Purification of Flue Gas by Ionic Liquids: Carbon Monoxide Capture in [bmim][Tf_2N]

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Knowledge of the solubility of carbon monoxide in ionic liquids is important for investigating the potential use of ionic liquids in reactions and gas separations. There is very limited information available on the capacity of ionic liquids to capture CO. The solubility of CO in 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide was investigated experimentally up to temperatures and pressures of 460 K and 10 MPa, respectively. The solubility was shown to be only slightly temperature-dependent, and the P–T diagram for a constant composition mixture is concave-downward with a peak in pressure. By comparing the single-gas solubility results of CO with various other gases in the same ionic liquid, it seems that ionic liquids can potentially be used as gas-separating media. However, although the single-gas solubilities vary considerably among some gases, further studies on mixed (multicomponent) gases should be carried out as the presence of other components can influence the solubility of each gas component. © 2013 American Institute of Chemical Engineers *AIChE J*, 59: 3886–3891, 2013

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Introduction

As fossil fuels are not renewable, it is necessary to wisely use the remaining resources, in a manner to obtain the highest energy/product efficiency possible and to have the least negative impact on the environment. Unfortunately, there is no magical solution to this issue, where one bright idea will resolve the energy and environmental problems of the planet. This is a battle that must be fought globally, where various sectors of civil and industrial consumers each take responsibility and action with the step(s) possible to them, no matter how small the step. Even minor percentages of reductions in energy consumption and pollution emission, when carried out globally, will have a significant impact on the planet.

Among the numerous routes that can be followed with this green mindset, is the attempt to provide alternative resources for the petrochemical industries, which is currently heavily ethylene-based. Carbon monoxide can be the starting material for many chemicals, including methanol, formaldehyde, acetic acid, isocyanates, aldehydes, formic acid, pesticides, and herbicides.[1] With the Fischer–Tropsch process becoming increasingly important, there is further demand for CO as raw material. Very often, CO is among the gases released from industrial processes.[2] Therefore, if CO is separated and recovered from industrial flue gases, it not only helps the environment as less polluting gases are introduced into the atmosphere, but also provides raw material to replace fossil fuels, which again, helps the environment. For example, David et al.[3] propose flue gases from carbon black manufacturing as a new source for carbon monoxide separation and recovery, because the typical concentration of the major constituents is about 60% N_2, 20% CO, 15% H_2, and 5% CO_2 (vol % on a dry basis).

The separation of CO from H_2 is also of utmost significance in the petrochemical industries based on synthesis gas (essentially a mixture of H_2 and CO). This is because the products derived from synthesis gas require either a particular feed H_2/CO ratio or pure CO.[2] The reforming of natural gas to produce syngas often produces a H_2/CO ratio higher

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than that required for downstream processing, so it is necessary to purify the stream to the desired concentration.[2]

In addition to the major objectives mentioned above, CO separation from gases is also important in some small scale requirements. Purification of life support system gases in a self-contained environment is one example.[2] Work spaces with combustion devices and underground mining require the removal of CO from air.[2]

When purifying flue gas, in addition to extracting CO, there are other polluting gases which should also be prevented from being vented into the atmosphere. Among these is the most important greenhouse gas, CO_2. For obvious reasons, much research has already been carried out on the separation of CO_2 from various gas streams, including flue gas (postcombustion), synthesis gas (precombustion), coal/biomass gasification gas, reformate, or even from atmospheric air.[4] These methods include absorption by physical or chemical solvents, the use of membranes, adsorption onto solids, and cryogenic separations. Ryckebosch et al.[5] conveniently review the advantages and disadvantages of the various techniques used for CO_2 removal. Currently, the most utilized method for capturing CO_2 is absorption by an amine-based solvent. However, operational costs are very high. As Merkel et al.[6] point out, to capture 90% by volume of the CO_2 in the flue gas produced in a powerplant, it would consume 30% of the electricity produced by the powerplant. Therefore, there is much room for improvement and so, extensive research is being carried out to make CO_2 capture more practical and less expensive. Jones[7] reviewed recent advances in solvents, adsorbents, and membranes for postcombustion CO_2 capture, but specifically highlighted the potential of room-temperature ionic liquids, supported amine materials, mixed matrix membranes, facilitated transport membranes, and metal-organic frameworks.

