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Effect of L-arginine on Solubility of CO₂ in Choline Chloride + Glycerol Based Deep Eutectic Solvents

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Abstract

In the present study, measurements on the solubility of CO₂ based on the pressure drop method in mixtures of (choline chloride (ChCl) + glycerol) + L-arginine at various molar ratios (1:2:0, 1:2:0.1, 1:2:0.2, 1:3:0, 1:3:0.1, 1:3:0.2, 1:4:0, 1:4:0.1 and 1:4:0.2) were experimentally screened at 303.15 K and pressures up to approximately 20 bar. Based on the results of this screening, (ChCl + glycerol) + L-arginine mixture at 1:2:0.1 molar ratio exhibited the highest solubility of CO₂ and further experimental measurements on the solubility of CO₂ in this mixture were carried out at temperatures ranging from 303.15 to 323.15 K and pressures up to approximately 70 bar. The effect of L-arginine on the solubility of CO₂ in ChCl + glycerol based DESs were observed. The results showed that L-arginine improved the solubility of CO₂ in (ChCl + glycerol) + L-arginine mixtures. Besides, the solubility of CO₂ increased with increasing pressure and decreased with increasing temperature. Henry's law constant, enthalpy and entropy of dissolution were calculated from the correlation of experimental solubility data.

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Keywords: Deep eutectic solvents; choline chloride; glycerol; L-arginine; solubility of CO₂

1. Introduction

Carbon dioxide (CO₂) is one of the main acid gases which critical to be removed from the source of energy like natural gas. High content of CO₂ will diminish the burning efficiency of natural gas. One of the most effective and reliable technologies for capturing CO₂ from process gas is chemical absorption by aqueous alkanolamine solutions such as monoethanolamine (MEA), diethanolamine (DEA) and methyl diethanolamine (MDEA) [1]. Despite the good performance of high loading capacity of CO₂, several drawbacks of these amine based solvents are observed include amine oxidation degradation, high energy demand for regeneration. Moreover, the emissions of degradation products can cause serious environmental impact and human health risks [2,3]. Recently, ionic liquids (ILs) are gaining much attention to be outstanding alternative choices for CO₂ capture due to their various characteristics such as good thermal and chemical stability, non-flammable, negligible vapor pressure, recyclable and high CO₂ solubility are remarkable [4-7]. However, ILs still have several weaknesses such as high production cost, complicated synthetic and purification processes [8].

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Over the past few years, deep eutectic solvents (DESs) as green alternative solvents have been studying comprehensively in many research areas. DESs are a type of ILs which also have an ionic character, as DESs have both ionic and non-ionic components connected by a hydrogen bonding network. The first generation eutectic solvents were based on mixtures of quaternary ammonium salts with hydrogen bond donors (HBD) such as amines and carboxylic acids. DESs have a very high potential as promising green solvents that can be tuned to any targeted application and the variety of possible combinations of the starting components provides a good way to control the physical properties of DESs [9]. DESs are found to possess similar solvent properties with ILs while having several advantages over the latter. They are easy to be prepared in high purity at low cost as compared to ILs. Furthermore, they are non-toxic and can be constituted from natural and renewable non-toxic

bioresources, wide liquid range, negligible vapor pressure, water compatibility and good thermal stability [10]. Due to these attractive advantages and their potential use, DESs probably present to be an alternative environmentally friendly solvent for the industrial requirements for large scale applications.

