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SYNTHESIS AND CHARACTERISATION OF 2-(METHYLAMINO)ETHANOL-BASED DEEP EUTECTIC SOLVENTS FOR CO_2 CAPTURE

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Abstract: In recent years, deep eutectic solvents (DESs) have attracted the interest of many researchers for application in a wide range of industrial and scientific fields, including carbon dioxide (CO_2) capture. DESs exhibit favourable solvent properties for CO_2 removal applications; hence, they have become promising alternatives to common amine solutions and ionic liquids (ILs). In this context, a novel DES was synthesised by mixing 2 (methylamino)ethanol (2-MAE) as a hydrogen bond donor with choline hydroxide (ChOH) as a hydrogen bond acceptor with a molar ratio of ChOH:2-MAE of 1:1. The solubility of CO_2 in the prepared systems was determined and characterised before and after CO_2 absorption by measuring their physicochemical properties (density and viscosity) and analysing their FTIR spectra. The results showed that the DES and 2M DES aqueous solutions exhibited CO_2 absorption capacities comparable to those of other reported DESs. These physicochemical properties were comparable to those reported in the literature. Besides, the FTIR analysis of the studies systems after absorption indicates the formation of carbamate.

Keywords: Deep eutectic solvents, 2 (methylamino)ethanol, choline hydroxide, physiochemical properties, CO_2 solubility.

1. Introduction

One of the primary factors in global warming and climate change is seen to be the growing and continuous emission of the greenhouse gas carbon dioxide into the atmosphere as a result of the increased demand for fossil fuels in the industrial activity and automation era. (Dincer & Abu-Rayash, 2020). According to reports, carbon dioxide (CO_2), which accounts for 60% of all greenhouse gases released into the environment, is primarily produced by the steel, cement, petrochemical, energy, and transportation industries (IEA, 2023; Kassim, Sairi, Yusoff, Alias, & Aroua, 2016). In 2019, the combustion of fossil fuels in the United States was responsible for around 80% of all greenhouse gas emissions (Agency, 2021, April 14). According to Earth System Models (ESMs), the average results of ESMs predict that the temperature will rise by between 1.0 and 3.7 °C in the twenty-first century (Anderson, Hawkins, & Jones, 2016). These increasing signs thus necessitate action to create effective and affordable technology to reduce carbon dioxide emissions at their source.

Since the first decade of the 20th century, much research has been done to create effective methods for capturing and storing carbon dioxide. Numerous developments for CO_2 collection, including physicochemical and biological methods, have undergone extensive research (Nanda, Reddy, Mitra, & Kozinski, 2016; Zubeir, Lacroix, Meuldijk, Kroon, & Kiss, 2018). Today, the majority of CO_2 is removed or collected by physical and chemical absorption employing solvents including aqueous ammonia amines, ionic liquids, amino acid salts, and deep eutectic solvents (P.-C. Chen & Lin, 2018). Ionic liquids (ILs) and deep eutectic solvents (DESs) are two solvents that can be developed to be utilised broadly, effectively, and efficiently in large-scale applications. They are among the most promising, new, and environmentally friendly solvents (Suzuki, 2018; Zhang, De Oliveira Vigier, Royer, & Jérôme, 2012).

Since their potential was found, research on amine solvents and ionic liquids (ILs) has increased during the past 20 years. Deep eutectic solvents (DESs) were designed to address the shortcomings of traditional ionic liquids (ILs), including their high cost of synthesis, toxicity, and poor biodegradability. DESs were presented and classed as a unique type of ionic liquid (C.-C. Chen et al., 2021; Zurob et al., 2020). DESs are defined as a combination of two to three inexpensive, safe, and mostly quaternary ammonium salts or halide salts with hydrogen bond donor (HBD) components (Smith, Abbott, & Ryder, 2014). These mixes mostly consist of nonsymmetric ions, which have low melting temperatures due to their low lattice energy. As a result, the creation of DES is easier than that of ILs; in fact, nearly all DESs may be synthesised by combining a hydrogen bond acceptor (HBA) and a hydrogen bond donor (HBD) (Tomé, Baião, da Silva, & Brett, 2018; Zhang et al., 2012).