So, among the various advantages of using ionic liquids (IL) for CO separations, for example in the form of supported ionic liquid membranes, is that they can also separate other undesired gases such as CO_2, H_2S, SO_2, N_2O, and hydrocarbon gases.[8] Because of their surprisingly high solubility for CO_2, ionic liquids have recently received much attention as CO_2 capturing media. Excellent reviews were published in the current decade.[9–12] However, there are quite a limited number of studies which are concerned with gas separations involving CO, using ionic liquids,[3,13–17] and even these mostly report solubility data at low pressures. Knowledge of CO solubility in ionic liquids is also important for reactions that replace conventional solvents with the "greener" ionic liquids, for example in hydroformylation or in the copolymerization of CO with styrene. Because of its importance and the serious lack of CO solubility data in current literature, in this work we present high-pressure solubility data of carbon monoxide in the ionic liquid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (abbreviated to [bmim][Tf_2N]). To the best of our knowledge, there are only two literature studies on this system; however, they are both very limited in the amount of information that they present: Ohlin et al.[14] only present data at a single (low) temperature and Gan et al.[15] do not present any experimental data points, but only the Henry's constants. Therefore, the data presented in this work will be useful for the more realistic process conditions, covering a wide a range of temperatures and up to high pressures.

Table 1. Experimentally Measured Solubility Data (Bubble-Point Curves) for CO (1) in [bmim][Tf_2N] (2)

x_1	T (K)	P (MPa)	T (K)	P (Mpa)	T (K)	P (Mpa)
0.051	322.99	4.805	337.96	4.895	352.98	4.945
	367.99	4.995	382.98	4.975	398.03	4.945
	413.03	4.925	428.03	4.875	443.06	4.825
	458.09	4.785				
0.063	323.04	6.126	338.02	6.156	353.03	6.181
	368.02	6.206	383.02	6.196	398.06	6.176
	413.09	6.136	428.11	6.116	443.14	6.096
	458.15	6.056				
0.075	322.95	7.514	322.97	7.524	337.88	7.554
	337.90	7.574	352.85	7.604	352.88	7.624
	366.00	7.604	367.85	7.624	382.83	7.604
	397.75	7.579	397.82	7.604	412.75	7.529
	427.77	7.484	442.78	7.414	457.78	7.354
0.099	309.19	10.343	322.99	10.323	337.98	10.313
	352.69	10.303	367.65	10.293	382.66	10.263
	397.64	10.193	412.62	10.123	427.95	10.033

Experimental

Experiments were carried out in the Cailletet equipment that consists of a glass equilibrium cell, in the form of a closed-ended tube, in which the appearance/disappearance of phases is observed visually. The equilibrium cell is injected with known amounts of CO and ionic liquid ad closed off. At any desired temperature, obtained by a circulating thermostat fluid around the tube, the pressure is increased until the last bubble of vapor disappears. The equipment can tolerate temperatures up to 450 K and pressures up to 15 MPa, respectively. The uncertainties of measurements are within 0.001 for mole fraction, 0.02 K for temperature, and 0.03% of the reading for pressure. 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide was supplied by Fluka with a purity of >98% and carbon monoxide had a purity of 99.997%. The experimental setup and procedure is explained in detail elsewhere.[18,19]

Results

Table 1 presents the experimental results of this work in the form of bubble point pressures for various mixtures of CO + [bmim][Tf_2N]. The data cover a temperature range from 323 to 458 K and pressures up to 10 MPa. Figure 1 shows the pressure–temperature relationship for different isopleths. The slopes of the curves in this system are interesting, as they show a maximum in pressure. This shows that up to a certain temperature the solubility decreases with temperature, after which the temperature effect is switched to increased solubility upon temperature increase. However, the slopes on either side of the peak are small, which indicates that the solubility of carbon monoxide is not greatly affected by temperature. This is in contrast to all of the published data on binary systems of ionic liquids with CO_2, which all show positive slopes throughout entire temperature ranges. However, such a concave-downward shape was observed previously for mixtures of CO and a higher member of this same ionic liquid family, 1-hexyl-3-methylimidazolium bis(-trifluoromethylsulfonyl)imide.[17] The thermodynamic basis of this behavior is explained extensively elsewhere.[20]

Table 2 presents the interpolated P–x data at constant temperatures and Figure 2 shows the P–x isotherms. Although, the behavior seems to be linear, the best-fit line does not

