

Solubility of Carbon Dioxide in an Aqueous Blend of Diethanolamine and Piperazine[†]

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The solubility of CO₂ in aqueous blends of diethanolamine (DEA) and piperazine (PZ), from mixtures of CO₂ and N₂, was measured for temperatures and CO₂ partial pressures ranging from (303.14 to 353.14) K and (10.133 to 20.265) kPa, respectively. Measurements were made by a saturation method using a laboratory scale bubble column. The results of CO₂ solubility in liquid are expressed as α_{CO_2} (mol CO₂/mol amine) for all experimental runs. A solubility model is developed to correlate and predict the solubility data of CO₂ in aqueous blends of DEA and PZ. There is an acceptable degree of agreement between the experimental data of the present study and predictions of the solubility model with an average absolute deviation of less than 4.5 %.

Introduction

Environmental issues due to emissions of pollutants from combustion of fossil fuels have become global problems, including air toxics and greenhouse gases (GHG). Among these GHG, CO₂ is the largest contributor in regard of its amount present in the atmosphere contributing to 60 % of global warming effects.¹ Therefore, CO₂ needs to be separated and captured from the flue gases of such point sources before direct sequestration.

The removal of acid gas impurities (in particular, carbon dioxide and hydrogen sulfide) from a gas stream is a significant operation in gas processing.² Due to their active nature, acidic gases may be absorbed from a gas stream by a number of different chemical or physical absorbents. When both CO₂ and hydrogen sulfide are present in a gas stream, the most commonly used absorbents are aqueous solutions of a single or mixed alkanolamine. The monoethanolamine (MEA) absorption process is the most extensively studied CO₂ absorption system.^{3–5} The MEA process disadvantages include: (1) low CO₂ loading capacity; (2) high equipment corrosion rate; (3) amine degradation by SO₂, NO₂, HCl, HF, and oxygen in flue gas which include a high absorbent makeup rate; (4) high energy consumption during high temperature absorbent regeneration.^{6,7}

Besides MEA, diethanolamine (DEA) and methyldiethanolamine (MDEA) are also used as absorbents. Primary and secondary amines react rapidly with CO₂ to form carbamate with a stoichiometric loading of 0.5 mol CO₂/mol amine. For the tertiary amine MDEA, the CO₂ loading approaches a value of 1.0 mol CO₂/mol amine due to their not forming carbamates. Mixed amines can bring about considerable improvement in gas absorption and great savings in energy requirement for regeneration. These are reports of a substantial reduction in regeneration cost and modest reduction in circulation rates for amine blends relative to the corresponding single amine system of similar total amine concentration.⁸

Table 1. Experimental Conditions Used in the Present Work

CO ₂ partial pressure in inlet gas stream (p_{CO_2})/kPa	10.133 to 20.265
temperature of the liquid bed (T)/K	303.14 to 353.14
volume of the liquid/cm ³	500
height of the liquid bed/cm	28.2
pH of the distilled water used	6.95
mole ratio of PZ in total amine (X_1)	0.01 to 0.20
total amine concentration (C_T)/mol·dm ⁻³	1.0 to 4.0

Table 2. Temperature Dependence of the Equilibrium Constant and Henry's Constant^a

parameter	expression	references
He	$\ln \text{He} = a_1 + a_2/(T/K)$	Abu-Arabi et al. ¹⁶
K_1	$\ln K_1 = 13.3373/(T/K) + 4218.71 \ln(T/K) - 0.0099$	Austgen et al. ⁹
K_6	$\ln K_6 = -29.31 + 5616/(T/K)$	Bishnoi and Rochelle ¹⁷
K_7	$\ln K_7 = 11.53 + 4345.5/(T/K)$	Pagano et al. ¹⁸
K_9	$\ln K_9 = 140.932 - 13445.9/(T/K) - 22.4773 \ln(T/K)$	Edwards et al. ¹⁹
K_{10}	$\ln K_{10} = 220.067 - 12431.71/(T/K) - 35.4819 \ln(T/K)$	Edwards et al. ¹⁹

^a He is in kPa·m³·kmol⁻¹. K is based on molarity scale.