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Among all DES's potential uses, absorbing CO_2 appears to provide the best prospects for sustainability and effectiveness (Smith et al., 2014; Zurob et al., 2020). However, a large variety of these solvents may be generated due to the ease with which DESs can be made utilising HBD and salts as HBA. As a result, there will be much interest in the use of these solvents in CO_2 absorption in the future years.

Numerous studies have been conducted to develop efficient technology, to tackle CO_2 emissions from its sources, such as using novel green solvents such as deep eutectic solvents (DESs). Several studies have been conducted on absorbing CO_2 using an aqueous mixture of 2- (methylamino)ethanol (2-MAE), and good gas solubility was observed in certain conditions. Therefore, the great performance of 2- (methylamino)ethanol (2-MAE) shows potential to be used as a hydrogen bond donor (HBD) and different types of salts as hydrogen bond acceptor HBA. However, up to our knowledge, the scope of some of these studies was limited to certain types of hydrogen bond acceptors and certain absorbing conditions. Therefore, this work aims to synthesise and characterise 2 (methylamino)ethanol (2-MAE)-Based DES and 2M DES aqueous solution as well as to evaluate their performance on the capacity of CO_2 absorption.

2. Materials and methods

2.1 Materials and Synthesis

Materials used in this work are 2- (methylamino)ethanol (2-MAE), choline chloride (ChCl), choline hydroxide solution 46 wt. % in H_2O were purchased from Sigma-Aldrich. Carbon dioxide (CO_2) was purchased from Alpha Gas Solution. All materials were used without further purification.

2.2 Synthesis of DES

ChOH:2-MAE was synthesised according to the literature (Hussin, Aroua, & Yusoff, 2021). An equimolar mixture of 45 wt% ChOH in methanol and 2-MAE was mixed in a round bottom flask with heating and stirring at 353.15 K for 30 minutes. Then, the solvent removal takes place in a rotary evaporator for 2 hours under reduced pressure. The sample was then dried in a vacuum oven overnight with 70 mbar pressure to remove excess moisture and the acquired DES was obtained and kept in tightly sealed bottles.

2.3 Sample preparation

2M DES aqueous mixtures were prepared by weighing each compound using a calibrated single pan digital balance (HR-250AZ, A&D Japan) with an accuracy of 0.1 mg. To reduce moisture absorption from the environment, all mixes were maintained in tightly sealed bottles.

2.4 DES and aqueous solutions characterisation

The DES and its 2M aqueous solutions were characterised by measuring their physicochemical properties before and after CO_2 absorption (density and viscosity). The density measurements were conducted using Anton Paar DMA 4200 M, at 30 °C and ambient pressure. Viscosity was measured using a BROOKFIELD viscometer at 30, 40 and 50 °C and ambient pressure. The systems were further characterised using FTIR to identify the functional groups and the DES formation.

2.5 CO_2 solubility measurements

The performance evaluation of DESs in absorbing CO_2 was accomplished by the method described by (Kassim et al., 2016). The experimental setup for measuring CO_2 uptake is illustrated in Figure 1. The schematic diagram mainly consists of an air vent tube, a welded stirrer assembly, an inlet gas tube, and a thermocouple. The experiment is initiated by flushing CO_2 through the system to purge the air out of the gas reservoir. Once the reservoir is purified, it is filled with pure CO_2 from the gas tank after that CO_2 is heated and pressurized into the required experimental conditions. The thermostated high-pressurized reactor is filled with a known volume of fresh solutions or DES (20 mL).