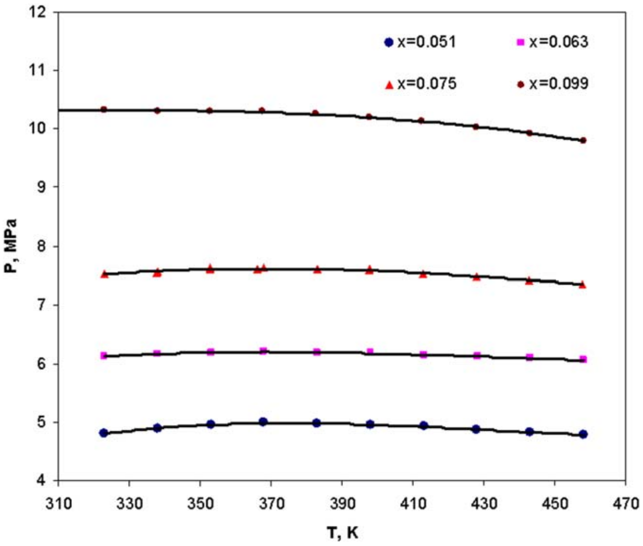

Figure 1. Experimentally measured bubble point pressures of CO in [bmim][Tf_2N] as a function of temperature for four different molar concentrations of CO: ● 0.051; ■ 0.063; ▲ 0.0.075; ◆ 0.099.

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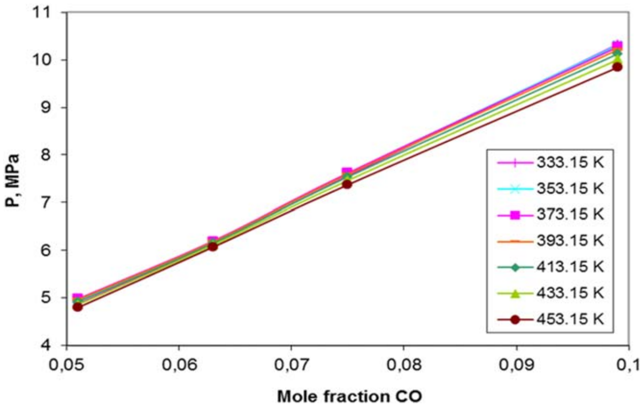

Figure 2. Bubble point pressure vs. molar concentration of CO in [bmim][Tf_2N] at seven different temperatures.

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pass through the origin, so it is incorrect to assume that Henry's law is applicable in the CO molar concentration range measured in this study. In this figure, it can also be seen that temperature effects are slightly higher at higher pressures, but nearly coincide at low pressures for the various isotherms, confirming that CO solubility is only slightly temperature-dependent. Only one other literature study[14] presents solubility data points for this binary mixture, which only presents data at 295 K. The data are compared with our results in Figure 3. It is seen that the two sets of data are rather consistent, even though the temperatures are different. As mentioned above, solubility of CO in [bmim][Tf_2N] does not vary much with temperature, therefore, such a comparison at two different temperatures is not a cause of concern. Gan et al.[15] have published the Henry's constant for [bmim][Tf_2N] + CO at four different temperatures ranging from 303 to 333 K. However, they did not mention the applicability range of Henry's law on this mixture. The Henry's constant-driven phase behavior of Gan et al.[15] is also plotted on Figure 3, which shows somewhat higher solubilities compared to both the data of Ohlin et al.[14] and this work.

Figure 4 compares the pressures required to dissolve carbon monoxide in [bmim][Tf_2N] to those in conventional organic solvents (acetone, benzene, cyclohexane, ethanol, and heptane).[21] Among the solvents investigated, only heptane has higher dissolving power for CO than the ionic liquid. However, compared to the rest of the solvents, CO can dissolve more readily in the investigated ionic liquid, for example, to dissolve CO to a mole fraction of 0.07, while it is adequate to operate at about 6.7 MPa for [bmim][Tf_2N], it is necessary to increase the pressure to about 7.2, 9.2, 11.3, and 14.6 MPa, respectively, for cyclohexane, acetone, benzene, and ethanol. This is of particular importance in reactions involving CO, where ionic liquids can be beneficially used to replace these organic solvents.