The low vapor pressure of DEA makes it suitable for low pressure operations, as vaporization losses are quite negligible. DEA solutions are, in general, less corrosive than MEA solutions. In view of this, DEA-based blends appear to be potential solvents for gas treating. Austgen et al.⁹ reported the CO₂ solubility in (2.0 kmol·m⁻³ MDEA + 2.0 kmol·m⁻³ DEA or MEA) aqueous solution at (313 and 353) K for CO₂ partial pressure below 315 kPa. Li and Shen¹⁰ expressed the chemical equilibrium constants for (MEA + MDEA) aqueous solution as a function of temperature, amine concentration, and CO₂ solubility on the basis of the model of Kent-Eisenberg.¹¹ Liu et al.¹² measured the CO₂ solubility data in an aqueous mixture of MDEA and PZ at CO₂ partial pressures from (13.16 to 953.3) kPa. Bishnoi and Rochelle¹³ showed that PZ has a large effect on solubility when the ratio of total CO₂ to PZ is less than unity. PZ is an effective component for the industrial CO₂ removal process; however, the solubility of CO₂ in aqueous blends of DEA with PZ is very scarce in the literature.

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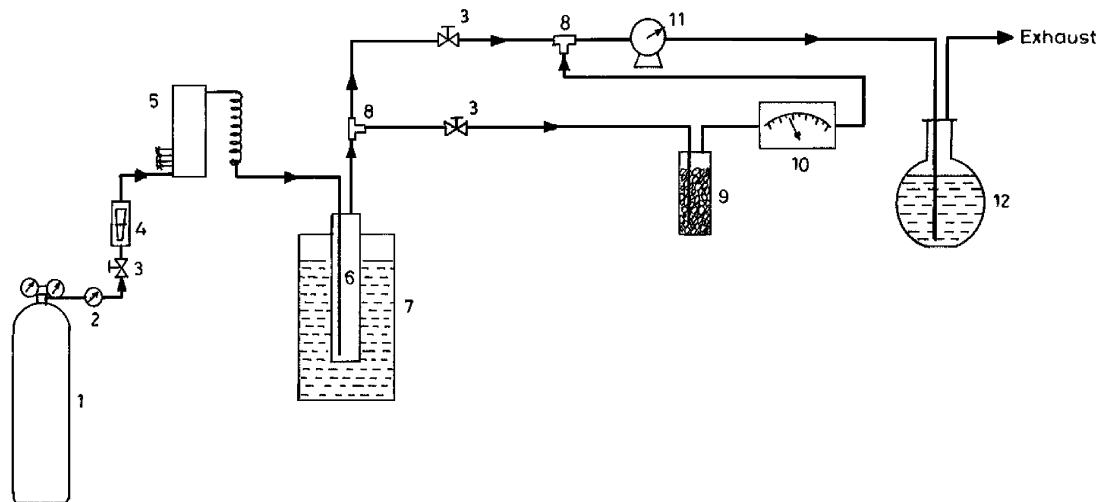


Figure 1. Experimental set-up for bubble column: 1, Gas cylinder (CO₂ and N₂); 2, Two-stage S.S. pressure regulator; 3, S.S. valve; 4, Gas rotameter; 5, Gas mixing and pressure release chamber; 6, Bubble column; 7, Constant temperature water bath; 8, Glass tee; 9, Moisture trap column; 10, CO₂ analyzer; 11, Wet gas flow meter; 12, Flat bottom flask with absorbing solution.