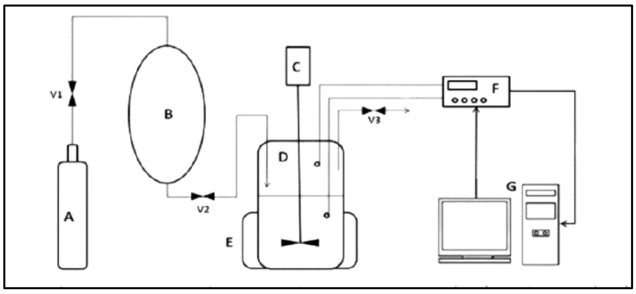


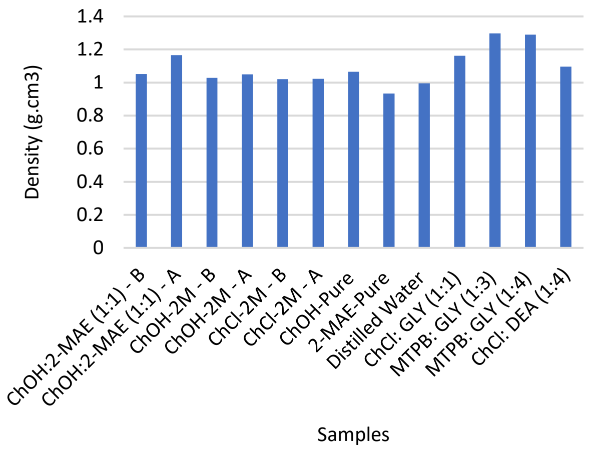
Figure 1. Schematic diagram of the experimental set-up for measuring the CO_2 solubility: A. Gas (CO_2) cylinder, B. Gas (CO_2) reservoir, C. Motor, D. High-pressure reactor vessel (equilibrium cell), E. Heater, F. Reactor controller, G. PC graphical user interface, V1. Control valve, V2. Needle valve, V3. Pressure relief valve.

3. Results and discussion

3.1 Characterisation of DES and its aqueous solution

3.1.1 Density

The densities of the synthesised systems were measured at 30 °C under ambient pressure. The uncertainties in the density data were within a range of ± 0.00001 $g.cm^{-3}$. Previous work data for other synthesised DESs were used for comparison with the values of this work, as shown in Figure 2. The density experimental data showed that the density decreased when HBD (amine or water) was added to the salt (ChOH or ChCl). Additionally, the density decreased as the molar ratio of HBD increased, as shown in the literature-reported data for the MTPB: GLY DES system (Shahbaz, Bagh, & Mjalli, 2013). This might be due to the increase in the repulsion forces between the salt and HBD, which leads to an increase in the volume of the solution as the HBD increases (Pishro, Murshid, Mjalli, & Naser, 2020). Comparing the densities of the studied system before and after carbonation, as shown in Figure 2, the density increased in all the synthesised systems after absorption, where the increase was significant, up to approximately 11 % in the solvent that involved both chemical and physical absorption (ChOH: 2-MAE). However, the density increases in (ChOH (2M) and ChCl (2M)) were insignificant which was found to be less than around 2.0 %. This could be due to the chemical absorption influencing the packing or chemical structure of the DES; therefore, because the density depends on the packing or chemical structure of the DES, this can also be applied to explain the increase in density after absorption. In the case of the ChOH: 2-MAE system, the density data are comparable with other reported DESs (Murshid, Mjalli, Naser, Al-Zakwani, & Hayyan, 2019).



B: Before absorption; A: After absorption;

Figure 2: Density of the studied systems.

3.1.2 Viscosity

Viscosity is an essential solvent property for designing the equipment used in any absorption process. Therefore, the viscosity of 2-MAE-based DES and its aqueous solutions were measured before and after CO_2 absorption at three different temperatures and ambient pressures. The experimental results are listed in Table 1 and illustrated in Figure 3. Some other viscosities of reported DESs are used for comparison with the data of this work, and the viscosities of ChCl: DEA and TBAB: AP are estimated using the experimental models (Murshid et al., 2019; Nowosielski, Jamrógiewicz, Łuczak, Śmiechowski, & Warmińska, 2020).

