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Solubility of CO_2 in dialkylimidazolium dialkylphosphate ionic liquids

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The solubility of carbon dioxide in room temperature ionic liquids (RTILs), dialkylimidazolium dialkylphosphates, was measured at 313–333 K and at pressures close to atmospheric pressure, from which Henry's law coefficients, standard Gibbs free energy, enthalpy, and entropy changes of solvation were derived. The CO_2 solubility in the dialkylimidazolium dialkylphosphate was found to increase with increasing chain length of the alkyl groups on the cation and/or the anion as was similarly found in other RTILs. Among various dialkylimidazolium dialkylphosphates tested, 1-ethyl-3-methylimidazolium diethylphosphate ([EMIM][Et_2PO_4]) and 1-butyl-3-methylimidazolium dibutylphosphate ([BMIM][Bu_2PO_4]) exhibited the comparable or better capability of dissolving CO_2 in comparison with that of [BMIM][BF_4], but their absorption capacities were still lower than that of 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([BMIM][Tf_2N]).

1. Introduction

Carbon dioxide (CO_2) capture from flue gases has become an important issue in recent years for a sustainable society. Currently, most of industrial processes employ primary or secondary alkanolamine-based aqueous solutions to chemically trap CO_2 through the formation of ammonium carbamate [1,2].

Even though these amine-based systems are highly effective for CO_2 capture, there are many drawbacks [3,4] such as the uptake of water into the recovered CO_2 gas stream, the loss of amines during reclamation due to their volatile nature, and the degradation of amines by small amount of sulfur-containing compounds contained in the flue gas or natural gas. Therefore, the development of a system to sequestrate CO_2 without loss of the capturing agent is highly demanded. In this context, the use of room temperature ionic liquids (RTILs) has been proposed as CO_2 capturing reagents because RTILs possess negligible vapor pressures and capability to dissolve CO_2 through a physical interaction [5,6]. The separation of CO_2 using a physical interaction is particularly attractive because the stripping of CO_2 from a RTIL can be operated at a much milder condition than that from an aqueous amine-based solution. A number of reports have been published on the application of RTILs to the reversible CO_2 absorption, but most of the reports have focused on the use of costly dialkylimidazolium-based RTIL with a fluorinated anion such as tetrafluoroborate ([BF_4]), hexafluorophosphate ([PF_6]), and (bistrifluoromethylsulfonyl)imide ([Tf_2N]), possibly due to their relatively strong affinities to CO_2 [7–13]. Weakly Lewis acid–base interaction of CO_2 with BF_4^- in 1-butyl-3-methylimidazolium tetrafluoroborate ([BMIM][BF_4]) has been demonstrated by an in situ ATR-IR study [14].

As a means of improving CO_2 solubility in non-fluorinated RTILs, a modification on the cation has been suggested either by introducing a long chain alkyl group or an ether linkage to create greater "free volume" or by incorporating a CO_2-philic carbonyl functional group [15]. Surprisingly, however, dialkylimidazolium dialkylphosphates, halogen-free RTILs, have never been the subject of detailed investigation for CO_2 capture in spite of their easy synthesis and high stability toward hydrolysis [16].

Herein, we report on the ability of a series of dialkylimidazolium dialkylphosphates to capture CO_2 as well as the related thermodynamics.

2. Experimental

General procedure: All of the chemicals (purities 98–99%) used for the synthesis of RTILs were purchased from Aldrich Chemicals Co. and were used as received. CO_2 with purity of 99.9% was purchased from Sin Yang Gas Co., Korea. ^1H NMR spectra were recorded on a 400 MHz Bruker NMR spectrometer. Densities of neat RTILs were carefully estimated at 313.15, 323.15, and 333.15 K at an ambient pressure. In a typical density measurement, the RTIL was loaded in a 5.573 ± 0.004 mL pycnometer (previously calibrated using distilled water at 298.15 K) immersed in an ethylene glycol bath. The