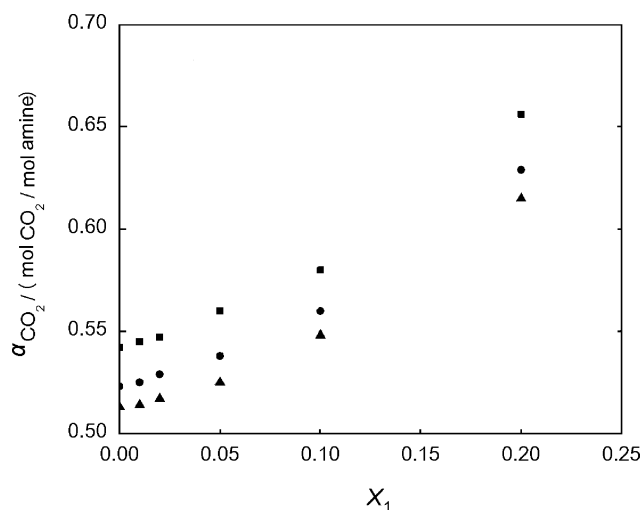


Figure 2. CO₂ loading α_{CO_2} with different mole ratios of PZ in total amine X_1 at $T = 313.14$ K and $p_{\text{CO}_2} = 15.199$ kPa for total concentration of amine blend C_T of ■, 2.0 mol·dm⁻³; ●, 2.5 mol·dm⁻³; ▲, 3.0 mol·dm⁻³.

The present work has been undertaken with an objective to study coal fired thermal power plant emitted CO₂ solubility (loading) in an aqueous blend of (DEA + PZ). For coal fired thermal power plant stack gas in India, CO₂ gas composition exhibits less than 20 % by volume at atmospheric pressure. The paper presents the equilibrium solubility of CO₂ in aqueous solutions of the (DEA + PZ) blend and also a comparison of the data of the present study with the data obtained from the developed rigorous thermodynamic model.

Experimental Apparatus and Procedure

The previous experimental setup with little modification¹⁴ used to determine the experimental data for this work has been shown in Figure 1. Experiments were carried out at a total pressure of 101.3 kPa using CO₂ and N₂ mixtures in the CO₂ partial pressure up to 20.265 kPa. The solubility of CO₂ was determined by a saturation method where the gaseous mixture was bubbled through the liquid phase. The dimensions of various main units including material of construction were reported elsewhere,¹⁵ and the experimental conditions used in the present work are shown in Table 1.

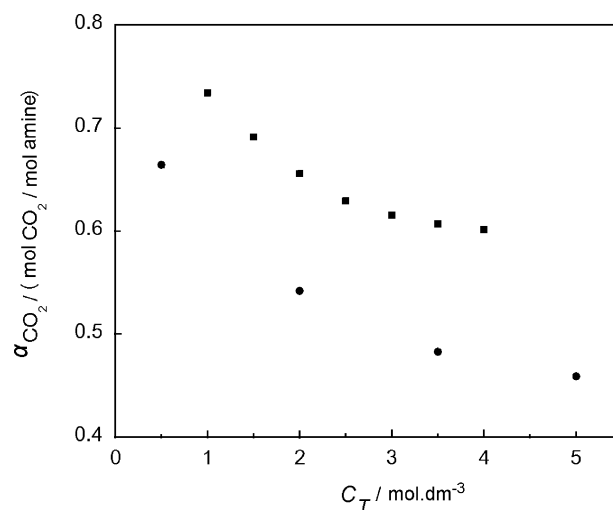


Figure 3. Effect of total concentration of amine blend C_T on CO₂ loading α_{CO_2} at $T = 313.14$ K, $p_{\text{CO}_2} = 15.199$ kPa, and mole ratio of PZ in total amine $X_1 = 0.2$ of ■, (DEA + PZ) blend, present study; ●, aq DEA, Lee et al.²⁰

