Evaluation of 1-Butyl-3-methylimidazolium Bis(trifluoromethylsulfonyl)imide–Alkanolamine Sulfolane-Based System as Solvent for Absorption of Carbon Dioxide

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ABSTRACT: A mixture of 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (BMIM)[NTf2]) and monoethanolamine (MEA) were added into a dipolar aprotic solvent, sulfolane to form a nonaqueous physical-chemical absorbent for CO2 absorption. The CO2 solubility achieved highest value of 2.861 mol/kg in mixture of 30 wt % MEA + 70 wt % sulfolane (0B30M) at 303.15 K and 2004 kPa. Comprehensive data on effect of temperature, pressure, and composition on the multicomponent were measured and correlated with maximum average absolute relative deviation of 4.89%. Formation of biphasic layer was observed and it was analyzed using nuclear magnetic resonance (NMR).

1.0. INTRODUCTION

As reported by the International Energy Agency, a steady rise of CO2 emission was recorded in the past 10 years. CO2 is the main gas that contributes to the global warming; more than 60% in greenhouse gases composition1 and the major source of CO2 is from the industries, for instance, fossil–fuel power plants and natural gas treatments as well as hydrocarbon processing. A significant increase of global CO2 emission was observed in 2013, an increase of 2.2% over the previous year with 68% of total global emission originates from the energy sector. Therefore, it is necessary to capture CO2 to cope with the worsen scenario of global warming.

There are various methods in CO2 capture technology such as physical or chemical absorption into liquid,2−5 adsorption on a solid,6,7 and membrane separation.8,9 One of the most widely used CO2 capture technologies processes is the chemical absorption onto different types of alkanolamines solvent. The common aqueous alkanolamine solutions applied in the industrial CO2 removal processes are monoethanolamine (MEA), diethanolamine (DEA), methylethanolamine (MMEA), methyldiethanolamine (MDEA), aminomethylpropanol (AMP), diethylene glycolamine (DGA), diisopropanolamine (DIPA), piperazine (PZ), triethanoleamine (TEA) and were used either as a single or blended aqueous solution.10,11 Although this process removes CO2 efficiently, it does pose severe economic and environmental problems, for example high energy cost for solvent regeneration, solvent loss due to evaporation, thermal, and oxidative degradation due to high desorption temperature and equipment corrosion during the process.12,13 The drawbacks of this CO2 removal processes with aqueous amine solutions are commonly attributed to water.14

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Therefore, by replacing water with other high-boiling point organic solvents, a considerable amount of thermal energy can be saved during the solvent heating cycle and the corrosion will also be prevented.\textsuperscript{15}

Currently, studies on nonaqueous solution are being conducted for the CO\textsubscript{2} absorption process. It was reported that improvement in terms of CO\textsubscript{2} solubility was observed by substituting water with ethanol in the MEA solution.\textsuperscript{16} Other polar organic solvents were also studied for mixture with MEA, MDEA, DIPA, and TEA.\textsuperscript{17,18} But, because of the high vapor pressure of these nonaqueous mixtures using methanol, ethanol, or other polar organic solvent, they are not suitable for CO\textsubscript{2} capture solvent. Some researcher replace water with lower vapor pressure solvent like polyols. A mixture of MEA + triethylene glycol (TEG), AMP + diethylene glycol (DEG), and AMP + TEG were develop to absorb CO\textsubscript{2}.\textsuperscript{15,19,20} But such solvents tend to have high viscosity, which will affect the liquid−film for mass transfer.\textsuperscript{21}

The commercial organic solvent, sulfolane is often used in the industries, such as the petrochemical, polymer, and photographic chemical, textile, hydrocarbon extraction and plasticizer industries,\textsuperscript{22,23} because of its high thermal and hydrolytic stability with high density and boiling point.\textsuperscript{24} Sulfolane is one of the major components in the common industrial CO\textsubscript{2} absorption process called Sulfinol process. Up to 45% of the total formulation, which acts as physical absorbent, facilitates the CO\textsubscript{2} absorption process and possess higher absorption capacity among other physical solvent.\textsuperscript{25} Therefore, sulfolane is a suitable candidate in replacing water in the nonaqueous application.