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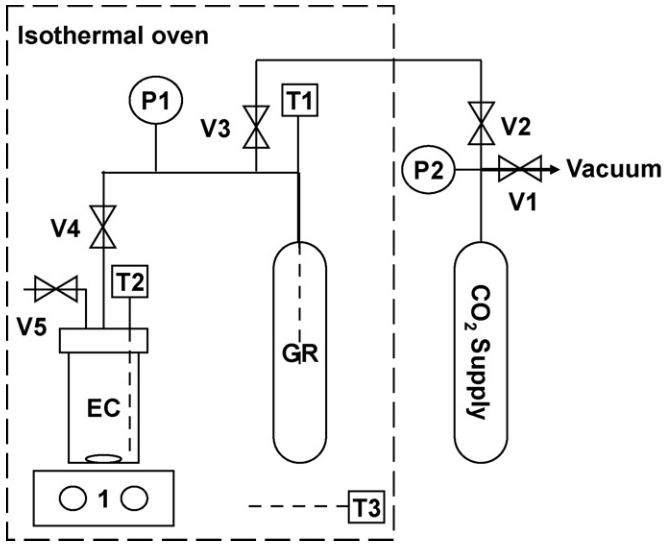
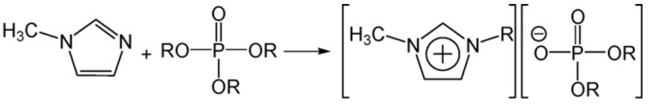


Fig. 1. Schematic diagram of the apparatus for CO_2 solubility test: EC, equilibrium cell; GR, gas reservoir; V1–5, valves; T1–3, thermocouples; P1 and P2, pressure transducers; 1, magnetic stirrer.

weight of RTIL was measured using a Mettler AJ180 balance with an accuracy of 10^{-4} g. Water contents in RTILs were determined using a Karl–Fischer Moisture Titrator (MKC-520, KEM Co., Ltd.).

2.1. Synthesis of RTILs

The dialkylimidazolium dialkylphosphates were synthesized from the alkylation of 1-methylimidazole with trialkylphosphate at 90–120 °C in the absence of a solvent as shown in Eq. (1), according to the literature procedures [16,17].



(1)

where $R = CH_3$, C_2H_5, and n-C_4H_9.

The prepared RTILs, 1,3-dimethylimidazolium dimethylphosphate ([DMIM][Me_2PO_4]), 1-ethyl-3-methylimidazolium diethylphosphate ([EMIM][Et_2PO_4]), and 1-buyl-3-methylimidazolium dibutylphosphate ([BMIM][Bu_2PO_4]) were dried under vacuum (<1 mbar) at 348 K for at least 8 h to completely eliminate the residual volatiles and moisture before use. Purities of the RTILs were in the range of 98–99% as deduced from the ^1H NMR results. Water contents of these RTILs were below 200 ppm as determined by Karl–Fischer titration.

2.2. CO_2 solubility measurement

CO_2 solubility measurements were carried out based on an isochoric saturation method [7,18]. An in-house, stainless steel-made apparatus, consisting of an equilibrium cell (EC) and a gas reservoir (GR) was constructed as illustrated in Fig. 1. The volume of EC was measured by filling it with distilled water at room temperature. The volume of GR and the rest of the system were determined from an expansion of nitrogen gas employing the *pVT* relation. The solubility test unit was placed in an isothermal oven and the temperature inside was carefully controlled with an accuracy of 0.1 °C. CO_2 pressure was monitored using a pressure transducer (OMEGA Engineering PX309-030AI, 0–207 kPa, accuracy 0.25% full scale). In a typical CO_2 absorption experiment, a measured amount of a RTIL was loaded into the EC and degassed under vacuum at 343 K at least for 2 h prior to each test. At a specified oven temperature, the valve (V4), connecting two vessels (EC and GR) was closed and then CO_2 was fed from the CO_2 supply cylinder to the GR through V2 and V3 valves. At this time, the amount of CO_2 at equilibrium was calculated from the *pVT* relation. CO_2 was then brought into contact with the absorbent in the EC by opening the V4 valve. The absorbent was stirred vigorously to facilitate the CO_2 absorption. After the absorption reached equilibrium, the amount of dissolved gas was calculated from a difference between the initial gas concentration in the GR and the concentration in the gaseous phase. CO_2 concentrations were calculated using the gas virial equation of state truncated after the second term employing the second virial coefficients taken from the compilation by Dymond and Smith [19].