The bubble column containing an aqueous blend of (DEA + PZ) was placed into a constant temperature water bath. Thermometers with subdivision of ± 0.1 K were used to monitor both the bath and the liquid temperature. Before starting the experiment, the bubble column of about 500 cm³ capacity was filled with an aqueous blend of (DEA + PZ) solution. When a constant temperature was attained inside the bath, then the main gas stream is slowly turned on and maintained the minimum possible gas flow rate so that the CO₂ and N₂ mixture was bubbled through the liquid. After a regular time interval of 10 min, the CO₂ composition in both inlet and outlet gas streams was measured with the help of a microprocessor-based CO₂ analyzer (UNIPHS 225 p.m., (0 to 100) % CO₂ by volume) with an accuracy of ± 0.2 %. This procedure is continued until the outlet gas composition becomes equal to the inlet gas composition. Once equilibrium is reached, the equilibrium CO₂ loading in the liquid phase was estimated by acidulating a known volume of the loaded liquid sample with 0.6 mol % of HCl solution and measuring the volume of the evolved gas. At a given temperature and pressure, at least two liquid equilibrium samples were taken to check the reproducibility, and the estimated error in the

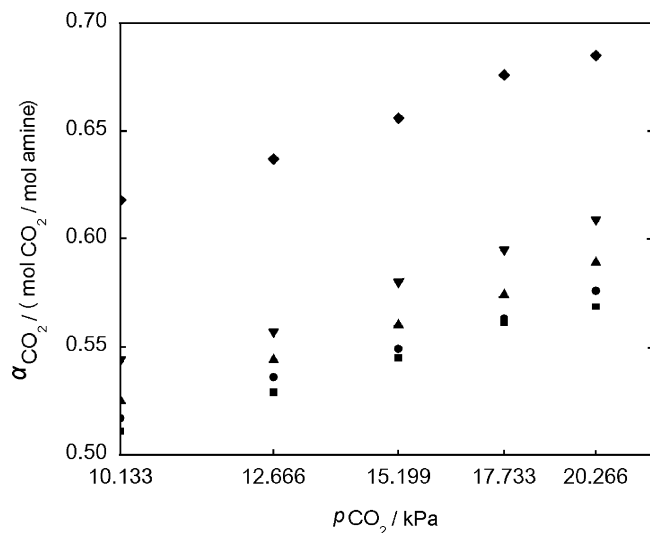


Figure 4. Effect of CO₂ partial pressure in inlet gas stream p_{CO_2} on CO₂ loading α_{CO_2} with mole ratio of PZ in total amine X_1 of ■, 0.01; ●, 0.02; ▲, 0.05; ▼, 0.10; ◆, 0.20 in (DEA + PZ) blend at $T = 313.14$ K and total concentration of amine blend $C_T = 2.0$ mol·dm⁻³.

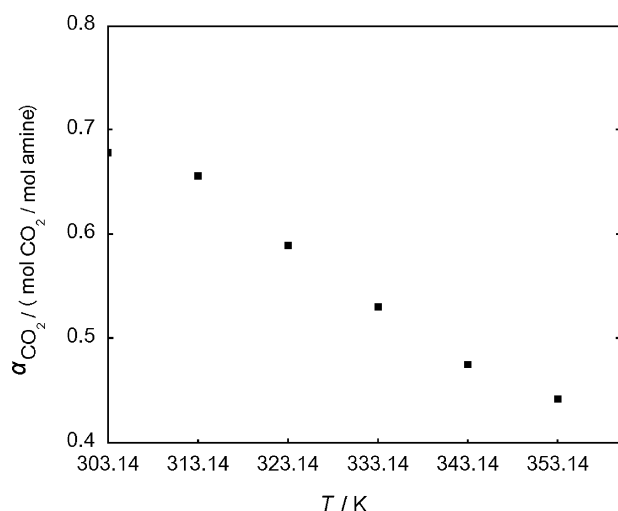


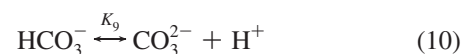
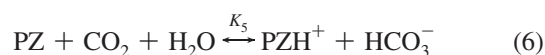
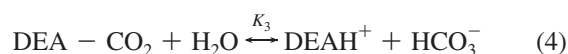
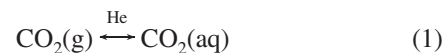
Figure 5. Effect of temperature T on CO₂ loading α_{CO_2} in (DEA + PZ) blend at $p_{\text{CO}_2} = 15.199$ kPa, total concentration of amine blend $C_T = 2.0$ mol·dm⁻³, and mole ratio of PZ in total amine $X_1 = 0.2$.