Over the years, ionic liquids have emerged as new attractive solvents have been used in various applications and were reported to be a potential solvent for CO\textsubscript{2} removal. This is due to their almost neglectable volatility, thermal stability, and low heat capacity.\textsuperscript{26−30} Ionic liquids seem to be suitable alternative solvents for energy efficient gas separations.\textsuperscript{31−33} Furthermore, ionic liquid can be tailored to specific requirement by varying the combinations of constituent ionic counter-parts. Investigation on the possible application of ionic liquids either in pure form\textsuperscript{34−38} or in combination with alkanolamines\textsuperscript{39−43} for physical/chemical absorption processes were reported in literature. Ionic liquid–alkanolamine combination was reported to offer reductions in energy by forming a carbamate precipitate and also prevent corrosion of the system.\textsuperscript{41,42}

Phase changing solvent system has emerged and drawing much focus in the past few years. This promising CO\textsubscript{2} capturing system form a precipitation\textsuperscript{44} or biphasic carbonated solution after contact with CO\textsubscript{2}. The DMX process developed by French Institute of Petroleum (IFP) Energies Nouvelles undergoes biphasic layer formation into CO\textsubscript{2}-rich and CO\textsubscript{2}-lean layers upon completion of CO\textsubscript{2} absorption,\textsuperscript{45} which shows a decrease in energy consumption throughout the process.

Therefore, the objective of this works is to evaluate a nonaqueous mixture system for CO\textsubscript{2} removal using blends of ionic liquid and alkanolamine as an active component in sulfolane nonaqueous media in CO\textsubscript{2} removal process. A few systems were formulated with different composition of the ionic liquid and alkanolamine blends and the absorption capacities were compared. To our knowledge, this system has

<table>
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Figure 1. Schematic diagram of the experimental setup for measuring the CO\textsubscript{2} solubility: A, gas (CO\textsubscript{2}) cylinder; B, gas (CO\textsubscript{2}) reservoir; C, motor; D, high pressure reactor vessel (equilibrium cell); E, heater; F, reactor controller; G, PC graphical user interface; V\textsubscript{1}, control valve; V\textsubscript{2}, needle valve; V\textsubscript{3}, pressure relief valve.
not being reported in literature. It was also observed that our studied system displays a phase changing behavior.

2. EXPERIMENTAL SECTION

2.1. Materials. Ionic liquid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([BMIM][NTf2]) (>98%) was purchased from Iolitech. Tetrahydrothiophene 1,1-dioxide, sulfolane (>98%) was purchased from Merck. Monoethanolamine (MEA), diethanolamine (DEA), 2-amino-2-methyl-1-propanol (AMP), and 2-(2-aminoethylyamine)ethanol (AEEA) were purchased from Merck. Purified CO2 (99.995%) was purchased from Linde Malaysia Sdn Bhd (Linde). All chemicals were used as received. The chemical used in this work were supplied with high purity and summarized in Table 1 with source purity and CAS number of the chemicals

2.2. Sample Preparation. Binary and ternary mixtures were prepared by weighing the materials using a calibrated single pan digital balance (Sartorius BSA224S-CW, Germany) with precision of 0.1 mg. All mixtures were kept in tightly sealed bottles to minimize moisture absorption from the atmosphere.