3. Results and discussion

3.1. Physical properties of ionic liquids

Table 1 shows the densities and the molar volumes of dialkylimidazolium dialkylphosphates. The literature values of [BMIM][Tf_2N], [BMIM][BF_4], and [EMIM][$EtSO_4$] were also included for comparison. The density of dialkylimidazolium dialkylphosphate was not significantly affected by the variation of temperature, but strongly influenced by the chain length of alkyl groups on the cation and the anion. Dialkylimidazolium dialkylphosphate with longer alkyl substituents exhibited lower density, suggesting that longer alkyl chain creates larger free volume within the RTIL [12,15,20,21]. Such a density behavior in a homologous series of asymmetric imidazolium-based ionic liquids is also well documented in many literature [9,12,16,22–24]. The molar volume of dialkylimidazolium dialkylphosphates increases as going from [DMIM][Me_2PO_4] to [BMIM][Bu_2PO_4]. The molar volumes of dialkylimidazolium dialkylphosphates were compared with the literature values of [EMIM][$EtSO_4$], [BMIM][BF_4], and [BMIM][Tf_2N] and were in the order of [BMIM][Bu_2PO_4] > [BMIM][Tf_2N] > [EMIM][Et_2PO_4] > [EMIM][$EtSO_4$] > [BMIM][BF_4] > [DMIM][Me_2PO_4].

3.2. CO_2 solubility measurements

Solubility measurements were performed at pressures ranging from 20 to 200 kPa and at 313.15, 323.15, and 333.15 K. The amount of dissolved CO_2 was calculated based on a pressure-decay observation. The CO_2 solubility based on mole fraction, on absorbent volume, and the CO_2 equilibrium pressure above the liquid absorbent are listed in Table S-1 (Supplementary data). The CO_2 solubilities increase linearly with the pressure rise for all the dialkylimidazolium dialkylphosphates tested (Fig. S-1, Supporting Information), showing positive deviations from Raoult's law [13,28]. Such a solubility behavior may suggest that CO_2 is physically absorbed, as also can be found in other imidazolium-based non-functionalized RTILs [5,7–13,15,18,20,25–29].

The solubility of a solute in a liquid absorbent can be expressed in terms of Henry's law coefficient as follows:

$$H_{2,1}(p,T) \equiv \lim_{x_2 \to 0} \frac{f_2(p,T,x_2)}{x_2} \tag{2}$$

where $f_2(p, T, x_2)$ is the fugacity of component 2 (CO_2), p is pressure of the gas, and x_2 is its mole fraction in the liquid phase.

At an equilibrium state, fugacities of each component in the liquid phase and in the vapor phase are equal.

$$f_2^{liq}(p,T,x_2) = f_2^{vap}(p,T,y_2) = \phi_2(p,T,y_2)y_2p \tag{3}$$

where $\phi_2(p, T, y_2)$ is the fugacity coefficient of component 2 and y_2 is the mole fraction of component 2 in the gaseous phase. Since

Table 1
Densities (ρ) and molar volumes (V_m) of dialkylimidazolium dialkylphosphates, [BMIM][BF$_4$], [EMIM][EtSO$_4$], and [BMIM][Tf$_2$N] at different temperatures.

$T = 313.15$ K		$T = 323.15$ K		$T = 333.15$ K	
ρ (g cm^{-3})[a]	V_m (mL mol^{-1})	ρ (g cm^{-3})	V_m (mL mol^{-1})	ρ (g cm^{-3})	V_m (mL mol^{-1})
[DMIM][Me$_2$PO$_4$]					
1.2510	177.6	1.2444	178.5	1.2380	179.5
[EMIM][Et$_2$PO$_4$]					
1.1386	233.4	1.1321	233.4	1.1255	234.8
[BMIM][Bu$_2$PO$_4$]					
1.0400	335.0	1.0337	337.1	1.0271	339.2
[BMIM][BF$_4$][b]					
1.1899	188.9	1.1828	190.1	1.1757	191.4
[EMIM][EtSO$_4$][c]					
1.2302	192.1	1.2242	193.0	1.2182	194.0
[BMIM][Tf$_2$N][d]					
1.4236	294.6	1.4141	296.6	1.4046	298.6

[a] $\Delta\rho = \pm 0.0001$.
[b] Densities and molar volumes were calculated based on the literature [7].
[c] Literature [25].
[d] Literature [26].