measured solubility (with respect to CO₂ loading) is about ± 0.5 %. The temperature of the liquid inside the bubble column was controlled within ± 0.1 K up to 353.15 K. The total pressure was measured for each run with an uncertainty of ± 0.5 kPa up to 20.265 kPa.

The solutions were prepared from double-distilled water. Reagent grade DEA was obtained from Sisco Research Laboratory Pvt. Ltd., Mumbai, with a purity of 98 %. Piperazine (PZ) was obtained from SD Fine Chem. Ltd., Mumbai, with a purity of better than 99 % and was used without further purification.

CO₂ Solubility Model

The chemical reaction equilibria and gas–liquid phase equilibria for the (CO₂ + DEA + PZ + H₂O) system are coexistent. When CO₂ is absorbed into an aqueous solution of PZ mixed with DEA, the following reaction may occur



By neglecting reactions 3 and 5 in the solution, the thermodynamic equilibrium (or dissociation) constant relations are

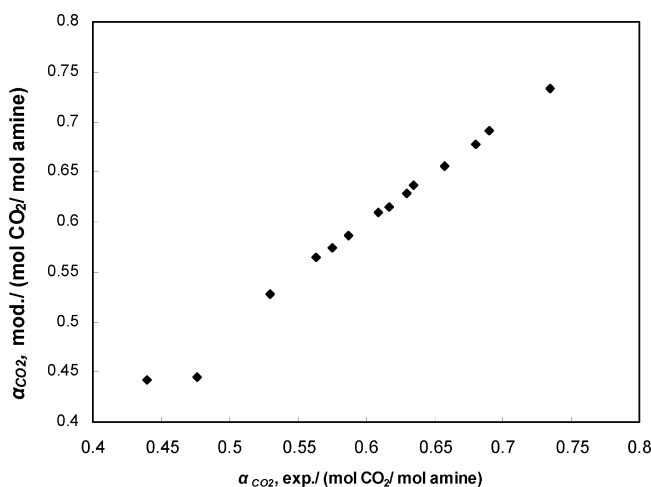


Figure 6. Comparison of the experimental solubility of CO₂ and model values.

$$p_{\text{CO}_2} = \text{He}C_{\text{CO}_2} \quad (12)$$

$$K_1 = C_{\text{DEAH}^+}/C_{\text{DEA}}C_{\text{H}^+} \quad (13)$$

$$K_3 = C_{\text{DEAH}^+}C_{\text{HCO}_3^-}/C_{\text{DEA-CO}_2}C_{\text{H}_2\text{O}} \quad (14)$$

$$K_5 = C_{\text{PZH}^+}C_{\text{HCO}_3^-}/C_{\text{PZ-CO}_2}C_{\text{H}_2\text{O}} \quad (15)$$

$$K_6 = C_{\text{PZCOO}^-}C_{\text{H}^+}/C_{\text{PZ}}C_{\text{CO}_2} \quad (16)$$

$$K_7 = C_{\text{PZH}^+}/C_{\text{PZ}}C_{\text{H}^+} \quad (17)$$

$$K_8 = C_{\text{DEA-CO}_2}C_{\text{PZ}}/C_{\text{PZ-CO}_2}C_{\text{DEA}} \quad (18)$$