2.3. Measurements. Figure 1 illustrates a schematic diagram of experimental setup for measuring CO2 solubility, which has a welded stirrer assembly, a thermocouple, an inlet gas tube, and air vent tube. Both integrated reactors with a thermostated heater and thermocouple (accuracy is ±0.1 K). Operation was initially started by purging the air out from the gas reservoir by sufficiently flushing CO2 throughout the system. The gas reservoir was then loaded with purified CO2 from gas tank before it was heated and pressurized to the required condition. A known volume of fresh sample was initially filled in the thermostated high-pressure reactor cell. The temperatures of both gas container and reactor were kept constant, throughout the process. The initial and equilibrium pressures were recorded to compute using a pressure transducer (0−500 PSI, ± the differences in CO2 amount throughout the absorption of which the solubility was expressed as per mole of total mol or mass of solvent. The solubility was calculated using eq 1 and 2 as follows:

\[
\chi = \frac{(P_i - P_f) \times V_c}{zRT} - \frac{(P_f - P_i) \times (V_c(V_{sol} - V_{sol}))}{zRT} \quad n_{total}
\]

\[
\alpha = \frac{(P_i - P_f) \times V_c}{zRT} - \frac{(P_f - P_i) \times (V_c(V_{sol} - V_{sol}))}{zRT} \quad m_{total}
\]

where, \(\chi\) is the CO2 loading in mol of CO2/total mol of absorbent, \(\alpha\) is the CO2 loading in mol of CO2/total mass of absorbent, \(P_t\) is the total pressure, \(P_v\) is the vapor pressure, \(V_c\) is volume of gas container, \(V_{sol}\) is volume cell, \(V_{sol}\) is volume solution, \(i\) is initial condition, \(f\) is final condition. All measurement was in triplicate and reported as average with standard deviation.

2.4. Apparatus Reliability Validation. Several runs for CO2 absorption using pure [BMIM][NTf2] at reference temperature was performed for validation purposes. These data were compared with the data reported in literature46 as shown in Figure 2. The average absolute relative deviation (AARD) was 0.33% which demonstrates the reliability and practicability of the equipment.

3. RESULTS AND DISCUSSION

3.1. Solubility of CO2 in [BMIM][NTf2] + Sulfolane Binary Mixtures. Figure 3 shows the solubility of CO2 in...
binary mixture of $[\text{BMIM}][\text{NTf}_2] +$ sulfolane at pressure range of 500 to 2000 kPa at $T = 323.15$ K. The results indicated that the solubility of CO$_2$ increased with the increase of $[\text{BMIM}][\text{NTf}_2]$ composition. This is due to the higher affinity of CO$_2$ to $[\text{BMIM}][\text{NTf}_2]$ in comparison to sulfolane. CO$_2$ solubility in ionic liquids is dependent on the interaction between ionic liquids and molecules and the CO$_2$ molecules occupied the free space between anion and cation. Furthermore, the solubility of CO$_2$ increased as the pressure increase. From Figure 3, it is shown that CO$_2$ solubility increase with higher composition of $[\text{BMIM}][\text{NTf}_2]$ in the mixture, but it is economically hindered due to higher cost of $[\text{BMIM}][\text{NTf}_2]$. It is worth mentioning that 0.1 mole fraction of $[\text{BMIM}][\text{NTf}_2]$ in the binary mixture is equal to 30 wt % of $[\text{BMIM}][\text{NTf}_2]$ in the composition. Therefore, reformulation using an alkanolamine is suggested to further increase the CO$_2$ capacity of the solvent mixture.

### 3.2. Screening for Alkanolamine for Ternary Mixtures.

On the basis of the $[\text{BMIM}][\text{NTf}_2] +$ sulfolane binary mixture study, an alkanolamine was included into the mixture to increase the CO$_2$ solubility via chemical reaction. Several alkanolamines representing different types of molecules structure were formulated into the mixture for screening purposes. Tertiary amine was excluded for the screening because of their unable to react with CO$_2$ in a nonaqueous solution. Solubility studies were conducted to investigate the CO$_2$ absorption and result were show in Figure 4. On the basis of the screening, it shows that composition with MEA has the highest absorption capacity compared with others alkanolamine. Furthermore, despite the higher CO$_2$ capacity of AMP and AEEA as compared to MEA, composition with AMP and AEEA have a lower CO$_2$ loading due to the formation of homogeneous semisolid formation that inhibited stirring process and absorption via physical absorption. Therefore, MEA was chosen as a part of the solvent mixture in this work.