RTILs have negligible vapor pressures, Eq. (3) can be simplified into Eq. (4).

$$\phi_2(p, T, y_2) y_2 p = \phi_2(p, T) p \tag{4}$$

The fugacity coefficient of component 2 can be expressed as (5) by using the virial equation with two terms.

$$\phi_2(p, T) = \exp\left[\frac{pB_{22}(T)}{RT}\right] \tag{5}$$

At a very low solute concentration in an absorbent, it holds that

$$H_{2,1}(p, T) = \lim_{x_2 \to 0} \frac{f_2(p, T, x_2)}{x_2} = \lim_{x_2 \to 0} \frac{\phi_2(p, T) p}{x_2} \tag{6}$$

In the limit as $x_2 \to 0$, Henry's law coefficient can be calculated using the following Eq. (7):

$$H_{2.1}(p, T) \approx \frac{\phi_2(p, T) p}{x_2} \tag{7}$$

In our experiments, Henry's law coefficient was estimated from the slope of an isotherm linear fit of fugacity versus mole fraction. The Henry's law coefficients at various temperatures and the standard error of the isotherm slopes are given in Table 2. As listed in Table 2, Henry's law coefficients in all RTILs increase with increasing temperature.

It was previously reported that the solubility of CO$_2$ in a dialkylimidazolium salt with a fluorinated anion such as [BF$_4$] [10] or [Tf$_2$N] [26] increases with increasing chain length of an alkyl substituent on the cation. Such a phenomenon was explained in terms of free volume in the ionic liquid, which is believed to contribute to dissolve gaseous molecules [15,20]. A similar phenomenon was also observed with dialkylimidazolium dialkylphosphates, but the effect of alkyl substituent was more pronounced, possibly due to the presence of larger number of alkyl groups with longer chain on the cation as well as on the anion.

Table 2
Variation of Henry's law coefficient with temperature.

T(K)	$H_{2.1}$ (MPa)	Error[a]
[DMIM][Me$_2$PO$_4$] + CO$_2$		
313.15	10.64	0.12
323.15	12.72	0.03
333.15	15.22	0.15
[EMIM][Et$_2$PO$_4$] + CO$_2$		
313.15	6.99	0.03
323.15	8.12	0.04
333.15	9.66	0.02
[BMIM][Bu$_2$PO$_4$] + CO$_2$		
313.15	4.98	0.02
323.15	5.76	0.02
333.15	6.85	0.03

[a] Standard errors of the isotherm slopes.

Fig. 2 shows the solubility of CO$_2$ in various RTILs at 0.1 MPa in the temperature range from 313 to 334 K. The CO$_2$ solubilities in dialkylimidazolium dialkylphosphates increased with the increase of the alkyl chain length on the cation and the anion of the RTIL, *i.e.*, with the increase of the molar volume of RTIL. These results strongly indicate that there is a close correlation between the molar volume of RTIL (Table 1) and the CO$_2$ solubility, as was similarly observed in the other RTILs–CO$_2$ systems [10,12,25–27].

Among dialkylimidazolium dialkylphosphate tested, [BMIM][Bu$_2$PO$_4$] exhibited the highest CO$_2$ absorption capacity. For comparison, solubility data of CO$_2$ in [BMIM][BF$_4$], [EMIM][MeSO$_4$] and [BMIM][Tf$_2$N] taken from the literature [7,25,26] were also included in Fig. 2. In general, the CO$_2$ solubility is in accordance with the order of the molar volume of RTILs except for [BMIM][Tf$_2$N] and [EMIM][Et$_2$PO$_4$]. The solubility of CO$_2$ was in the order of

