

Thermophysical aspects for sustainable non-aqueous amine solvents containing ionic liquids in co₂ capture process

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Graphical abstract



Abstract

Climate change is a prime threat to human health and the environment. Carbon dioxide (CO₂) emissions play a pivotal role in global warming, the greenhouse gas effect, destroys the ecosystem. This impact of climate change can be prevented with the help of a solvent-based CO2 absorption process. In this study, amine-ionic Liquids (IL) were used as a solvent blend for the CO2 absorption blend process. The non-aqueous solvent Monoethanolamine (MEA) - Ionic Liquid (IL) namely tetrabutylammonium hexafluorophosphate [TBA][PF₆] and their total concentration was kept at 30 wt% throughout the study. The thermophysical properties such as density, dynamic viscosity, surface tension, pH and carbon loading of virgin and carbon-loaded non-aqueous amine-IL have been measured before and after the absorption process experimentally. This study was extensively carried out for varying temperatures (293.15 to 333.2 K) and IL concentration (1-2 wt%) at intervals of 10 K and 0.5 wt% respectively. All the measured thermophysical properties of amine-IL show a significant increase in the IL concentration. Conversely, it declines while increasing temperature. Higher carbon loading was observed for 2wt% IL+28wt% MEA, compared to 30wt% MEA, even though increased viscosity was obtained at this

composition. This non-aqueous amine-IL solvent might favor sustainable development in CO₂ capture process.

Keywords: Climate change, CO₂ absorption, Carbon loading, amines, Ionic Liquids

1. Introduction

The most common greenhouse gas, carbon dioxide (CO₂), is considered the primary reason behind environmental impacts like climate change and global warming. $\ensuremath{\text{CO}_2}$ is released into the atmosphere through a variety of industrial processes, including the creation of hydrogen, coal-fired power stations, oil refineries, and the sweetening of natural gas. The CO₂ is continuously released into the atmosphere without any restrictions which causes global warming, melting of glaciers and polar ice caps, a rise in sea level, and extreme weather patterns. The release of CO₂ from various sources accounts for more than 60% of greenhouse gas; CO2 is the primary gas responsible for the rise in world temperatures (Aghel et al. 2022). Several industrial procedures have been developed for extracting CO2 from gas stream mixtures such as flue gas and off gas, that include technologies such as physical and chemical solvent absorption, molecular sieves, carbamation, membrane separation, and cryogenic distillation. Absorption by physical and chemical solvents is a more advanced method of capturing CO₂ (Amirkhani et al. 2023). Aqueous alkanolamines and mixes are a popular absorption method that requires amine scrubbing. Alkanolamines are widely used in most processes due to their high CO_2 reactivity, low cost, and low hydrocarbon absorption. Alkanolamines, including monoethanolamine (MEA), diethylamine (DEA), N-methyl-4-piperidinol (MPDL), 2amino-2-methyl-1-propanol (AMP), and Nmethyldiethanolamine (MDEA), are widely used chemical absorbents in industry. MEA is the benchmark solvent for CO₂ absorption in fossil-fueled thermal industries (Kontos et al. 2023). Alkanolamines do, however, have a variety of disadvantages, like high volatility, thermal and oxidative degradation, equipment corrosion, high operating costs, higher energy consumption, and harmful environmental pollution (Elmobarak et al. 2023). To overcome these

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drawbacks for CO₂ capture, looking into better alternative technologies was required. Ionic liquids are a recently discovered category of potential solvents. Compared to frequently used aqueous amines, they can provide various advantages.