### 3.3. Solubility of CO$_2$ in $[\text{BMIM}][\text{NTf}_2] +$ MEA + Sulfolane Ternary Mixture and Model Correlation.

Table 2 represents composition of the mixture samples used in the study. Throughout the study, it was observed that as the composition of MEA in the mixture increased the CO$_2$ loading increases. This is primary due to a higher absorption of CO$_2$ into the solution by chemical reaction with MEA leading to a higher CO$_2$ loading. Regardless of the observation, it was found that the absorption took a shorter time to complete for solvent with high composition of $[\text{BMIM}][\text{NTf}_2]$, as shown in Figure 5.

Figure 6 shows a general trend in the equilibrium whereby the CO$_2$ loading decreases with temperature and increases with pressure. Each mixtures shows a decreasing trend in the graphs with increment of temperature from 303.15 to 333.15 K which indicates a high loading of CO$_2$ at lower temperature at constant pressure. This is due to the higher vapor pressure at a higher temperature, which reflects on lower loading of CO$_2$. On the other hand, higher pressures give better CO$_2$ solubility due to the diffusion of gas into liquid is directly proportion to the pressure of gas above the surface of the solution. It is observed that composition with high $[\text{BMIM}][\text{NTf}_2]$ has more prominent changes in CO$_2$ loading with increments of pressure. The absorption process is likely to be controlled by both chemical and physical absorptions. At high temperature and low MEA composition, physical absorption plays major role in the absorption process, whereas at low temperature and high MEA composition, the chemical reaction shows prominent effect.

### 3.4. Formation of Biphasic Layer.

It is interesting to note that formation of biphasic layers was observed after the absorption in mixture with MEA in the composition. These biphasic layers were shown in Figure 7. The biphasic phenomenon could be due to the formation of insoluble MEA–carbamate in the mixture. Strong ionic interaction and hydrogen bonding between MEA–carbamate make it difficult to dissolve in hydrophobic $[\text{BMIM}][\text{NTf}_2]$ and dipolar sulfolane. Similar observation was reported for CO$_2$ absorption using emulsion of diethanolamine (DEA) and 1-hexyl-3-methylimidazolium bis(trifluoromethylsulfonylimide) ([HMMIM][NTf$_2$]). It was observed that in mixture with higher $[\text{BMIM}][\text{NTf}_2]$ composition (30B-0M, 25B-5M, and 15B-15M), the insoluble MEA–carbamate appears in the upper layer, whereas $[\text{BMIM}][\text{NTf}_2]$ and sulfolane is in the lower layer. For the mixture with higher MEA composition (5B-25M and 0B-30M), the insoluble MEA–carbamate appears in the lower layer while $[\text{BMIM}][\text{NTf}_2]$ and sulfolane is in the upper layer. This could be due to the different in density of the $[\text{BMIM}][\text{NTf}_2]$ and sulfolane layer to the MEA–carbamate layer. Due to the high density of the pure $[\text{BMIM}][\text{NTf}_2]$, mixtures with high composition of $[\text{BMIM}][\text{NTf}_2]$ have a higher density of the $[\text{BMIM}][\text{NTf}_2]$ and sulfolane layer compare to the MEA–carbamate layer.

The observation of biphasic layers formation was in line with several findings. Arshad et al. reported a formation of biphasic layers when an aqueous blend of 2-((diethy lamino)ethanol (DEEA) and 3-((methylamino)propylamine after absorption of CO$_2$. The CO$_2$-rich layer was at the bottom layer, whereas the CO$_2$-lean layer was the upper layer. Biphasic formation was also reported by Xu et al. in utilizing mixture of 1,4-butane diamine (BDA) and 2-((diethy lamino)ethanol.
(DEED). The French Institute of Petroleum (IFP) Energies Nouvelles developed a class of undisclosed DMX absorbent that formed biphasic layers after absorption of CO$_2$.