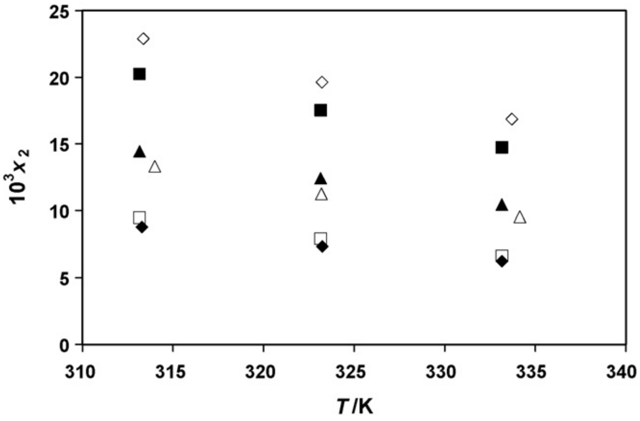


Fig. 2. Variation of CO$_2$ solubility with temperature at 0.1 MPa of CO$_2$: □, [DMIM][Me$_2$PO$_4$]; ▲, [EMIM][Et$_2$PO$_4$]; ■, [BMIM][Bu$_2$PO$_4$]; △, [BMIM][BF$_4$] [7]; ♦, [EMIM][EtSO$_4$] [25]; ◇, [BMIM][Tf$_2$N] [26].

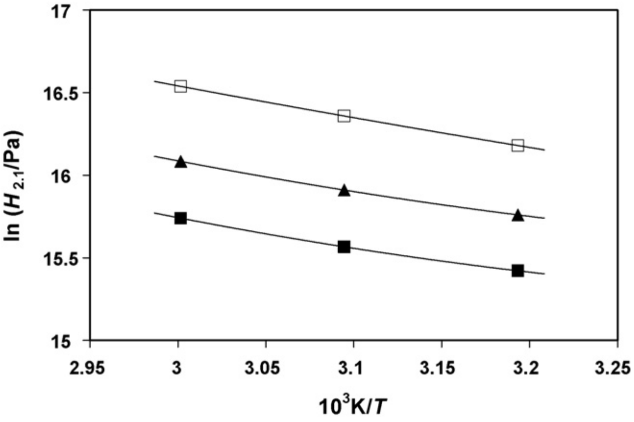


Fig. 3. Variation of Henry's law coefficient at different temperature of absorption: □, [DMIM][Me_2PO_4]; ▲, [EMIM][Et_2PO_4]; ■, [BMIM][Bu_2PO_4]. Lines represent the smoothed data correlation using the parameters in Table S-2 (Supplementary data).

[BMIM][Tf_2N] > [BMIM][Bu_2PO_4] > [EMIM][Et_2PO_4] > [BMIM][BF_4] > [DMIM][Me_2PO_4] > [EMIM][$EtSO_4$]. It is worth to mention here that [BMIM][Tf_2N], one of the most intensively studied ionic liquids for the absorption of CO_2, shows higher CO_2 absorption capacity than that of [BMIM][Bu_2PO_4] even though its molar volume is smaller than that of [BMIM][Bu_2PO_4]. This result supports the previous findings [12,15,20,27] that in addition to the free volume of the RTIL, the stronger interaction of CO_2 with a fluorinated anion enhances the CO_2 solubility. The presence of highly electronegative fluorine atoms in [Tf_2N] anion would provide favorable effect on the CO_2 solubility through an additional interaction with CO_2. The significantly reduced CO_2 solubility in [EMIM][$EtSO_4$] compared with that in [EMIM][Et_2PO_4] could be ascribed in part to the inherent lower basicity of alkyl sulfate anion than that of the dialkyl phosphate anion. Even though the solubility of CO_2 in dialkylimidazolium dialkylphosphates are relatively lower than in an imidazolium-based RTIL bearing a [Tf_2N] anion, these RTILs still possess many advantages in terms of their simple, cost effective, and halogen-free synthesis.

Fig. 3 displays the effect of temperature on Henry's law coefficient. As expected, all the dialkylimidazolium dialkylphosphates showed the negative slopes of the correlation, indicating that the CO_2 solubility decreases with the increase of absorption temperature.

The dependence of Henry's law coefficients with temperature was correlated as a function of temperature using an empirical Eq. (8).

$$\ln\left[\frac{H_{2.1}(T)}{10^5\ \mathrm{Pa}}\right] = \sum_{i=0}^{n} B_i \left(T/\mathrm{K}\right)^{-i} \tag{8}$$

The optimized coefficients, B_i, obtained using a linear regression of multiple-variables calculation, are listed in Table S-2 in the Supplementary data along with the average absolute deviation (AAD) for each compound.