Ionic liquids, or molten salts, arise when an asymmetric organic cation and an inorganic or organic anion mix. The appealing properties of ILs include their low melting point, non-volatility, strong thermal and chemical stability, and low vapor pressure. Because of these qualities, ILs are "green solvents" or "environment-friendly called solvents." Numerous researchers have reported favorably on using ILs in numerous industrial and academic sectors. Catalytic processing, enzymatic processes, electrochemical usage, acid gas treatment, processing of polymer, and battery electrolyte are just a few of the IL applications. A few ammonium-based ILs, as well as several IL classes such as pyridinium, phosphonium, pyrrolidinium, and imidazolium, have previously been identified (Shojaeian et al. 2021). Room-temperature ionic liquids (RTILs) absorb CO₂ by physical absorption and have a limited capacity and selectivity. Task-specific ionic liquids (TSILs) are designed by changing the cation or anion and lengthening the chain to improve selectivity, solubility for certain gases, or catalytic activity. Anions have a greater influence on the solubility of ILs in CO2 absorption than cations (Yan et al. 2024). Anions that have recently been described in the literature include hexafluorophosphate [PF₆], tetrafluoroborate [BF₄], and bis(trifluoromethylsulfonyl)imide [Tf₂N]. [Tf₂N]-based ILs have considerable CO_2 solubility. The [Tf₂N] anion's extraordinary CO₂ solubility might be attributable to its enormous size (Orhan, 2021). Ionic liquids functionalised with amines, known as amino acid-based ionic liquids (AAILs), were created to increase ILs' ability to absorb CO2. AAILs are more capable of absorbing CO2 than most pure amino acids, ethanolamine derivatives, and amino acid salts. According to Noorani et al. (2021), these ILs boost the capacity of CO₂ absorption by chemisorption.

Perumal et al. (2020) studied the physical characteristics by varying the temperature (293.4 to 333.4 K) and IL content (10 to 50 wt%) of ammonium functionalised ionic liquids at intervals of 10 wt% and 10 K. The study found that the concentration, density, and surface tension of tetrabutylammonium bromide [TBA][Br] increased, but tetrabutylammonium acetate [TBA][OAC] and tetrabutylammonium hydroxide [TBA][OH] showed the reverse tendency. The result shows that [TBA][Br] and water have a stronger molecular bond than other ILs. All aqueous ILs have an increase in dynamic viscosity with IL concentration. It is well known that ILs' physical with properties deteriorate temperature. The thermophysical properties of three binary liquid solutions of the MDEA, DEA, and AMP with the ionic liquid 1-ethyl-3-methylimidazolium acetate ([Emim][Ac]) T = 293.15–333 at intervals of 10 K was studied by (Shojaeian et al. 2021). Based on the findings, temperature increases resulted in a drop in viscosity, density, and refractive index for both pure and binary systems. Because of the mixture's intricate behaviour, each system's effect of concentration on the thermophysical parameters of the investigated solutions was distinct, and no overarching pattern was discovered.

The impact of density of ammonium-based IL, aqueous tetrapropylammonium hydroxide (TPAOH) at various temperatures (298.15 to 333.15 K) and concentrations (5, 10, 15, 20, and 25 wt%) was studied by (Kartikawati et al. 2018). It was shown that density increases along with temperature and IL concentration. This happens as a result of the molecule's tendency to disperse when both temperature and kinetic energy increase. Yusoff et al. (2014) studied the surface tension and heat capacity of different types of ILs, namely 1-butyl-3methylimidazolium dicyanamide, 1-butyl-3methylimidazolium tetrafluoroborate ([bmim][BF4]), and [bmim][DCA]. The following trend was observed when temperature increases and ionic liquid concentration rises the surface tension tends to decrease. The heat capacity of the mixes increases as temperature rises and ionicliquid concentration decreases. Aqueous ionic liquidamine hybrids may be a superior solvent for CO₂ collection due to their high loading capacity, higher absorption rate, and energy-efficient method. Only a limited number of published investigations could be found on the nonaqueous hybrid solvent composed of monoethanolamine (MEA), tetrabutylammonium hexafluorophosphate [TBA][PF₆]), and ethanol. Physical characteristics of the solvents are thought to be particularly important when examining the rate of absorption and absorption capacity. The study of physicochemical features at a lower temperature (293.15K) is required for CO₂ absorption process. In this work, the physical property behavior of monoethanolamine (MEA), non-aqueous tetrabutylammonium hexafluorophosphate [TBA][PF₆] and ethanol mix at a range of IL concentrations (1-2 wt%) and temperatures (293.15-333.4 K) was studied.