Based on the reported and experimental results, the DES viscosity depends on the molar ratio, temperature, and type of salts and HBD used. The data showed an exponential decrease in the viscosities of all DESs and aqueous solutions as the temperature increased. This viscosity behaviour is due to the higher temperatures, which increase the kinetic energy of the molecules and ions and weaken the attractive forces between them, allowing the molecules to move easily, thereby decreasing the viscosity. Additionally, based on the reported binary DESs, the viscosity decreased as the molar ratio increased, as can be seen in the case of TBAB: AP DES, where the viscosity decreased by almost 40 % at 30 °C when the molar ratio increased from 1:4 to 1:8.

The viscosity of the solvents used in this study decreased after CO_2 absorption. The viscosities of ChOH: 2-MAE, ChOH, and ChCl decreased at 30 °C by approximately 32, 29, and 13 %, respectively. However, different analogous relations were observed where they reported an increase in the viscosity after absorption for binary and ternary ethanolamine-based DESs (Sarmad, Shokat, & Nikjoo, 2020). For example, the viscosity of ChCl: EA (1:7) increased at 25 °C by almost 16 times that before absorption. Moreover, comparing the studied DES viscosity with other reported DESs, ChOH:2-MAE (1:1) ^{B} has the second highest value after ChCl: DEA (1:4) ^{B} with a value of 290.5 cP at 30 °C while the lowest reported is ChCl: EA (1:7) ^{B} with a value of 32.480 cP at 30 °C. lastly, even though pure amine (2-MAE) has slightly better CO_2 absorption performance compared to ChOH: 2-MAE (1:1), 2-MAE is found to be highly viscous after absorption experiment and can not be measured with the available viscosity equipment in the lab.

Table 1: Viscosity of synthesised and reported DESs as a function of temperature.

Sample Name	Abbr.	Reference
ChOH:2-MAE (1:1)	DES 1 ^{B}	This work
ChOH:2-MAE (1:1)	DES 1 ^{A}	This work
ChOH-2M	Soln 1 ^{B}	This work
ChOH-2M	Soln 1 ^{A}	This work
ChCl-2M	Soln 2 ^{B}	This work
ChCl-2M	Soln 2 ^{A}	This work
ChOH-Pure	Soln 3 ^{B}	This work
2-MAE-Pure	Soln 4 ^{B}	This work
ChCl: EA (1:7)	DES 15 ^{B}	(S. Sarmad, Xie, Mikkola, & Ji, 2017)
TBAB: EA (1:6)	DES 16 ^{B}	
ChCl: DEA (1:4)	DES 17 ^{B}	(Murshid et al., 2019)
TBAB: AP (1:4)	DES 18 ^{B}	(Nowosielski et al., 2020)
TBAB: AP (1:8)	DES 19 ^{B}	

B: Before absorption; A: After absorption; EA: Ethanolamine; AP: 3-amino-1-propano; TBAB: Tetrabutylammonium bromide; DEA: Diethanolamine.

3.2 CO_2 solubility of DES and aqueous solutions

The solubility of CO_2 in the studied systems, of DES, pure 2-MAE, and 2M aqueous solutions of choline hydroxide, choline chloride, and DES, were determined at 30 °C under the pressure range of 50.0 – 150.0 psi. Figure 4 illustrates all the measured experimental data for all studied systems in mol CO_2/mol solvent. The solubility of CO_2 in DESs appears to be influenced by various factors, including pressure, the characteristics of the hydrogen bond donor (HBD), the alkyl chain length in both HBD and hydrogen bond acceptor (HBA), the properties of salts, the strength of hydrogen bonding within the solutions, the molar ratio, and the molecular weight of the components (S. Sarmad et al., 2017).