$$K_9 = C_{\text{CO}_3^{2-}}C_{\text{H}^+}/C_{\text{HCO}_3^-} \quad (19)$$

$$K_{10} = C_{\text{H}^+}C_{\text{OH}^-}/C_{\text{H}_2\text{O}} \quad (20)$$

Where C stands for amount concentration of ions and molecular species. K_1 to K_{10} and He are concentration equilibrium constants and Henry's constant. In addition, the following mass and charge balances governing the reacting species can be formed

$$C_{\text{DEA}}^0 = C_{\text{DEA}} + C_{\text{DEAH}^+} \quad (21)$$

$$C_{\text{PZ}}^0 = C_{\text{PZ}} + C_{\text{PZH}^+} + C_{\text{PZCOO}^-} \quad (22)$$

$$C_{\text{HCO}_3^-} + C_{\text{CO}_3^{2-}} + C_{\text{CO}_2} + C_{\text{PZCOO}^-} = \alpha_{\text{CO}_2}(C_{\text{DEA}}^0 + C_{\text{PZ}}^0) \quad (23)$$

$$C_{\text{DEAH}^+} + C_{\text{PZH}^+} + C_{\text{H}^+} = C_{\text{OH}^-} + C_{\text{HCO}_3^-} + 2C_{\text{CO}_3^{2-}} + C_{\text{PZCOO}^-} \quad (24)$$

Where C^0 and α_{CO_2} are the initial concentration of molecular species and solubility of CO_2 in the liquid phase, respectively. Defining α_{D} as the ratio of DEAH^+ to DEA , α_{P_1} as that of PZCOO^- to PZ , and α_{P_2} as that of PZH^+ to PZ in the solution

$$\alpha_{\text{D}} = C_{\text{DEAH}^+}^+/C_{\text{DEA}}^0 \quad (25)$$

$$\alpha_{\text{P}_1} = C_{\text{PZCOO}^-}/C_{\text{PZ}}^0 \quad (26)$$

$$\alpha_{\text{P}_2} = C_{\text{PZH}^+}/C_{\text{PZ}}^0 \quad (27)$$

From eqs 16 and 17, a relationship between PZCOO^- and PZH^+ is obtained

$$C_{\text{CO}_2} = \alpha_{\text{P}_1}\alpha_{\text{P}_2}/((1 - \alpha_{\text{P}_1} - \alpha_{\text{P}_2})^2K_6K_7) \quad (28)$$

From eqs 14, 15, and 18, the distribution between DEA and PZ is given by

$$K_1/K_7 = (K_3K_8/K_5) = ((1 - \alpha_{\text{P}_1} - \alpha_{\text{P}_2})\alpha_{\text{D}})/(\alpha_{\text{P}_2}(1 - \alpha_{\text{D}})) \quad (29)$$

Abu-Arabi et al.¹⁶ measured the physical solubility and diffusivity of CO_2 in aqueous (0 to 30) % DEA solutions for temperatures between (298 and 333) K. In this work, because the content of the added PZ is low, the Henry's constant correlation given by Abu-Arabi et al. has been modified and is extended to the ($\text{DEA} + \text{PZ} + \text{H}_2\text{O}$) system. The equilibrium constants needed for calculation are listed in Table 2. With the help of equilibrium constants, He , C_{DEA}^0 , C_{PZ}^0 , T , and p_{CO_2} , the values of C_{CO_2} , $C_{\text{HCO}_3^-}$, $C_{\text{CO}_3^{2-}}$, C_{H^+} , C_{OH^-} , α_{D} , α_{P_1} , and α_{P_2} , C_{PZCOO^-} can be calculated. Thereby, finally by using eq 23, the values of α_{CO_2} can be obtained.

Results and Discussion

The solubility of CO_2 in an aqueous blend of DEA and PZ was measured at a molar ratio of piperazine in total amines between (0.01 and 0.20) for total amine concentration between (2 and 3) $\text{mol}\cdot\text{dm}^{-3}$ at temperature 313.14 K and 15.199 kPa CO_2 partial pressure as shown in Figure 2. For studying the effect of total amine concentration, amine concentration was varied from (1 to 4) $\text{mol}\cdot\text{dm}^{-3}$ and given in Figure 3. The effect of partial pressure of carbon dioxide (p_{CO_2}) was studied by varying p_{CO_2} between about (10.133 and 20.265) kPa and shown in Figure 4. Figure 5 shows the effect of temperature where temperature was between about (303.14 and 353.14) K.