The single-gas solubility data of various gases of concern in gas separations involving CO are presented in Figure 5 for comparison. This figure includes gases, not only of concern for flue gas separations such as N_2O, O_2, and CO_2,[21–23] but also gases concerned in precombustion decarbonization

Table 2. Interpolated Isothermal Solubility for CO (1) in [bmim][Tf_2N] (2)

x_1 T (K)	0.051 P (MPa)	0.063 P (MPa)	0.075 P (MPa)	0.099 P (MPa)
323.15	4.804	6.121	7.516	10.331
333.15	4.871	6.153	7.558	10.326
343.15	4.921	6.175	7.588	10.319
353.15	4.955	6.189	7.607	10.307
363.15	4.974	6.196	7.617	10.292
373.15	4.981	6.196	7.616	10.271
383.15	4.977	6.190	7.608	10.245
393.15	4.964	6.180	7.591	10.212
403.15	4.944	6.165	7.568	10.172
413.15	4.919	6.148	7.538	10.125
423.15	4.889	6.129	7.503	10.068
433.15	4.858	6.109	7.463	10.003
443.15	4.827	6.088	7.419	9.927
453.15	4.798	6.068	7.372	9.841

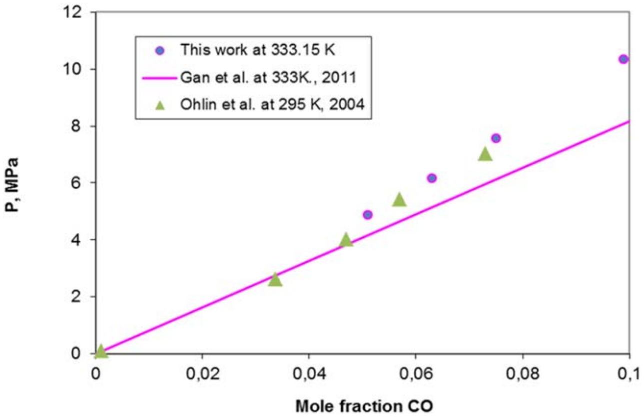

Figure 3. Comparison of the experimental results of this work with literature data.[14,15]

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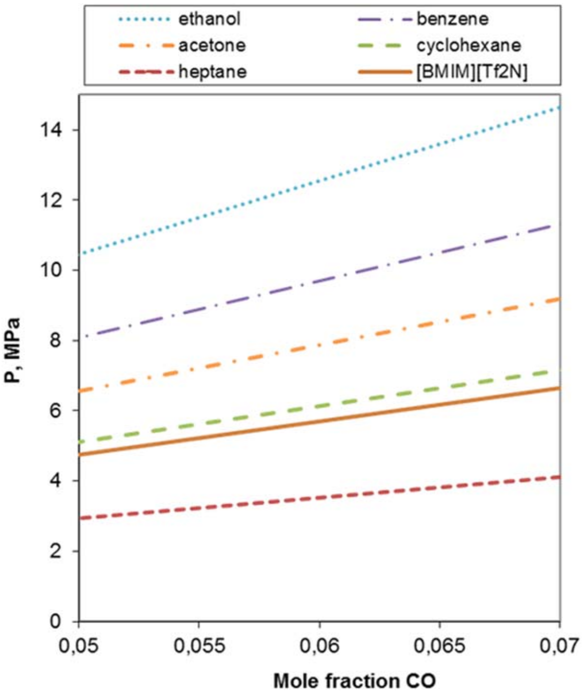

Figure 4. Comparison of CO solubility in conventional solvents and in [bmim][Tf_2N].

The curves for the organic solvents are plotted using the Henry's Constant values at 298 K given by Anthony et al.[21] The curve of [bmim][Tf_2N] is from the data of this work at 333 K. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and synthesis gas purification, for example, H_2, H_2S, CH_4, C_2H_4, etc.[20–28] Although Figure 5 only presents single-gas solubilities, it is still very valuable as a qualitative indication of how well different gases dissolve in the ionic liquid. Of course, it is a well-known fact that the presence of other compounds does indeed affect the solubility of a particular gas in an ionic liquid. For example, Hert et al.[27] observed that in the presence of CO_2, the solubility of either O_2 or CH_4 increased in [hmim][Tf_2N] compared to the case when no CO_2 is present. CO_2 itself showed decreased solubility in this ionic liquid when O_2 or CH_4 were present. Therefore, for exact design purposes, it is necessary to also measure solubilities in the multicomponent mixture of concern. The comparison of the relative location of the various single-gas solubility curves in this figure, however, confirms the idea that, at least from a thermodynamic solubility perspective, separations between particular gases is possible by using ionic liquids as solvents, for example, in supported ionic liquid membranes. While gases such as hydrogen and oxygen have insignificant solubilities in [bmim][Tf_2N], other gases such as CO_2, H_2S, N_2O, and hydrocarbon gases, especially the heavier ones, dissolve in high concentrations. Therefore, CO separations are potentially possible for a wide range of gas separations.