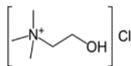
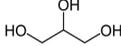
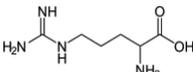
The most of the DESs studied in the literatures are ChCl-based in combination with very different types of HBDs. ChCl which is a quaternary ammonium salt that is non-toxic, biodegradable and relatively inexpensive. Choline cation belongs to an important class of vitamin B family which plays an essential role in our daily life by assisting in various metabolic mechanisms [11]. Glycerol is a non-toxic, colorless, odorless, viscous liquid which is derived from both natural and petrochemical feedstock. Glycerol has been successfully used as a HBD to form DES with ChCl [12-15]. As DESs are sharing too many advantages, one of the applications that currently being explored is the solubility of CO₂ in DESs and many new DESs have been rapidly developed as designer solvents for this purpose [13]. Lin et al. measured the CO₂ solubility using the mixtures of ChCl + glycerol and ChCl + ethylene glycol and reported that the CO₂ solubility in these DESs are comparable to that of imidazolium based ILs [16]. Recently, the application of ternary deep eutectic solvents (TDESs) for CO₂ solubility was also studied by Sze et al. [15]. In their study, TDESs comprising of ChCl, glycerol with various superbases (i.e., 1,5-diazabicyclo[4.3.0]-non-5-ene (DBN), 7-methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (MTBD), 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), etc.) were used and the effect of superbases on CO₂ solubility were highlighted. Among the third components that are used for DES synthesis, amino acids are one of the alternative components that have been used before. Since amino acids possess the characteristic and functional group similar to the amines are expected to perform the similar task for CO₂ capture. Sistla et al. [17] studied the solubility of CO₂ in synthesized ILs consist of butylmethylimidazolium ([bmim]⁺) as cation with different amino acids as anion and the results showed that the highest CO₂ solubility was observed with L-arginine ([bmim][ARG]) (0.62 mol CO₂/mol IL at 298 K and 2 bar). Song et al. [18] also studied the solubility of CO₂ in various aqueous amino acid based salt solutions where maximum CO₂ absorption was achieved with L-arginine (1 mol CO₂/mol solvent). Based on the literature studies, L-arginine presented a good performance for CO₂ absorption. However, additional studies are required to establish the extent of CO₂ absorption using new DESs. Therefore, in the present study L-arginine will be used as a third component for ChCl + glycerol based DESs. The objectives of the present study are to synthesize (ChCl + glycerol) + L-arginine mixtures at various molar ratios for CO₂ capture application and to study the effect of temperature, pressure and molar ratio of L-arginine on solubility of CO₂ in synthesized mixtures. The Henry's law constant, enthalpy and entropy of dissolution were also calculated from the correlation of experimental solubility data and presented.

2. Materials and methods

2.1. Materials

Choline chloride ($\geq 98\%$ mass fraction purity, Sigma Aldrich), glycerol ($\geq 99\%$ mass fraction purity, Merck) and L-arginine ($\geq 98\%$ mass fraction purity, Sigma Aldrich) were used as received without further purification. The details of chemical structure, molecular formula and molar mass of the chemicals used are presented in Table 1. Purified gases supplied by Mox-Linde Gases Sdn Bhd, Malaysia were used for the CO₂ solubility measurements. The gases used are: carbon dioxide with purity 99.9% and nitrogen with purity 99.9%.

Table 1. The chemical structure, molecular formula and molar mass of the chemicals used.

	Choline chloride	Glycerol	L-arginine
Chemical structure			
Molecular formula	C ₅ H ₁₄ ClNO	C ₃ H ₈ O ₃	C ₆ H ₁₄ N ₄ O ₂
Molar mass (g·mol ⁻¹)	139.62	92.09	174.20

2.2. Synthesis

Nine (ChCl + glycerol) + L-arginine mixtures were synthesized in an air-tight sampling bottles which were immersed in oil bath at 353.15 K and stirred at 400 rpm until a homogeneous liquid were formed. Before synthesizing, the precursors namely, ChCl and L-arginine were dried at 358.15 K in order to remove the possible moisture content. ChCl + glycerol based DESs were first synthesized at different molar ratios 1:2, 1:3 and 1:4 and different molar ratios of L-arginine (0.1 and 0.2) were subsequently added into the pre-prepared (ChCl + glycerol) based DESs and the mixtures were then stirred at 353.15 K and 400 rpm until homogeneous liquids were formed. All synthesized mixtures were left overnight and sealed with parafilm film to ensure that no recrystallization and precipitation occurred. The samples were then dried under vacuum ($T = 353.15$ K, $P = 0.5$ bar) for 48 hours and kept in sealed glass vials and further used for the measurement of CO_2 solubility. During synthesizing process, no purification steps and no additional solvents were employed. The water content of synthesized mixtures were determined using Coulometric Karl Fischer titrator, DL 39 (Mettler Toledo) and the estimated water content of all mixtures were ≤ 0.00285 (mass fraction). The sample weight was measured using an analytical balance with an estimated uncertainty of ± 0.001 g.