Figures 8 and 9 show the $^{13}$C NMR spectra of the lower and upper layer of the CO$_2$-loaded 5B-25M sample (taken in methanol-$d_4$) with tetramethylsilane (TMS) as reference. The 5B-25M sample displays six peaks in range of 22.48 to 63.02 ppm. Two intense peaks at 41.89 and 58.99 ppm originated from the CH$_2$−CH$_2$ carbons of the protonated amine (MEA$^+$) and two less intense peaks at 43.69 and 63.02 ppm arise from the ethylene carbon of carbamate MEA. The chemically captured CO$_2$ are shown by the low intensity resonance at 164.57 ppm. Two peaks at 22.34 and 50.64 ppm belongs to sulfolane that partially dissolves in the lower layer. Similar observation can be seen for the 0B-30M sample. Trace of [BMIM][NTf$_2$] was not found in the lower layer of both samples.
samples, which indicates that the hydrophobic ionic liquid, [BMIM][NTf₂], does not dissolve the polar carbamate MEA. It can be assumed that in MEA in all of the nonaqueous samples react with CO₂ forming a MEA carbamate in the lower layer. It is worth mentioning that the samples were initially a homogeneous solution but turned into heterogeneous solution with two layers after CO₂ absorption. The reaction between the nonaqueous amine mixture and CO₂ were represented as follows:

$$\text{RNH}_2 + \text{CO}_2 \leftrightarrow \text{RNH}_2^+\text{COO}^- \quad (3)$$

$$\text{RNH}_2^+\text{COO}^- + \text{RNH}_2 \leftrightarrow \text{RNHCOO}^- + \text{RNHCONH}_3^+ \quad (4)$$

The MEA carbamate and protonated MEA form an ion pair that possesses a high molecular weight which leads to a higher density and viscosity of the lower layer. Sulfolane and [BMIM][NTf₂] are a chemically stable component and do not react with CO₂. The biphasic layers could lead to a reduction in regeneration cost. This can be done by separating the CO₂-rich layer from the mixture and transported to the stripper, instead of transporting the whole mixture.⁴⁷

Based on correlation suggested by Jou and Mather,⁵⁰ the measured CO₂ loading were fitted linearly as a function of pressure according to eq 5. The coefficients for all the mixture systems are summarized in Table 3.

$$\ln P = A + B \ln \alpha$$

where,

$$A = a + bT(K) + cT(K)$$

$$B = d + eT$$

Figure 10 shows a comparison of the calculated and experimental data at varying temperatures and composition, and it is evident that there is a good agreement between the calculated and experimental data. The AARD values are calculated as

$$\text{AARD} = \frac{1}{N_p} \sum_{i=1}^{N} \left( \frac{\alpha_{\text{exp},i} - \alpha_{\text{cal},i}}{\alpha_{\text{exp},i}} \right) \times 100\%$$

Figure 7. CO₂-rich layer in the mixture samples (dotted box).

Figure 8. $^{13}$C NMR spectrum of CO₂-loaded 5B-25M solution, lower layer.
Where \( N_p \) is the number of experimental points and \( \alpha_{\exp} \) and \( \alpha_{\text{cal}} \) are the experimental and calculated values of CO\(_2\) loading, respectively.