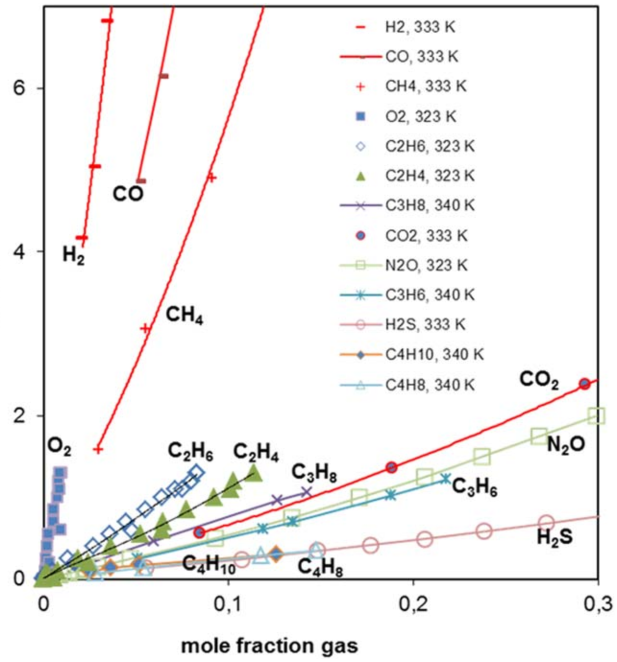

Figure 5. Comparison of the solubility of various gases in [bmim][Tf_2N].

The data are taken from Refs. 20–28 and this work. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

In the case of flue gas purification in particular, it is of interest to investigate the absorption capacity of [bmim][Tf_2N] for CO_2, CO, SO_2, N_2O and compare them with N_2 and O_2 solubility, as these are the polluting gases with the highest concentrations in flue gas. It was already shown that CO_2 is very soluble in this ionic liquid.[22] For example, the single-gas solubility of CO_2 at 323 K and 2 MPa is about 0.3 in mole fraction. The corresponding values for CO, N_2O, and CH_4 at the same temperature and pressure are 0.02, 0.30, and 0.04, respectively.[23,28] Unfortunately, isotherms are not available for the solubility of SO_2 in [bmim][Tf_2N]. The only reported data is by Huang et al.[26] who reported that at a temperature of 293 K and a pressure of 0.1 MPa, the molar fraction of SO_2 is 0.57. This is almost twice the solubility of CO_2, which was already very high. It is expected that the ionic liquid can efficiently capture the SO_2 impurities of flue gas. There is also no information in literature about the solubility of nitrogen in [bmim][Tf_2N]. However, all of the studies on ionic liquids so far, have indicated very small dissolving power for nitrogen and oxygen gas. Hence, it is very likely that the solubilities of N_2 will be insignificant also in [bmim][Tf_2N]. Just as a very rough estimate, if we assume N_2 solubility to be more or less the same as O_2 solubility, as is the case with other investigated ionic liquids, the single-gas concentration ratio (molar) of carbon dioxide to nitrogen at 323 K and 2 MPa is roughly $CO_2/N_2 \approx 17$, and the corresponding values for other gases are: $SO_2/N_2 >> 17$, $N_2O/N_2 \approx 17$, $CO/N_2 \approx 1$, $CH_4/N_2 \approx 2$.[21–23,26,28] The solubilities of the various gases will increase at higher pressures, quite sharply for the highly soluble gases such as SO_2, N_2O, and CO_2 and much less for the low-dissolving gases such as N_2 and O_2. Therefore, for flue gas purifications, it is suggested to operate at high pressures. Based on the above estimates of single-gas solubilities, which were shown to have differences of up to one order of magnitude with one another, it should be possible from a thermodynamic point of view, to absorb some of the impurities of flue gases in this ionic liquid to separate them from N_2 and O_2, before being vented into the atmosphere.