2.3. CO_2 Solubility Measurement

CO_2 solubility measurements were carried out based on an isochloric method where is the volume of the system is held constant as well as the temperature and the pressure difference is recorded during gas absorption into the solvent and also called as the pressure drop method. The CO_2 solubility in all synthesized mixtures was measured using a SOLTEQ High Pressure Gas Solubility Cell (Model: BP-22). The components of solubility cell consist of a jacketed gas mixing vessel (3L) where the gas pressure was raised up to 70 bar, a jacketed equilibrium cell (50 mL) where the solubility measurement were completed. The temperature of the system was controlled by thermostat water bath JULABO with reported accuracy of ± 0.1 °C and the temperature inside the mixing vessel and solubility cell was measured with YOKOGAWA (7653) digital thermometer with reported accuracy of ± 0.01 °C. Other associated components are including: liquid feed pump, gas booster, vacuum pump, magnetic stirrer, liquid degassing unit and instrumentations such as mass flow controllers, pressure and temperature indicators.

For the operation of solubility cell. Primarily, both mixing vessel and equilibrium cell were purged with nitrogen and vacuumed. After both cells are cleaned and cleared, the mixing vessel was pressurized with CO_2 at the desired pressure and heated to the desired temperature. Approximately 5 ml of sample was injected into the equilibrium cell which the temperature of both mixing vessel and equilibrium cell were controlled by thermostat water bath. After pressurizing CO_2 into the mixing vessel and injecting the sample into the equilibrium cells, wait for the pressure and the temperature to be stable. Once the system was stable, the CO_2 was transferred from the mixing vessel into the equilibrium cell and the stirrer was turned on. The sample was continuously stirred to enhance the contact between the gas and liquid mixture. In equilibrium cell CO_2 are dissolved in the liquid. Thus, the pressure inside the equilibrium cell started to drop. The pressure drop in the equilibrium cell was recorded in the computerized system.

2.4. Calculation of CO_2 Solubility

Once the pressure in the equilibrium cell become constant, then the equilibrium was achieved and the values were recorded. The solubility of CO_2 in the mixture can be calculated using the following equations: Firstly, the moles of CO_2 transferred into the equilibrium cell were calculated using pressure drop in mixing vessel, volume of the mixing vessel and the temperature by the following equation:

$$n_{\text{CO}_2} = \frac{V_T}{RT_a} \left(\frac{P_1}{z_1} - \frac{P_2}{z_2} \right) \quad (1)$$

Where n_{CO_2} (mol) is the moles of CO_2 transferred into the equilibrium cell, V_T (L) is the volume of gas container (mixing vessel), R ($\text{L}\cdot\text{bar}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$) is the real gas constant, T_a (K) is the ambient temperature, P_1 and P_2 (bar) are the pressure before and after transferring, z_1 and z_2 are the compressibility factors for each pressure (P_1 and P_2) which were calculated using Peng Robinson equation of state (EOS).

Secondly, the remaining moles of CO_2 in the gas phase in the equilibrium cell were calculated by the following equation:

$$n_{\text{CO}_2}^g = \frac{V_g P_{\text{CO}_2}}{z_{\text{CO}_2} RT} \quad (2)$$

Where $n_{\text{CO}_2}^g$ (mol) is the remaining moles of CO_2 in the gas phase, V_g (L) is the volume of gas in the equilibrium cell, P_{CO_2} (bar) is the equilibrium pressure, z_{CO_2} is the compressibility factor for P_{CO_2} , R ($\text{L}\cdot\text{bar}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$) is the real gas constant and T (K) is the operating temperature. In this case the vapor pressure of solvents are assumed as negligible, therefore the total pressure in the equilibrium cell is considered to be equal to that of pure CO_2 gas.

Next, the moles of CO_2 that absorbed in the liquid phase were calculated by the following equation:

$$n_{CO_2}^i = n_{CO_2} - n_{CO_2}^g \quad (3)$$

Where $n_{CO_2}^i$ (mol) is the moles of CO₂ in the liquid phase, n_{CO_2} (mol) is the moles of CO₂ transferred into the equilibrium cell and $n_{CO_2}^g$ (mol) is the remaining moles of CO₂ in the gas phase that were previously calculated.