2. Materials and methods

The current investigation employed a minimum purity of 99 weight percent (Laboratory Grade) monoethanolamine (MEA). TetraButylAmmonium Hexafluorophosphate ([TBA][PF₆]), a highly pure ionic liquid from Spectrochem was utilised. The concentrations of MEA and IL ([TBA][PF₆]) are only stated in weight percent. The animated flue gas of 15% CO₂ and 85% nitrogen was commercially purchased. The physical properties were measured by various instruments and the details were mentioned in previous work (Perumal *et al.* 2021).

2.1. Density measurement

The density of MEA and non-aqueous [TBA][PF6] mix (1 to 2 wt%) was computed by adding the sample to the specific gravity bottle (5 cm3). The bottle was maintained in the water bath with a temperature control. The density was calculated for a range of temperatures (293.15-333.15 K). The sample was given 10 to 20 minutes to reach thermal equilibrium at each temperature rise of 10 K (Khan *et al.* 2017). Each experiment is calibrated by measuring the density of Ethanol as a reference fluid.

2.2. Dynamic viscosity and surface tension measurement

The surface tension and dynamic viscosity of blends of non-aqueous $[TBA][PF_6]$ and MEA was measured experimentally for concentrations (1 to 2 wt%) and temperatures (293.15 to 333.15 K). Figure 1a illustrates the cooling setup that included a chiller, a water bath with a temperature controller, a cylindrical tank with a viscometer, and a similar setup for the tensiometer. The chilled water is recirculated at the proper temperature from the chiller to the cylindrical vessel. A K-type thermocouple (± 0 K accuracy) was installed inside the cylindrical jar to detect temperature. A similar arrangement is done with tensiometer for measuring surface tension as shown in Figure 1b.

The heating setup shown in **Figure 1c** consists of a heating coil, a water bath with a temperature controller, a cylindrical tank with a viscometer. The heating coil, which is located inside the water container, is connected to the temperature controller and power supply. The temperature controller keeps the temperature stable during the operation. Hot water is recirculated at the appropriate temperature from the heating bath to the cylindrical vessel. A K-type thermocouple (± 0 K accuracy) was installed inside the cylindrical jar to detect temperature. A similar arrangement is done with tensiometer for measuring surface tension as shown in **Figure 1d**. Each experimental run is calibrated using measurements of ethanol's surface tension and dynamic viscosity.



Figure 1a. Schematic representation of dynamic viscosity measurement in chiller setup



Figure 1b. Schematic representation of surface tension measurement in chiller setup



Figure 1c. Schematic representation of dynamic viscosity measurement in heating setup



Figure 1d. Schematic representation of surface tension measurement in heating setup

3. Results and discussion

3.1. Density

3.1.1. Impact of temperature and concentration on density

Volume fluctuates with temperature, which causes a change in density. The molecules of the solvent mixture push away as its temperature rises because of the rise in kinetic energy. The result is a decrease in density and an increase in material volume. The similar behaviour is reported by Rahim *et al.* (2023). The reduced molecular interactions between molecules result in lesser density because the kinetic energy of molecular interaction between the ionic liquid and solvent becomes weak (Wu *et al.* 2020).

The effect of IL concentration on density was investigated for non-aqueous [TBA][PF₆] and MEA blends at various concentrations. Density increases as the concentrations of [TBA][PF₆] in the MEA mix increase. The Coulombic forces in an ionic liquid strongly attract the unlike charges, when [TBA][PF6] is added to MEA, the volume of IL's ion pair increases and there is a rise in density. Furthermore, increasing the concentration of ionic liquids resulted in a rise in the density of the solution at constant temperature. This is owing to the larger molecular size of fluoride anion, which causes the weaker contact between cation and anion, resulting in a greater density of ILs that was observed, and similar behaviour was also described by Perumal *et al.* (2020). It is possible that the molecules exhibit moderate intermolecular interactions at higher temperatures and significant intermolecular forces at greater concentrations of ionic liquids. (Baj *et al.* 2013). The average kinetic energy of molecules in a material increases with temperature. The molecules travel quicker and collide more frequently, which increases the distance between them. (Mazari *et al.* 2021). The density of the substance reduces as the distance between molecules widens. This behavior is noticed as gas density is directly proportional to pressure and inversely proportional to temperature (Halim *et al.* 2016). **Figure 2a** shows for the specified solvent blend of MEA and [TBA][PF₆], density decreases with increasing temperature.