However, if one focuses on CO capture alone, the above results may seem discouraging. Although CO was shown to dissolve better in [bmim][Tf_2N] than in most conventional

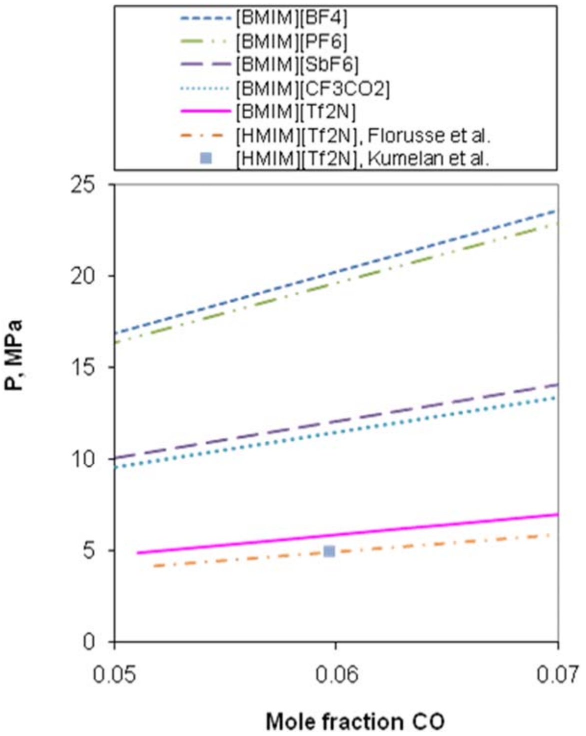

Figure 6. Comparison of CO solubility in different ionic liquids.

The curves for [bmim][BF_4], [bmim][PF_6], [bmim][SbF_6], and [bmim][CF_3CO_2] are plotted using the Henry's Constant values at 295 K given by Ohlin et al.[14] The curves of [hmim][Tf_2N] and [bmim][Tf_2N] are from the data of Florusse et al.,[17] Kumelan et al.[16], and this work at 333K, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

organic solvents, it still does not have the high solubility that one would hope for in order to achieve easy and pure separations. However, if one considers the common liquid absorbents that have been investigated for CO separations, it is seen that they are not capable to simultaneously remove other impurities from flue gas. Ionic liquids, on the other hand, are very good absorbents for CO_2, SO_2, and NO_x. Organic solvents are simply not good solvents for volatile gases such as CO; therefore, to increase the solubility in a liquid, researchers have mainly focused on absorbents which form complexes with CO. Examples include ammoniacal copper liquor, COSORB, Fe(TIM), palladium complex, and selenium and secondary amine.[29] However, such complex-forming absorbents do not make complexes with other flue gas pollutants. For example, in the COSORB process CO_2 and CH_4 are chemically inert to the solvent,[30] CO_2 does not react with the Fe(TIM) solvent,[1] the palladium complex is good for separating CO_2 and CH_4 from CO,[31] and the selenium and secondary amine system can be used to separate CH_4 from CO.[32] Therefore, using such absorbents for CO removal would call for additional unit operations to remove the other impurities of flue gases. However, ILs can simultaneously dissolve many gases, and in addition, as gas solubility in ILs is purely physical and no chemical interactions are involved, the ILs can be cheaply and very easily regenerated by pressure drop or temperature increase. One further advantage of ILs, compared to other liquid absorbents, is that upon regeneration, the separated gases are not polluted with the solvent, because an ionic liquid has insignificant vapor pressure. Because of all this, ionic liquids deserve further investigations due to their unique properties.

Figure 6 compares the solubility of CO in several ionic liquids previously investigated in literature.[14,16,17] Among the ionic liquids shown having various anions, the family with the [Tf_2N] anion shows the highest CO solution capacity. The trend of CO solubility is usually similar to CO_2 solubility in ionic liquids, i.e., the ionic liquids that have better solubility for CO_2 are also better CO dissolvers. Since one of the main advantages of ionic liquids is that they can be "designed" for desired properties, in the last few years much research activity has been devoted to finding functionalized ionic liquids, especially engineered for higher CO_2 capture. These same ionic liquids will most probably have higher CO absorption capacities as well. However, in contrast to the large amount of information already available on ionic liquids regarding CO_2 capture, unfortunately the solubility of CO in this new class of functionalized ILs remains to be investigated. It is expected that in the future, ILs will be available with higher CO-capturing capacities, making them more suitable for gas separation purposes.

Conclusions

CO_2 capture by ionic liquids has been the topic of much research. The high absorption of CO in the ionic liquids would be a further advantage in flue gas purifications. However, the results of this experimental investigation have shown that CO is not greatly soluble in [bmim][Tf_2N], although it still dissolves better than in most of the other ILs investigated in literature, as well as in conventional organic solvents. If the aim is to increase CO solubility, future research must focus on functionalized ionic liquids, specifically designed for CO capture. Fortunately, there seems to be a general trend that ionic liquids with higher CO solubility also have a greater solution capacity for CO_2.

Acknowledgments

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