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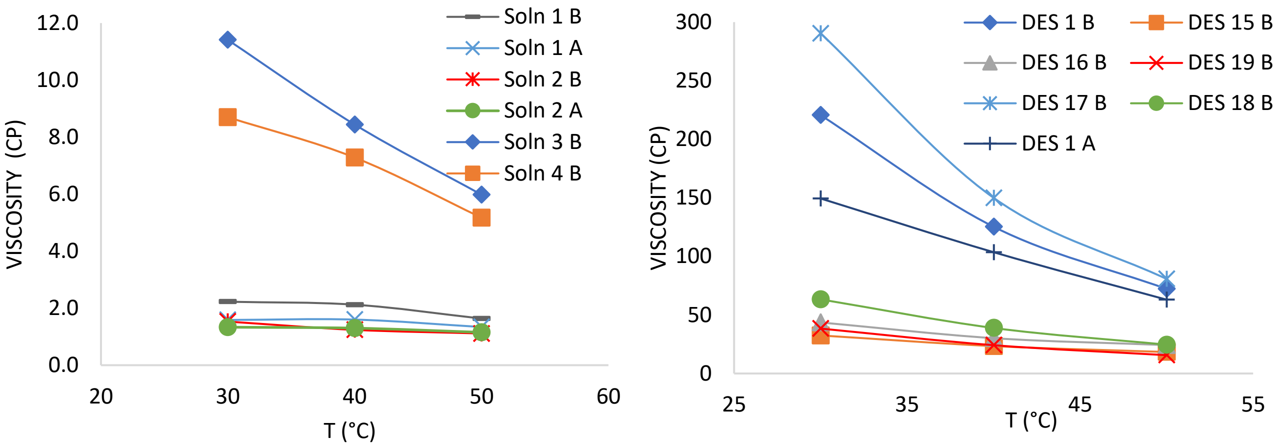

Figure 3: Viscosity of the studied systems and reported DESs as a function of temperature.

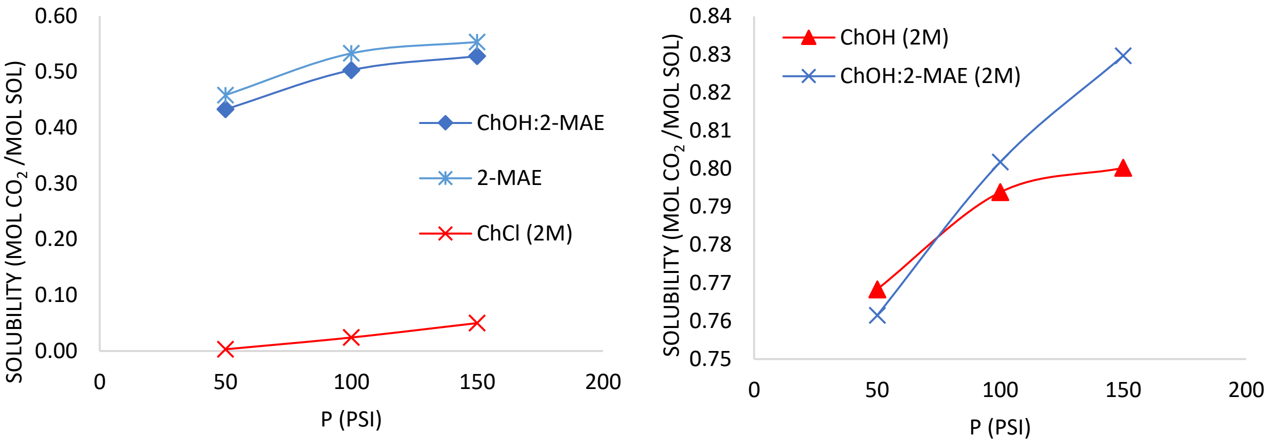

Figure 4: CO$_2$ solubility in terms of α_{CO2} as a function of pressure at T = 30 °C.

