonium ionic liquids showed higher viscosity because hydroxyl groups promote hydrogen bonding, while [EtOHA][BA] exhibited the highest CO_2 absorption owing to its lower viscosity[12]. Hydroxyl ammonium ILs displayed linear density reduction and nonlinear viscosity decline with temperature [14]. Surface tension decreases with temperature and is influenced by alkyl chain length and intermolecular forces during CO_2 absorption [15-16].

Moya *et al.*[17] examined the diffusivity coefficient of CO_2 in ionic liquids, recorded with a temperature of 293-323 K and a pressure of 1-20 atm, utilizing a high-pressure sorption analyzer. The results showed the diffusivity coefficient rises with higher pressure and temperature and reduces with decreasing viscosity**.** Diffusivity coefficient of CO_2 in room temperature ILs was measured at different temperature and viscosity. The results revealed that the diffusivity coefficient inversely proportional to viscosity and directly proportional to temperature. The diffusivity coefficient appeared to rise with increasing temperature and decreasing viscosity[18].

From the literature survey, it was clearly noted that existing literatures available for CO_2 absorption capacity and physiochemical properties of ammonium based ILs were not well understood by research to make Carbon Capture and Storage (CCS) as high CO_2 absorption capacity and less energy demand for solvent regeneration. On the other side the effect of diffusivity coefficient and viscosity of CO_2 in IL are not well discussed in the previous research. By considering these aspects the present study aimed to investigate the CO_2 solubility using Tetrabutylammnoium Acetate [TBA OAC] and Tributylmethylammonium dicyanamide [TBMA DCA] because these IL have not used in CO_2 capture process till now. After that, a detailed examination was done to assess the solvent's density, viscosity, pH, carbon loading, and surface tension before and after it absorbed CO_2. The diffusivity coefficient of the ionic liquid was measured at the reaction temperature in CO_2 absorption process.

Experimental Section

Materials required

The specifications of the substances utilized in this study are high purity monoethanolamine, tetrabutyl-ammonium acetate [TBA OAC purity >90.0% (TCI)], tributylmethylammonium dicyanamide [TBMA DCA purity >98.0% (TCI)], bromo-cresol green indicator (MERCK) Hydrochloric acid (EMPLURA- 35%), sodium chloride (EMPLURA), phenolphthalein Sodium bicarbonate (EMPLURA), Methyl 151 orange indicator (MERCK) and, sulphuric acid (EMPLURA-98%) The materials and equipment utilized in the present study, together with their specifications, are already reported in our previous work[19].

Experimental absorption study

CO_2 absorption experiments were performed using 15 wt% aqueous MEA, tetrabutylammonium acetate (TBA OAC), and non-aqueous tributylmethy lammonium dicyanamide (TBMA DCA). Initial properties (pH, density, viscosity, surface tension, and carbon loading) were measured before testing. Each 70 g solvent sample was placed in a temperature-controlled glass reactor supplied with CO_2 at 0.5 psi for one hour. Post-absorption, carbon loading, physical properties, and pH were analyzed using standard instruments, while diffusivity was estimated via the Wilke–Chang equation. Results were discussed comparatively for MEA and ionic liquids [20-21].

Results and Discussion

Effect of reaction mixture temperature on carbon loading time

Fig. 1 illustrates that during CO_2 absorption, the temperature of a 15 wt% aqueous MEA reaction