Lastly, the CO₂ solubility in the solvent was then calculated in terms of mole of CO₂ per mass of the solvent by the following equation:

$$m_{CO_2} = \frac{n_{CO_2}^i}{\rho_{solvent} V_{solvent}} \quad (4)$$

Where m_{CO_2} (mol·kg⁻¹) is the CO₂ solubility in the solvent, $n_{CO_2}^i$ (mol) is the moles of CO₂ in the liquid phase, $\rho_{solvent}$ (g·cm⁻³) is the density of the solvent and $V_{solvent}$ (L) is the volume of solvent in the equilibrium cell.

The gas solubility at equilibrium conditions can be expressed as Henry's law constant. The Henry's law constant can be estimated from the solubility data in terms of molality and it is defined as:

$$K_H(p, T, m_{CO_2}) = \lim_{m_{CO_2} \rightarrow 0} \frac{f_1(p, T, m_{CO_2})}{m_{CO_2}/m^0} \quad (5)$$

Where K_H (MPa) is the Henry's law constant, f_1 is the fugacity of the gas dissolved in the liquid phase and m_{CO_2} (mol·kg⁻¹) is the gas solubility in terms of molality and m^0 is the reference solubility equal to 1 mol·kg⁻¹ [19].

In the phase equilibrium condition, the fugacity of the gas is equal in both phases at constant pressure and temperature as the following equation:

$$f_1^i = f_1^g \quad (6)$$

Where f_1^i and f_1^g are the fugacities of the CO₂ in the liquid and gas phase respectively. Since deep eutectic solvents have negligible vapor pressure, the fugacity of the gas in the gas phase is assumed to be equal to the pure gas. The fugacity of the pure gas can be expressed as:

$$f_1^i(p, T) = p_{eq} \phi_1(p_{eq}, T_{eq}) \quad (7)$$

Where p_{eq} (bar) and T_{eq} (K) are the pressure and temperature at equilibrium and ϕ_1 is the fugacity coefficient. Thus, the estimation of Henry's law constant is simplified to be:

$$K_H \cong \frac{p_{eq} \phi_1(p_{eq}, T_{eq})}{m_{CO_2}/m^0} \quad (8)$$

The enthalpy and entropy of gas dissolution are related to Henry's law constant can be estimated from the following equation:

$$\Delta H = R \left(\frac{\delta \ln K_H}{\delta (1/T)} \right)_p \quad (9)$$

$$\Delta S = -R \left(\frac{\delta \ln K_H}{\delta \ln T} \right)_p \quad (10)$$

Where ΔH (kJ·mol⁻¹) and ΔS (J·mol⁻¹·K⁻¹) are the enthalpy and entropy of gas dissolution, K_H (MPa) is the Henry's law constant, R (J·K⁻¹·mol⁻¹) is the real gas constant and T (K) is the temperature.

3. Results and discussion

3.1. Experimental method validation

To validate the experimental methods applied in the present study, the experiments on solubility of CO₂ in ChCl + glycerol based DES at 1:2 molar ratio were measured at T = 303.15 K and several pressures. The results from this study are shown in Table 2. The data obtained from this study were compared with those published in the literature reported by Leron et al. [19] and showed a good agreement with those of the literature. Minor differences in solubility data can be observed, this might be due to

the presence of impurities in the DES samples used, sources of chemicals (DESs in this work are synthesized in-house) and operating conditions.

Table 2. Comparison of experimental solubilities of CO₂ in synthesized ChCl + glycerol based mixtures at 1:2 molar ratio at 303.15 K and different pressures (molality basis) with literature data.

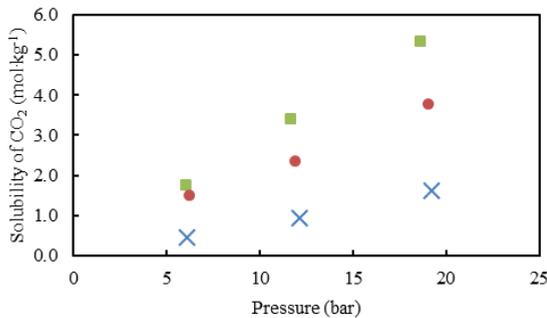
1:2:0 [19]		1:2:0	
P_{CO_2} (bar)	m_{CO_2} (mol·kg ⁻¹)	P_{CO_2} (bar)	m_{CO_2} (mol·kg ⁻¹)
6.16	0.4678	6.10	0.4625
12.22	0.9037	12.11	0.9483
19.95	1.4308	19.21	1.6083