Table 2: Composition and molar ratio of studied DES and other reported DESs.

DES No.	HBA	HBD	Molar ratio (HBA: HBD)	Temperature °C	Reference
DES 1	ChOH	2-MAE	1:1	30	This work
DES 2	ATPPB^1	DEG^1	1:4	30	(Ghaedi et al., 2017)
DES 3	ATPPB	DEG	1:10	30	
DES 4	ATPPB	TEG^1	1:4	30	
DES 5	ATPPB	TEG	1:10	30	
DES 6	ChCl	EA	1:7	25	(Sarmad et al., 2020)
DES 7	ChCl	EA: MDEA^1	1:7:5	25	
DES 8	TBPB^1	PhOH^1	1:4	40	(Wang et al., 2019)
DES 9	ATPPB	PhOH	1:4	40	

ATPPB: Allyltriphenyl phosphonium bromide; EA: Ethanolamine; MDEA: Methyldiethanolamine; DEG: Diethylene glycol; TEG: Triethylene glycol; TBPB: Tetrabutylphosphonium bromide; PhOH: phenol.

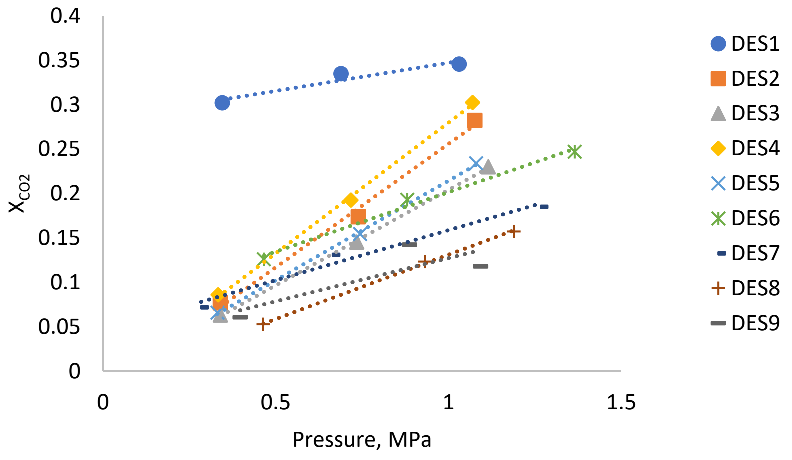

Figure 5: CO_2 solubility in mole fraction (**X_{CO2}**) as a function of pressure in synthesized and reported DESs at T = 30 °C, 25 °C, and 40 °C.

The described findings demonstrate that the tested system's capability for CO_2 absorption maintains consistency at the observed temperature. As seen in the two figures, there is a linear trend between pressure increases and the solubility of CO_2 in all solvents. This trend shows that physical absorption is the prominent route by which CO_2 is absorbed by the solvents. The absorption of pure 2-MAE, amine-based DES, and its aqueous solution, on the other hand, is mediated by a mix of chemical and physical processes. Therefore, based on the measured experimental data of the systems studied, the performance order is ChOH: 2-MAE (2M) > ChOH (2M) > 2-MAE > ChOH: 2-MAE > ChCl (2M). It is worthwhile to compare the solubility of CO_2 between the studied DES (ChOH:2-MAE) and reported DESs. The absorption capacities of various DESs were studied based on different HBA and HBD. Table 2 and Figure 5 list and illustrate CO_2 solubility in mole fraction (**X_{CO2}**) as a function of pressure. Hence, based on the tabulated data, the data reported corresponds with the literature finding where all DESs demonstrated an increase in the CO_2 absorption capacity as the pressure is increased. Additionally, it can be concluded that the studied DES (ChOH:2-MAE) has good performance compared with other reported DESs and it can be considered as a promising DES.