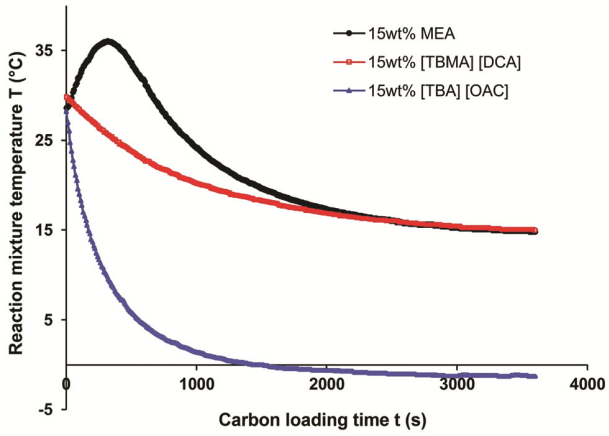

Fig. 1 — Effect of reaction temperature on carbon loading time

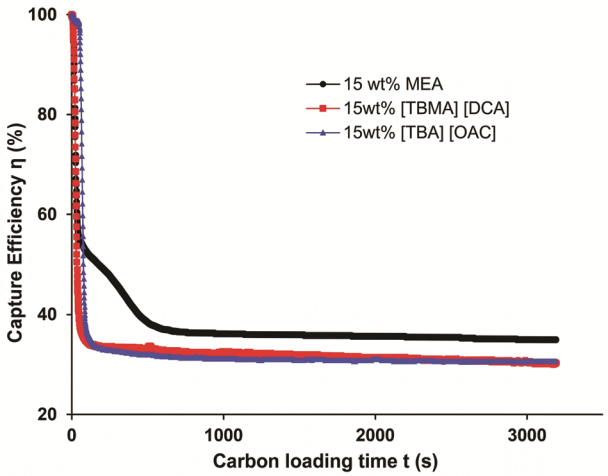

Fig. 2 — Effect of carbon capture efficiency on carbon loading time

mixture rises with increasing carbon loading due to the exothermic nature of the reaction between MEA and CO_2, which initiates the formation of carbamate via the zwitterion mechanism, subsequently leading to the hydration of CO_2 to produce carbonate and bicarbonate. The reaction mixture attained a maximum temperature of 35.9 °C from a starting temperature of 28.6 °C. Subsequently, the temperature of the reaction mixture reduces due to thermal dissipation to the environment and less CO_2 absorption. Conversely, the reaction between CO_2 and ILs transpires via cation-anion interactions. The interactions will be electrostatic interactions (hydrogen bonding) or van der Waals force of attraction, When CO_2 dissolute in 15 wt% aqueous Tetrabutylammonium acetate (TBA OAC) and 15wt% non-aqueous tributylmethylammonium dicyanamide (TBMA DCA) is an endothermic reaction. The TBMA DCA reaction mixture attained the minimum temperature of 15 °C from 29.8 °C initial reaction temperature. Subsequently TBA OAC mixture attained the minimum temperature of -1.3 °C from 28.2 °C during carbon loading. Ramdin *et al.* [22] reported that acetate based ionic liquids absorb CO_2 through chemical reactions that remain mildly exothermic although significantly lower heat of absorption (-40 kJ/mol) compared to MEA (-85 kJ/mol). The reduction in bulk temperature likely results from several factors. These include the ongoing expansion of CO_2 gas during sparging, evaporative cooling of the solvent, heat transfer between the solution and its surroundings, and the relatively low heat released during CO_2 absorption by the ionic liquids[23-24]. These elements can influence the measured bulk temperature when there is a continuous flow of gas. This is especially true for low-viscosity dilute ionic liquid solutions. In contrast, the higher heat release from MEA can mask these cooling effects.

Effect of carbon capture efficiency on carbon loading time

15 vol% CO_2 was loaded in 15wt% aqeuous MEA and TBA OAC and Non-aqeuous TBMA DCA for 1 h. The instantanuous CO_2 capture efficiency is calcualted using the Eq. 1. Fig. 2 illustrates that during the initial 10 seconds of carbon loading in fresh MEA, TBMA DCA, and TBA OAC was avaialable and the driving force for mass transfer was maximal, resulting in instantaneous capture efficiencies of 97%, 98%, and 99%, respectively. After 20 s the capture efficiency of MEA, TBMA DCA, TBA OAC attained nearly 83%, 82% and 98%. The high initial capture efficiency 90% TBA OAC is due to the strong CO_2 affinity of the acetate anion with the siginificanlty weak carion-anion interaction with bulky tetrabutylammonium cation thereby it facilitates the greater availability of the acetate anion for interaction with CO_2[25]. As CO_2 absorption improved, active sites became occupied gradually which reduce the mass transfer driving force and maximise the outlet CO_2 concentration. Hence the instantaneuous capture efficiencies decreased over a period of time. After 1 h of CO_2 loading, MEA achiveved 34% whereas as TBMA DCA and TBA OAC achieved 30% capture efficiency indicating the absorbents were come close to saturation under experimental conditions.

$$Capture\ efficiency\ (\%) = \frac{Inlet\ CO_2\ conc. - Outlet\ CO_2\ conc.}{Inlet\ CO_2\ conc.} \times 100 \quad \ldots (1)$$