Figure 2a. Temperature and concentration dependence of density of non-aqueous MEA-[TBA][PF6]



Figure 2b. Concentration dependence of density of non-aqueous MEA-[TBA][PF6] after absorption



Figure 2c. Time and concentration dependence of density of non-aqueous MEA-[TBA][PF6] after absorption

3.1.2. Impact of time and concentration on density

The density and molecular volume of the ionic liquids are related to the amount of CO2 absorption (Yunus et al. 2019). Density increases as the CO₂ solubility increases which causes the volume change. As the concentration of IL increases, CO₂ and mixture form new bonds which will result in new molecules thereby increasing the density of the mixture. The density for different concentration of the non-aqueous blends of MEA and IL was investigated for different absorption time. In the virgin solution MEA interacts with [TBA][PF₆] resulting in huge voids due to steric hindrance from bulk molecules of [TBA][PF6]. As absorption progress with the time, increase in density of the solvent mixture was noted. This trend was seen due to the chemical reactions and van der Waals attraction property of CO₂ molecules with solvents free space (Wang et al. 2023). During CO₂ absorption, the CO₂ molecules occupy the free spaces accessible on the solvent's surface due to the polarity effect. Figure 2b indicates that the density is linearly proportional to concentration of IL due to the accumulation of more CO2. As an amount of time passes CO₂ absorption rises, causing CO₂ to accumulate in the mixture's void spaces and form new bonds, increasing density. The experimental findings of density for the carbon loaded solution is shown in Figure 2c. The concentration of the IL is of the order 2 wt% [TBA][PF₆] > 1.5 wt% [TBA][PF₆] > 1wt% [TBA][PF₆]. Density increases due to the increase of molecular weight of the mixture (Ramkumar et al. 2019).

3.2. Dynamic viscosity

3.2.1. Impact of temperature and concentration on dynamic viscosity

Viscosity is the most important physical component of the gas separation process. As a result, the investigation of temperature and concentration on viscosity is essential to understand gas-liquid kinetics in solvents for a wide range of industrial applications (Wei *et al.* 2020). The effect of temperature and concentration on viscosity of non-aqueous MEA-IL blends is illustrated in **Figure 3**. It has been shown that dynamic viscosity of the non-aqueous MEA-IL blends decreases considerably as temperature rises for various concentration ranges (Li *et al.* 2021). The concentration directly affects the dynamic viscosity of the non-aqueous ILs examined. It was noticed that the viscosity in MEA-[TBA][PF6] mixes increased with the increase in IL concentration (Hafizi *et al.* 2021).

Viscosity rises as IL concentration in solvent mixtures rises because more molecules become present and create new interactions with one another. In the study of CO₂ absorption of IL systems, Zhang *et al.* (2012) showed that Henry's constant falls as more ILs are included in the system, and it was observed the primary cause was increased viscosity, which results from the addition of additional ILs. Higher kinetic forces between molecules cause faster movement, which reduces intermolecular interaction and resulting in lower viscosity at higher temperatures (Boualem *et al.* 2022).

When the IL concentration rises, the viscosity of the solvent mixture also rises. Viscosity rises as IL

concentration in solvent mixtures rises because this results in more molecules entering the mixture and forming new interactions with one another (Latini et al. 2022). Opposing charges passing one another and disrupting the network put limits on ion mobility within an ionic liquid, which increases viscosity as the concentration rises. The barrier to mobility is decreased by the presence of CO₂ by reducing the intermolecular contacts between the cations and anions. Due to the strong solubility and phase behavior of CO₂ in ILs, this has occurred (Theo et al. 2016). It was seen that viscosity increases with the solubility of CO2. Carbon-rich solutions exhibit higher viscosity during absorption due to greater hydrogen bonding between molecules. Higher viscosity hinders mass transfer kinetics between solvent blend and CO₂, resulting in lower absorption capacity. The similar behaviour is reported in Perumal et al. (2023).