3.3 FTIR

Figure 6 (a) and (b) illustrate the FTIR spectra for 2M Choline chloride and 2M choline hydroxide solution before and after CO_2 absorption. The major peaks are tabulated and listed in Table 3. For both 2M Choline chloride and 2M choline hydroxide solutions, the broad absorption peak at 3302 cm^{-1} is characteristic of the O–H stretching vibration of choline hydroxide. The peak at 1481 cm^{-1} is due to the C–H bending vibration of alkanes. The peaks at 1296 and 1088 cm^{-1} were attributed to the C–N and C–O stretching vibrations, respectively. As shown in Figure 6 (a), no significant difference was observed in the spectra of the choline chloride solution before and after CO_2 absorption. In contrast, the spectrum for 2M choline hydroxide shows a peak at 1351 cm^{-1}, which is characterised by HCO_3^- formed during the CO_2 absorption process. Figure 6 (c) shows the FTIR spectra of pure 2-MEA before and after CO_2 absorption. Major peaks are tabulated and assigned in Table 3. The spectra show the presence of carbonate peaks that reflect the formation of the carbamate salt of 2-MAE after absorption of CO_2 at 1378, 1296, and 1259 cm^{-1}. Figure 6 (d) illustrates the FTIR spectra for pure ChOH:2-MAE; before and after CO_2 absorption. Major peaks are tabulated and assigned in Table 3. The spectra showed peaks that were found in the respective singular components; however, some peaks may overlap. Owing to the large number of absorbing species in the infrared spectra, it is difficult to accurately assign all absorption bands. Some bands were close to each other, very broad, and/or strongly overlapping.

4. Conclusion

In this study, we successfully synthesised a 2 (methylamino)ethanol (2-MAE)-based deep eutectic solvent (DES) and a corresponding 2M aqueous solution. Our evaluation focused on the CO_2 absorption capacity at 30 °C and pressures reaching 150 psi. Furthermore, we conducted comprehensive characterisations of both the prepared solutions and the DES, encompassing measurements of their physicochemical properties, density, viscosity, and FTIR analysis. The results revealed a linear correlation between the CO_2 absorption capacity and increasing pressure across all the solvents tested. Notably, compared to other reported DESs, ChOH:2-MAE exhibited a promising performance. Pre- and post-absorption measurements of the physicochemical properties of the studied systems were conducted, in addition to comparisons with other reported DESs. FTIR analysis of the studied systems after absorption indicated the formation of carbamate. This research highlights the promising performance of the studied DES compared with the conventional amine (2-MAE) solvent and other reported DESs.