The Henry's law coefficients for [DMIM][Me_2PO_4], [EMIM][Et_2PO_4], and [BMIM][Bu_2PO_4] at 313.15 K obtained from Eq. (8) are 8.94, 6.28, and 4.41, respectively. All the Henry's law coefficients of these RTILs show positive deviations from the Raoult's law in that they are greater than the ideal value of $0.5f_2^{liq}$ or 2.44 MPa at 313.15 K [29]. The positive deviation from the ideal value may indicate that the CO_2 solubility in the RTIL is not exclusively dependent on the interaction between CO_2 and the RTIL, although, specific interactions, such as a Lewis acid–base interaction [14] should not be ruled out.

Table 3
Gibbs free energy, enthalpy, and entropy of CO_2 solvation in dialkylimidazolium dialkylphosphates at some discrete temperatures.

T (K)	$\Delta_{sol}G$ (kJ mol^{-1})	$\Delta_{sol}H$ (kJ mol^{-1})	$\Delta_{sol}S$ (J mol^{-1} K^{-1})
[DMIM][Me_2PO_4] + CO_2			
298.15	10.87	−12.88	−79.73
313.15	12.13	−14.54	−85.16
323.15	12.99	−15.56	−88.37
333.15	13.89	−16.52	−91.29
[EMIM][Et_2PO_4] + CO_2			
298.15	10.11	−6.34	−55.18
313.15	11.06	−11.14	−70.88
323.15	11.82	−14.09	−80.16
333.15	12.66	−16.86	−88.62
[BMIM][Bu_2PO_4] + CO_2			
298.15	9.26	−5.06	−48.02
313.15	10.12	−10.54	−65.97
323.15	10.83	−13.92	−76.58
333.15	11.64	−17.09	−86.24

The thermodynamic properties of the solvation for each RTIL were obtained from Eqs. (9)–(11) by incorporating Eq. (8) and coefficients in Table S-2. The Gibbs free energy, enthalpy, and entropy change of CO_2 solvation are given by the relations as follows:

$$\Delta_{sol}G = RT\ln\left(\frac{H_{2.1}(T,p)}{p^{\circ}}\right) \tag{9}$$

$$\Delta_{sol}H = R\left(\frac{\partial\ \ln(H_{2.1}(T,p)/p^{\circ})}{\partial(1/T)}\right)_p \tag{10}$$

$$\Delta_{sol}S = \left(\frac{\Delta_{sol}H - \Delta_{sol}G}{T}\right) \tag{11}$$

Thermodynamic properties of CO_2 solvation in a series of dialkylimidazolium dialkylphosphate at various temperatures and at standard pressure (p° = 101,325 Pa) are listed in Table 3.

It is well known that the enthalpy of solvation is largely associated with the molecular interaction between CO_2 and a RTIL, and the entropy of solvation is related to the solvent organization surrounding the solute [5,30]. Similarly to other RTIL–CO_2 systems [5–7,10,11,13,24,25,29], the solvation of CO_2 in a dialkylimidazolium dialkylphosphate is exothermic as indicated by the negative values of enthalpy of solvation. The enthalpy of solvation becomes less negative as the length of alkyl chain increases. This inconsistent trend with the amount of CO_2 dissolved may suggest that the enthalpy of solvation may not be a major factor in determining the solubility of CO_2 in a dialkylimidazolium dialkylphosphate. Meanwhile, the more negative values of entropy indicate a higher ordering degree when CO_2 dissolves in the RTIL. As shown in Table 3, the negative value of the entropy decreased as going from [DMIM][Me_2PO_4] to [BMIM][Bu_2PO_4], implying that the CO_2 dissolution is more favorable in a RTIL bearing alkyl groups with longer chain length, which is in good agreement with the experimental results. Higher CO_2 solubility in a dialkylimidazolium dialkylphosphate with longer alkyl substituents suggests that CO_2 solubility is more strongly dependent on the entropy of solvation rather than on the interaction between CO_2 and the RTIL.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tca.2009.04.022.

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