Figure 3. Temperature and concentration dependence of viscosity of non-aqueous MEA-[TBA][PF6]

3.3. Surface tension

3.3.1. Impact of temperature and concentration on surface tension

Experiments were conducted to study the impact of temperature and concentration on surface tension at temperatures ranging from 293.15 to 333.15 K. Figure 4a shows that the surface tension data decreases linearly as the concentration of the ionic liquids increases. With an increase in the ionic liquid concentration, the overall intermolecular tensions between the molecules of the solution decrease, resulting in a decrease in surface tension (Hasib-ur-Rahman et al. 2010). The MEA-IL [TBA][PF₆] mixtures surface tension decreased as the temperature was increased for all concentration levels as shown in Figure 4a. At greater temperatures, hydrogen bonding between molecules may weaken, which might explain the phenomenon. The molecules in the solvent mix have greater kinetic energy due to increased temperature and move with greater velocity. As a result, the thermal mobility of the molecules increases, which lessens their intermolecular attraction to one another and causes surface tension to drop at higher temperatures. Surface tension decreases with increasing [TBA][PF₆] concentration in MEA mixes because of fewer molecular interactions, which lowers the gas-liquid interface between the solvent molecules. The similar behaviour is reported in literature (Amirchand *et al.* 2022).



Figure 4a. Temperature and concentration dependence of surface tension of non-aqueous MEA-[TBA][PF₆]



Figure 4b. Concentration dependence of surface tension of nonaqueous MEA-[TBA][PF₆] after absorption





3.3.2. Impact of time and concentration on surface tension

Cohesive forces that hold the molecules at a liquid's surface together are measured by surface tension, when a liquid comes into contact with a gas, such as when carbon dioxide is absorbed into a liquid, the presence of both the gas and any dissolved molecules, such as ionic liquids, can change the liquid's surface tension. After carbon loading surface tension increases linearly with concentration due to high intermolecular forces between ionic liquid solution with CO₂ after absorption as shown in **Figure 4b**. Additionally, the ILs may build up at the liquid-gas interface over time and create a stable interfacial layer there. This layer may serve to raise the liquid's surface tension by effectively separating the liquid phase from the gas phase and reducing the surface area of the liquid that comes into contact with the gas. The IL concentration in the liquid may influence the stability and thickness of this interfacial layer, as well as the liquid's surface tension (Zacchello *et al.* 2017). Therefore, the increase in surface tension with time and concentration of IL after carbon dioxide absorption can be attributed to the pH changes, the intermolecular interactions of the ILs, and liquid-gas interface formation (Torralba-Calleja *et al.* 2013).

Based on these findings, increasing the concentration of ILs in virgin solution lowered surface tension due to the likelihood of lower molecular interactions (Perumal *et al.* 2023). The surface tension increases for the MEA and [TBA][PF₆] blends as the CO₂ absorption progress. **Figure 4c** illustrates surface tension increases linearly with time due to the increase in intermolecular forces between CO₂ and mixtures. The net intermolecular forces of attraction between molecules increase over time during CO₂ absorption in the mixture resulting in increased surface tension.



Figure 5a. Concentration dependence of pH of non-aqueous $MEA-[TBA][PF_6] after absorption$

3.4. pH

3.4.1. Impact of concentration on pH

pH is a crucial parameter in selecting suitable solvent for CO_2 absorption. When the concentration of CO_2 in the solvent mixture increases pH decreases due to the increase in [TBA][PF₆] which is weakly basic as compared to MEA. The Debye-Huckel equation states that as an aqueous solution's ionic strength increases, the activity of the hydroxide ion decreases. By improving the solubility of CO_2 in the solution, [TBA][PF₆] can be added to this mixture to further raise the acidity of the solution. It has been demonstrated that [TBA][PF₆] has a high CO_2 solubility and can increase the solubility of CO_2 in the solvent mixture (Numpilai *et al.* 2024). It was found the interaction of [TBA][PF₆], MEA, and ethanol may form

several weak acids, increasing hydrogen ions levels while decreasing pH levels in the solution. Drop in pH for the solvent blends was noted during the absorption due to presence of CO_2 (Choi *et al.* 2021).