Effect of temperature on the kinematic viscosity of both carbon-loaded and virgin samples

Viscosity significantly influences the mass transfer kinetics between CO_2 and solvent. Elevated viscosity will reduce CO_2 solubility. Fig. 3 illustrates that when the temperature rises, the viscosity of virgin and carbon-loaded MEA, TBMA DCA, and TBA OAC decreases. The increase in temperature results in elevated kinetic energy among the aqueous MEA and TBA OAC and the non-aqueous TBMA DCA. At higher temperatures, the molecules overcome the intermolecular interactions between MEA and water, TBMA DCA and methanol, as well as TBA OAC and water, leading to reduced viscosity. Subsequently intermolecular forces between the carbon loaded MEA, TBMA DCA & TBA OAC were surpassed during high temperature exposed lower viscosity. The viscosity of MEA, TBMA DCA decreases with increasing the temperature. The viscosity of TBA OAC decreases from 25 to 40 ℃ as similar to other solvents. At 55 ℃ virgin and carbon loaded TBA OAC viscosity got increased due to vapour loss.

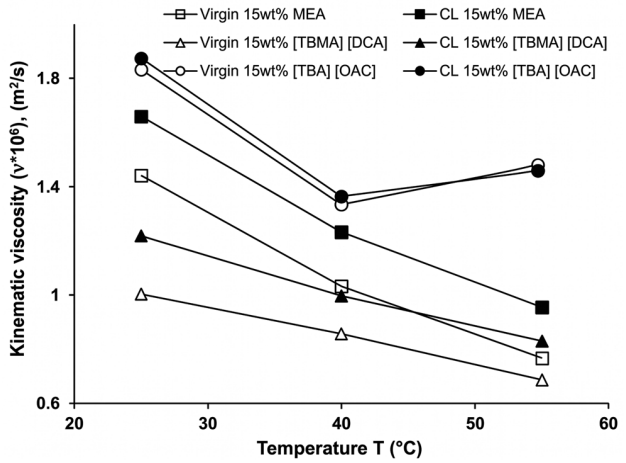

Fig. 3 — The effect of temperature on the kinematic viscosity of both carbon-loaded and virgin samples

Effect of carbon loading time on diffusivity coefficient and reaction temperature

During carbon loading the diffuisivity coefficient of 15 wt% aqeuous MEA & TBA OAC and non-aqeuous TBMA DCA were measured at a various range of reaction mixture temperature, 0.5 psi inlet pressure for 1 h. The diffusivity coefficient of the carbon loaded solutions were predicted by using Wilke-Chang equation[26]. The estimated diffusivity is mainly depends on solvent viscosity and temperature. From the Fig. 4 it revealed that the diffusivity coefficient of carbon loaded MEA increased with increasing the temperature & vice versa, reason being when heat is applied to the carbon loaded solution the