### 3.5. Comparison with Other Studies

Few studies have been conducted using a nonaqueous physical–chemical absorption solvent. Figure 11 shows a comparison of CO\(_2\) solubility between this work and literatures. Because of the limited scope of previous work in literature, the selected data were measured at temperature of 313.15 K and MEA composition of 30 wt % for comparison. As shown in Figure 11, at MEA composition of 30 wt % and temperature of 313.15 K, sample 0B-30M was compared to 30 wt % MEA + diglyme\(^{51}\) and 30 wt % MEA + H\(_2\)O\(^{52}\). The comparison shows that 0B-30M sample has the lowest CO\(_2\) loading even though with similar MEA composition. The difference in solubility maybe...
cause by the variance in the physical absorbent component, which is sulfolane and diglyme. Sample 0B-30M also has lower CO\textsubscript{2} loading compared to 30 wt \% MEA in H\textsubscript{2}O. This could be due to formation of stable carbamate facilitate CO\textsubscript{2} absorption by MEA in aqueous solution, whereas sulfolane is a dipolar aprotic solvent that unable to ionize MEA appropriately. Another possible reason for the lower CO\textsubscript{2} loading of the sulfolane based mixture system is due to the biphasic layer formed during absorption process. Formation of CO\textsubscript{2}-rich layer at the surface of the sample mixture during absorption process could limit the contact of CO\textsubscript{2} with physical absorbent component which leads to lower total CO\textsubscript{2} loading of the mixture.

Despite lower CO\textsubscript{2} loading in comparison to other solvent in literature, such disadvantage could be used as an advantage in term of total processing cost. Kim et al.,\textsuperscript{47} demonstrate the concept of phase transitional absorption/regeneration in their work with phase transitional alkanolamine–alcohol mixture. Arshad et al.,\textsuperscript{48} reported a formation of biphasic layers when an aqueous blend of 2-(diethylamino)ethanol (DEEA) and 3-(methylamino)propylamine after CO\textsubscript{2} absorption. They observed the presents of viscous lower phase saturated with CO\textsubscript{2} in comparison with CO\textsubscript{2}-lean upper phase. Similar observation was also reported by Xu et al.,\textsuperscript{49} in utilizing mixture of 1,4-butanediamine (BDA) and 2-(diethlamino)-ethanol (DEED) This CO\textsubscript{2}-rich layer can be easily separated and transported to the stripper in less quantity to be regenerate as illustrated in Figure 12. Furthermore, such approaches may overcome limitation faced due to corrosion and degradation in current industrial processes.

3.6. Recycling of Solvents and Effect of Water. In an effort to minimize the production cost and negative impact on the environment, recyclability of absorption solvent test was conducted. The absorption solvents were reused for further absorption study, which is presented in Figure 13. At the end of every cycle, the CO\textsubscript{2} saturated solvent was desorpted under reduced pressure and at temperature of 383.15 K for 4 h. Figure 13 shows the CO\textsubscript{2} absorption performance of 0B-30M solvent mixture for 5 cycles of absorptions. The result shows a drop in performance for 0B-30M solvent mixture, 46\% on the fifth cycle of absorption. This is possibly due to the loss of significant amount of viscous CO\textsubscript{2}-rich amine phase during repeated recovery activities, which include decantation and desorption. Having a higher number of steps involved exhibits a greater amount of solvent loss. Furthermore, the effect of water content in the feed CO\textsubscript{2} was simulated by addition of small amount of water into the solvent mixture prior to the absorption process. On the basis of the result presented in Figure 14, water has shown no significant changes in CO\textsubscript{2} loading with the biphasic phenomenon still observed.

4. CONCLUSIONS

In this study, solubility of CO\textsubscript{2} in sulfolane based ternary mixture of [BMIM][NTf\textsubscript{2}] and MEA was conducted at
temperature ranging from 303.15 to 333.15 K and pressure from 500 to 2000 kPa. The experimental data were correlated and calculated data were produced with good consistency to experimental data. Formation of a biphasic layer in the solution was observed and identified using $^{13}$C NMR. The CO$_2$-rich layer can be easily separated and transported to the stripper in less quantity to be regenerate. Furthermore, such approaches may overcome limitations faced due to corrosion and degradation in current industrial processes. Further studies are needed to examine the properties and separation of the CO$_2$-rich solution.

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**Notes**

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