3.4.2. Impact of concentration and time on pH

The concentration of hydrogen ions (H+) in a solution is represented by its pH value. A weak acid, such as ethanoic acid or propanoic acid, can nevertheless be formed when carbon dioxide dissolves in a non-aqueous solvent, such as an organic solvent like ethanol or acetone (Shukla *et al.* 2019). **Figure 5a** shows an increase in the concentration of hydrogen ions (H+) in the solution. As a result of the weak acid being produced from carbon dioxide and the solvent leads to a fall in pH (Kaviani *et al.* 2018). The pH lowers over time as a result of the CO₂ gas being absorbed by the combination of ionic liquids as shown in **Figure 5b**.



Figure 5b. Time and concentration dependence of pH of nonaqueous MEA-[TBA][PF₆] after absorption

3.5. Carbon loading

3.5.1. Impact of concentration and time on carbon loading

Ma *et al.* (2018) showed that the formation of a solid network of anions and cations, with CO_2 filling the interstices in the fluid, is what causes CO_2 solubility in ionic liquids. The slight differences in cation structure do not, however, have a major impact on the dominant CO_2 - anion interactions. As a result, in ionic liquids, anion affects CO_2 solubility whereas the cation does not affect the process (Yang *et al.* 2024). **Figure 6a** illustrates the effect of carbon loading on solvent mixture concentration.

The cation is considered to play a supportive function in the breakdown of CO_2 and anion is thought to perform a major role. The researchers discovered that CO_2 interacts largely with the [PF₆] anion, independent of the cation. Previous studies using in situ ATR-IR spectroscopy shown that the anion [PF₆] and CO_2 might interact positively. According to the spectroscopic data, the interaction is Lewis acid-base in nature, with the anion functioning as the Lewis base and the CO_2 as the Lewis acid. The spectroscopic results also provide compelling evidence of the interaction between CO_2 and the [PF₆] anion. According to Brennecke *et al.* (2010) the free volume mechanism is predicted to play a significant role in the dissolution of CO₂ because anion- CO₂ interactions alone are insufficient to explain the solubility. Given that the liquid volumes of ILs are not significantly altered when large amounts of CO₂ are dissolved, it is expected that CO₂ molecules will be hosted in the liquid's free spaces (cavities) during a free-volume process. The more CO2 can be dissolved by an anion the more fluoro groups it possesses. This was done by investigating a wide range of ILs, and it was discovered that Henry's constants of CO2 may be related to the excess enthalpy of CO₂ dissolution in IL (Abraham et al. 2019). This demonstrated that increased solubilities are connected with higher combination exothermicities. Moreover, the anticipated intermolecular interactions (van der Waals, hydrogen bonds, and electrostatic interactions) between species in a fluid phase. The effects of different interactions on the solubility of CO₂ in Ionic Liquids were studied, and the findings show that van der Waals forces dominate the process (Shohrat et al. 2022). The influence of absorption time and concentration on carbon loading was studied experimentally. Figure 6b illustrates carbon loading for solvent blends increases with increased CO₂ absorption time. It was noted that addition of IL to MEA caused increased carbamate production leading to better CO2 absorption. It was also noted that the presence of fluorinated anion of the IL promotes CO2 loading in biphasic absorbents. This behaviour has already been observed in literature (Yang et al. 2023).









4. Conclusion

The thermophysical characteristics study of non-aqueous solvent mix of monoethanolamine (MEA) and tetrabutylammonium hexafluorophosphate $[TBA][PF_6]$ were determined experimentally at different concentrations and temperatures. Physical attributes were observed to rise with higher concentrations of [TBA][PF₆] and decrease with higher temperatures as the kinetic energies of the molecules increase, which causes their intermolecular forces of attraction to weaken. Anion plays an important role in CO₂ absorption than cation of ILs. Carbon loading was higher at 2wt% IL+28wt% MEA when compared to other compositions, and this may need less regeneration energy than 30wt% MEA, despite the higher viscosity for this composition. The non-aqueous blend of MEA and [TBA][PF₆] can be a good absorbent for CO₂ separation.

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