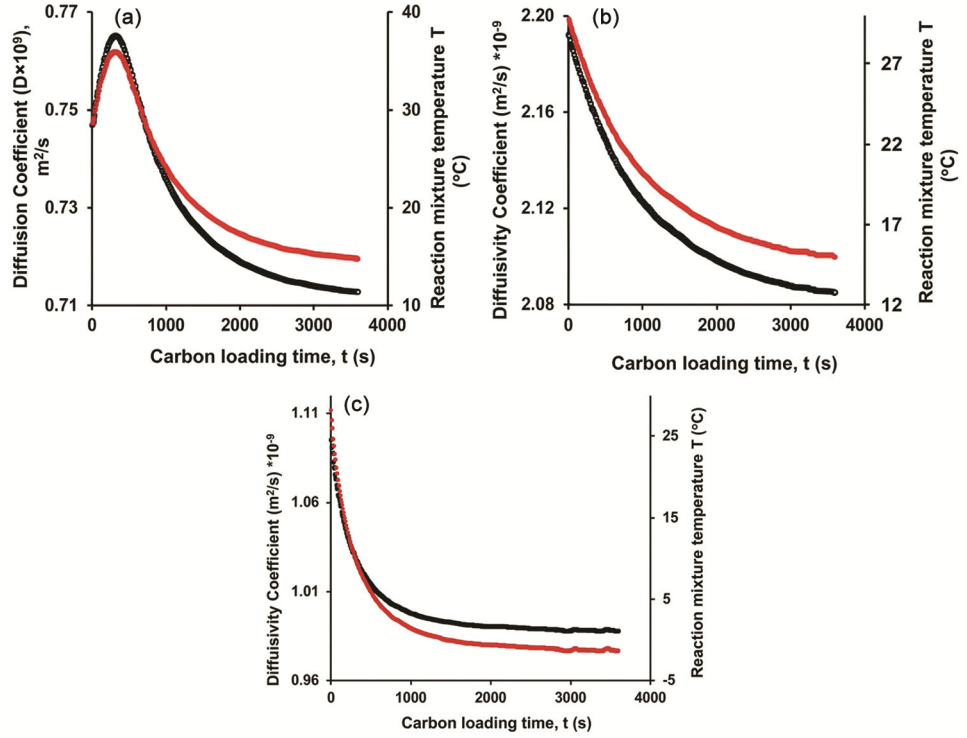

Fig. 4 — Effect of carbon loading time on diffusivity coefficient and reaction temperature [(a) MEA, (b) TBMA DCA and (c) TBA OAC]

Table1 — Physicochemical analysis

Conditions	pH	Temp. (°C)	Density (g/cc)	Carbon Loading (mol CO_2/kg)	Viscosity (mPas)	Surface Tension (dyn/cm)
15wt% MEA	12.07	28.6	1.024±0.00	0.882±0.01	1.37±0.00	67.671±0.04
Carbon loaded 15wt% MEA @0.5psi for 1 h	8.36	28.6	1.073±0.00	5.215±0.15	1.78±0.01	73.63±0.04
15wt% TBMA DCA	8.24	29.2	0.817±0.00	0.127±0.00	0.82±0.00	23.01±0.01
Carbon loaded 15wt% TBMA DCA @0.5 psi for 1 h	7.67	29.2	0.828±0.00	2.879±0.00	1.01±0.00	24.34±0.02
15wt% TBA OAC	9.99	28.2	1.005±0.00	0.183±0.03	1.84±0.02	46.78±0.00
Carbon loaded 15wt% TBA OAC @0.5 psi for 1 h	6.89	28.2	1.014±0.00	3.441±0.06	1.9±0.01	53.43±0.03

atoms in the solutions undergo vibrational motion and collide with another neighbour atom. However, the diffusivity coefficient of MEA in CO_2 was low compared to diffusivity coefficient of carbon loaded ILs. Since the viscosity of TBMA DCA OAC was significanlty lower that MEA as shown in Table 1.

Physio-chemical analysis

pH, surface tension, carbon loading, density, and viscosity are interrelated physicochemical qualities. Table 1 presents the physicochemical characterization of virgin and carbon-loaded 15 wt% aqueous MEA, TBMA DCA, and TBA OAC.

pH

CO_2 is an acidic gas. The pH of virgin 15 wt% aqueous MEA was 12.07. When CO_2 react with aqueous MEA, acid forming reaction took place and pH of carbon loaded MEA was 8.36. Previous studies reported that interaction of CO_2 with ILs depend on anion nature via van der Waals force of attraction or electrostatic force of attraction[27-28]. The pH of virgin 15 wt% non-aqueous TBMA DCA was 8.24 and 15wt% aqueous TBA OAC was 9.99 both ILs was slightly alkaline in nature. When CO_2 react with TBMA DCA through van der Waals force of attraction, the pH of carbon loaded TBMA DCA was reduced to 7.67 and for TBA OAC pH was 6.89. The pH change is not much in virgin and carbon loaded ILs compared to virgin and carbon loaded MEA. From the pH results it roughly indicates more CO_2 was absorbed in MEA compared to ILs.

Density

Aqueous MEA and TBA OAC density were more than 1 g/cc. The density of virgin MEA was 1.024 while after carbon loading the density of carbon loaded MEA was 1.073 g/cc due to higher mass of the substance. In case of non-aqueous TBMA DCA, the density was 0.817 g/cc because 15wt% TBMA DCA was prepared by diluting with methanol. During carbon loading the density of carbon loaded TBMA DCA was slightly increase to 0.828 g/cc. In TBA OAC, the density of the virgin sample was 1.005 g/cc, while the density of the carbon-loaded sample was 1.014 g/cc.