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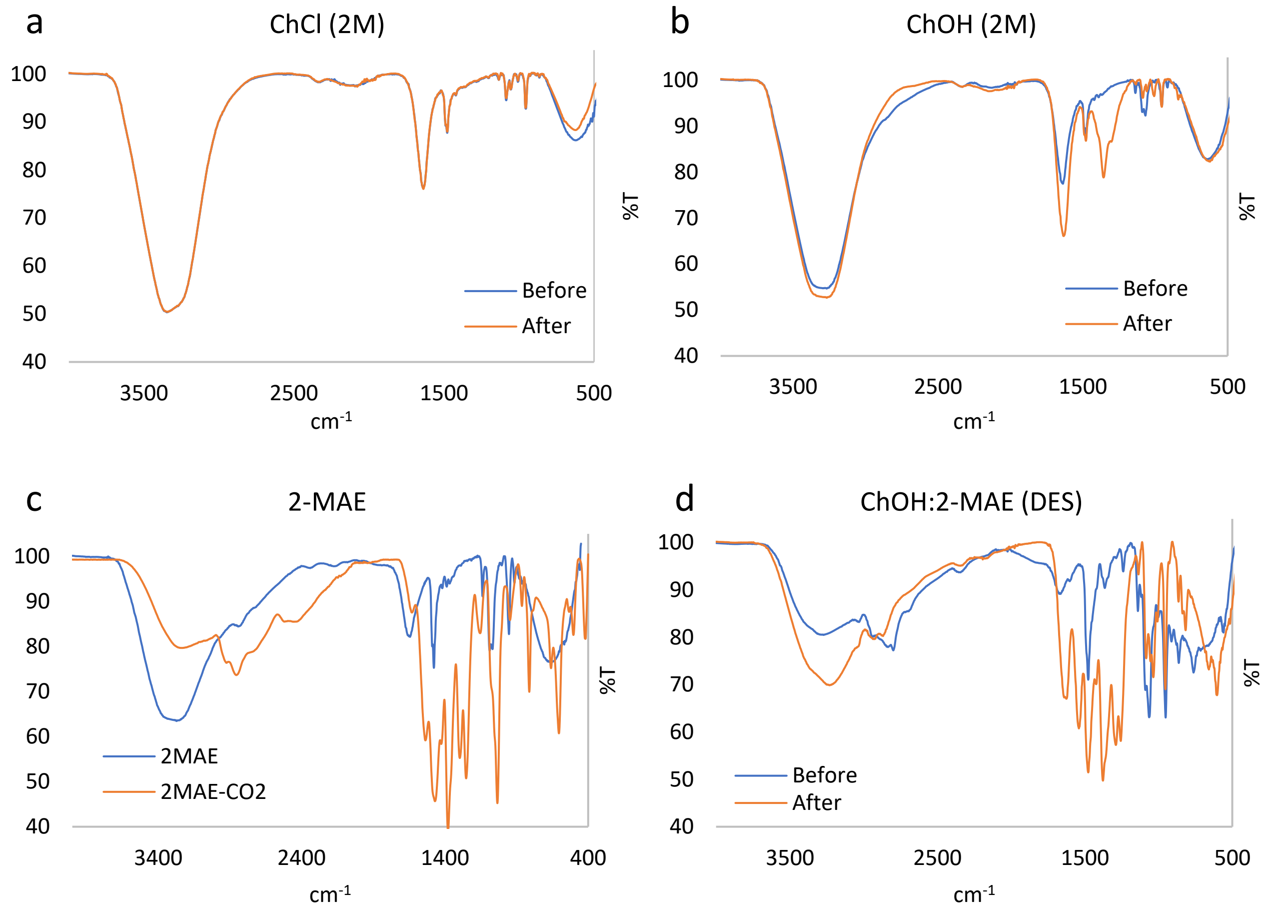

Figure 6: FTIR spectra of DES and 2M aqueous solutions; a: ChCl (2M); b: ChOH (2M); c: pure 2-MAE; c: ChOH:2-MAE (DES)

Table 3: IR peak assignment for the studied systems.

Assignment	**ChCl (2M)**		**ChOH (2M)**		**2-MAE**		**ChOH:2-MAE**	
	Peak	**Frequency**	**Peak**	**Frequency**	**Peak**	**Frequency**	**Peak**	**Frequency**
-OH, -NH stretching	50.5	3334	54.8	3278	64.0	3234	81.0	3227
-NH bending	76.2	1637	78.2	1631	84.3	1626	91.8	1621
-CH$_2$-OH bending	87.8	1479	87.5	1477	81.0	1470	71.1	1477
-C-N- bending	94.5	1085	-	-	-	-	-	-
-C-O- bending	97.1	1051	-	-	-	-	-	-
Carbonates band	-	-	78.9, 86.7	1356, 1304	-	-	-	-
Carbamates band	-	-	-	-	39.1	1378	49.8	1378
-CH$_2$-, -CH$_3$ stretching	-	-	-	-	84.7, 84.6	2896, 2847	80.0,80.1	2917, 2860
stretching of NCOO–	-	-	-	-	55.3, 54.0	1296, 1259	57.4,58.1	1287, 1256
-NH-CO$_2$	-	-	-	-	83.0	1153	-	-

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