3.2. Solubility of CO₂ in (ChCl + glycerol) + L-arginine mixtures

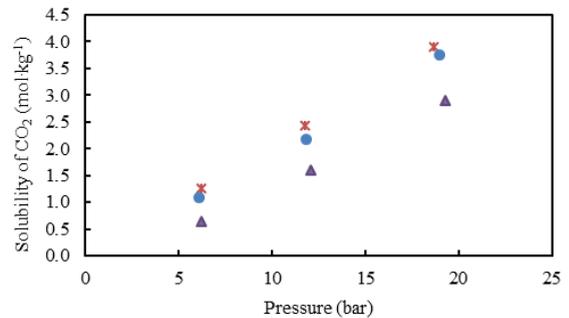
In a preliminary screening, the solubility of CO₂ in nine synthesized (ChCl + glycerol) + L-arginine mixtures were investigated at 303.15 K and pressures ranging from 6 to 20 bar and the solubility data are presented in Fig. 1. Based on the results of this screening, Fig. 2 shows the effect of glycerol on the solubility of CO₂ in ChCl + glycerol based DESs at 1:2, 1:3 and 1:4 molar ratios. Slight increase in solubility of CO₂ was observed with increasing glycerol molar ratio. Moreover, the (ChCl + glycerol) + L-arginine mixture at 1:2:0.1 molar ratio was shown the highest solubility of CO₂, for this mixture the solubilities of CO₂ were further measured for various temperatures (303.15 to 323.15 K) and pressures (6 to 70 bar). The solubility of CO₂ in the mixtures increased with increasing pressure and decreased with increasing temperature at all the pressures (Fig. 4). The results obtained in the present study follow typical trends in solubility of gases in other DESs [19,21].

3.3. Effect of L-arginine on solubility of CO₂ in ChCl + glycerol based DESs

The effect of L-arginine on solubility of CO₂ in ChCl + glycerol based DESs was observed in the present study. In Fig. 3, an increase in L-arginine molar ratio results in increase of solubility of CO₂ in the mixture. The increase of solubility of CO₂ was expected due to a high chance for CO₂ to react with one or more of these four available amine sites in L-arginine [17]. However, as more L-arginine was added (0.2 molar ratio) the solubility of CO₂ in (ChCl + glycerol) + L-arginine mixture become lesser (as compared to 0.1 molar ratio). The notable less solubility of CO₂ in the mixture could be attributed to its high viscosity.



(a)



(b)

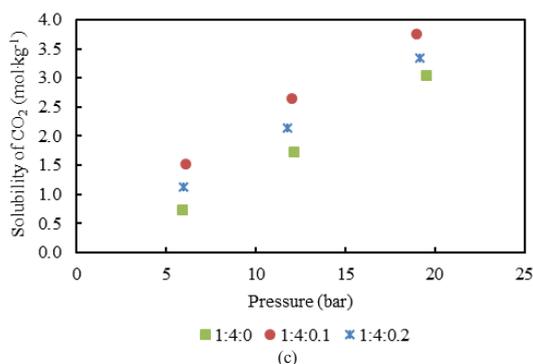


Fig. 1. Experimental solubilities of CO₂ in (ChCl + glycerol) + L-arginine mixtures at 303.15 K (a) 1:2 based mixtures (b) 1:3 based mixtures (c) 1:4 based mixtures.

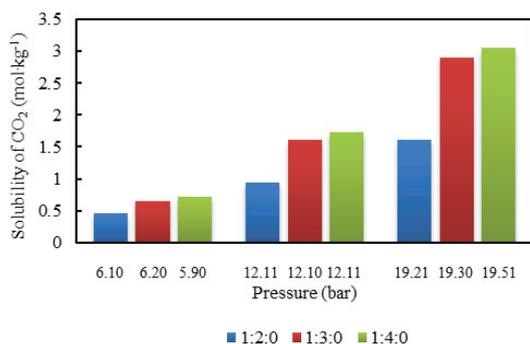


Fig. 2. Effect of glycerol molar ratio on solubility of CO₂ in ChCl + glycerol based DES at 303 K.