Viscosity

In general viscosity and surface tension should be low to enhance the mass transfer kinetics An increase in carbon loading elevates dynamic viscosity and surface tension, hence reducing the mass transfer rate and reaction kinetics between CO_2 and solvents. The viscosity of virgin MEA was 1.37 mPa s and for carbon loaded MEA viscosity was 1.78 mPa s. The viscosity of carbon loaded MEA was very high compared to ILs sample. Viscosity of IL is specifically examined by strength of van der Waals force of attraction between CO_2 and IL. DCA anions have low viscous due to the formation of hydrogen bond while carbon loading. The viscosity virgin TBMA DCA (μ-0.82 mPa.s) and carbon loaded TBMA DCA (μ-1.01 mPa.s) showed that there is a nominal change of viscosity before and after carbon loading. In case of virgin TBA OAC viscosity was 1.84 mPa.s and carbon loaded TBA OAC viscosity was 1.9 mPa.s is exhibited by the acetate anion due to hydrogen bonding with water molecules.

Surface tension

Table 1 shows that the surface tension of MEA, TBMA DCA and TBA OAC increased after carbon loading compared to virgin solvents. This rise is due to changes in the intermolecular interactions between CO_2 and solvents. When CO_2 reacts with aqueous MEA, the formation of carbamate, bicarbonate and

protonated amines species enhances the cohesive interaction within the liquid thereby increasing the energy required to make a new gas-liquid interface. Similarly, CO_2 absorption in ionic liquids changes the intermolecular interactions which can also rise the surface tension[29-30]. Since, surface tension depends on gas-liquids interphase phenomena, these changes influence the gas-liquid mass transfer and consequently affect the absorption kinetics

Carbon loading

When CO_2 react with solvents the major mechanisms take place are bond making or bond breaking or chemical reaction. The carbon loading was measured using Eqs (2) and (3)[31].

Solvent concentration was determined using Eq. 2.

$$C_1V_1 = C_2V_2 \quad \text{... (2)}$$

The carbon loading of the sample was determined using Eq. 3.

$$\text{Carbon loading} = \frac{V_{CO2}}{\frac{A \times B}{\frac{C_1 \times V_1}{B}}} \quad \text{... (3)}$$

Chemical absorption takes place between CO_2 and MEA, carbamate formation occurs due to acid hydrolysis, as an intermediate compounds then it hydrolyzed into carbonate and bicarbonate. In ILs cation-anion interactions takes place through hydrogen bonding between CO_2 and ILs. The solubility of CO_2 in ionic liquids mostly relies on the cation and anion components. Anions primarily influence CO_2 solubility, while cations have a minor effect. After 1 h continuous sparging of CO_2 gas, the carbon loading absorbed for MEA, TBA OAC and TBMA DCA were 5.215, 3.441 and 2.879 mol CO_2/kg of solvent. Although operating pressure was only 0.5 psi, the continuous gas supply maintained the driving force for mass transfer throughout the absorption process. Similar low pressure CO_2 absorption was already reported in our previous work on aqueous MEA-tetrabutyammonium hydroxide blends[21]. Carbon loading escalates with the augmentation of density, surface tension, and viscosity in the carbon-loaded samples of MEA, TBMA DCA, and TBA OAC. The results indicated that carbon loading in MEA was significantly higher than in TBMA DCA and TBA OAC.

Conclusion

The CO_2 absorption studies were performed for 15 wt% MEA, TBMA DCA and TBA OAC. The detailed physicochemical analysis of solvents was also measured experimentally before and after CO_2 absorption process. From the obtained results, it was concluded MEA exhibited highest carbon loading (5.215 mol CO_2/kg of solvent) followed by TBA OAC (3.441 mol CO_2/kg of solvent) and TBMA DCA (2.879 mol CO_2/kg of solvent). All solvents achieved initial CO_2 capture efficiencies greater than 97% which gradually decreased with increasing carbon loading time. However, the surface tensions of TBA OAC have less significant change during carbon loading compared to MEA. These findings revealed that, TBA OAC has better capture efficiency than TBMA DCA. Furthermore, by elevating the inlet operating pressure may enhances the solubility of CO_2 in ionic liquids due to higher diffusivity.

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