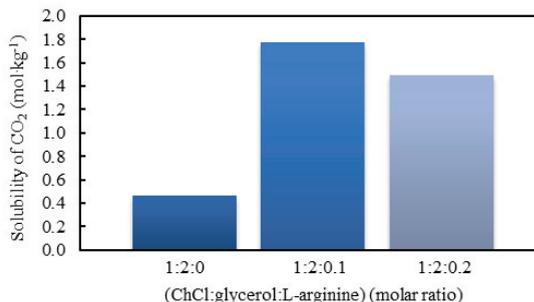


Fig. 3. Effect of L-arginine molar ratio on solubility of CO₂ in (ChCl + glycerol) + L-arginine mixture at 303.15 K and 6 bar.

3.4. Henry's law constant, enthalpy and entropy of dissolution

The Henry's law constants of CO₂ in all studied mixtures at temperature 303.15 K were presented in Table 3, and Henry's law constants of CO₂ in (ChCl + glycerol) + L-arginine mixture (1:2:0.1 molar ratio) at different temperatures were presented in Table 4 in Henry's law region (< 20 bar) where the solubility is directly proportional to the pressure [20]. It is evident that with the addition of L-arginine in (ChCl + glycerol) based DES improves the CO₂ solubility in the mixture and the values of the solubility of CO₂ in the present studied mixtures are higher than some other DESs reported in the literature [21]. Table 4 shows the negative value of ΔH indicates that the absorption of CO₂ in mixtures is exothermic and the intermolecular interaction of CO₂ with mixtures is strong. Besides, the value of ΔS is negative, meaning that a more ordered structure is achieved with the dissolution of CO₂.

Table 3. Calculated Henry's law constants of all synthesized (ChCl + glycerol) + L-arginine mixtures at 303.15 K.

Mixture	1:2:0	1:2:0.1	1:2:0.2	1:3:0	1:3:0.1	1:3:0.2	1:4:0	1:4:0.1	1:4:0.2
<i>K_H</i> (MPa)	1.1835	0.3216	0.4435	0.7461	0.4544	0.5081	0.6760	0.4237	0.5160

Table 4. Calculated Henry's law constant, enthalpy and entropy of dissolution of synthesized (ChCl + glycerol) + L-arginine mixture (1:2:0.1 molar ratio) at different temperatures and pressures (molality basis).

Mixture	303.15 K	313.15 K	323.15 K	<i>ΔH</i> (kJ.mol ⁻¹)	<i>ΔS</i> (J.mol ⁻¹ .K ⁻¹)
	<i>K_H</i> (MPa)	<i>K_H</i> (MPa)	<i>K_H</i> (MPa)		
1:2:0.1	0.3216	0.4908	0.5573	-22.52	-71.76

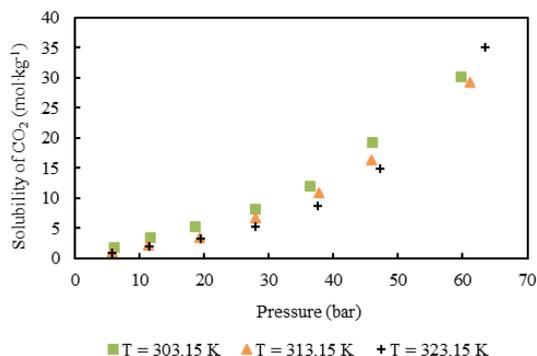


Fig. 4. Effect of pressures and temperatures on solubility of CO₂ in (ChCl + glycerol) + L-arginine mixture (1:2:0.1 molar ratio).

4. Conclusion

In the present study, the solubility of CO₂ in nine synthesized (ChCl + glycerol) + L-arginine mixtures were measured at temperature 303.15 K and pressures up to approximately 20 bar. The results showed that among all the mixtures the highest solubility of CO₂ was found in the mixture composed of (ChCl + glycerol) + L-arginine with the molar ratio of 1:2:0.1. The solubility of CO₂ in (ChCl + glycerol) + L-arginine mixture (1:2:0.1 molar ratio) was further measured at temperatures ranging from 303.15 to 323.15 K and pressures up to approximately 70 bar. The results indicated that the solubility of CO₂ in all synthesized mixtures increased with increasing pressure and decreasing temperature. The L-arginine showed pronounced effect on the solubility of CO₂ and the amount of CO₂ solubility was enhanced by the addition of L-arginine in ChCl + glycerol based DES. Moreover, Henry's law constants were calculated from the experimental data. Simultaneously, thermodynamic properties namely enthalpy and entropy of dissolution were also determined.

Acknowledgements

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