The experimental results are listed in Table 3. With increasing molar ratio of piperazine in total amine blend, the CO_2 solubility increases at a particular total concentration of amine blend. From Figure 3 it is clear that CO_2 solubility in amine blend exhibits a decreasing nature with total amine concentration of blend. The effect of total amine concentration is less pronounced at a high concentration range. A comparison of CO_2 solubility in aqueous DEA solution is also shown at complete concentration range with data of Lee et al.²⁰ It is seen from Figure 4 that solubility of CO_2 increases as the partial pressure of CO_2 in the inlet gas stream is increased at a particular temperature. The effect of partial pressure of CO_2 in the inlet gas stream is almost the same at all molar ratios of piperazine in total amine blend. From Figure 5, as the temperature increases, the CO_2 solubility decreases at a particular total amine concentration.

Piperazine in aqueous solution dissociates according to the following reactions



Therefore, piperazine in an aqueous solution exists not only as the molecular PZ but also as ionic forms like PZH^+ and PZH_2^{2+} . The dissociation constants for piperazine are obtained from the Handbook of Chemistry and Physics²¹ at 296 K. The concentrations of these three forms depend strongly on the hydrogen ion concentration. The pH of a carbonated alkanolamine solution of commercial application is in the range of (8 to 10). Simple analysis indicates that the ratio of $\text{PZH}^+/\text{PZH}_2^{2+}$ is the range from 275 to 27 000 at 296 K. It means that the second-order dissociation reaction for piperazine in the solution is almost negligible. Therefore, the values of equilibrium constants together with first-order dissociation constant of piperazine were used for fitting the model to the measured equilibrium data. Eqs 12 to 29 were solved by Newton's method.

Table 3. Experimental Solubility Data of CO₂ in Aqueous Solution of DEA (2) + PZ (1)

run number	T/K	X ₁	C _T /mol·dm ⁻³	p _{CO₂} /kPa	α _{CO₂} (mol CO ₂ /mol amine)
1.	313	0.01	2.0	15.199	0.545
2.	313	0.02	2.0	15.199	0.549
3.	313	0.05	2.0	15.199	0.560
4.	313	0.10	2.0	15.199	0.580
5.	313	0.20	2.0	15.199	0.656
6.	313	0.01	2.5	15.199	0.525
7.	313	0.02	2.5	15.199	0.529
8.	313	0.05	2.5	15.199	0.538
9.	313	0.10	2.5	15.199	0.560
10.	313	0.20	2.5	15.199	0.629
11.	313	0.01	3.0	15.199	0.514
12.	313	0.02	3.0	15.199	0.517
13.	313	0.05	3.0	15.199	0.526
14.	313	0.10	3.0	15.199	0.548
15.	313	0.20	3.0	15.199	0.615
16.	313	0.20	1.0	15.199	0.734
17.	313	0.20	1.5	15.199	0.691
18.	313	0.20	3.5	15.199	0.607
19.	313	0.20	4.0	15.199	0.601
20.	313	0.01	2.0	10.133	0.511
21.	313	0.02	2.0	10.133	0.517
22.	313	0.05	2.0	10.133	0.524
23.	313	0.10	2.0	10.133	0.543
24.	313	0.20	2.0	10.133	0.618
25.	313	0.01	2.0	12.666	0.529
26.	313	0.02	2.0	12.666	0.536
27.	313	0.05	2.0	12.666	0.544
28.	313	0.10	2.0	12.666	0.557
29.	313	0.20	2.0	12.666	0.637
30.	313	0.01	2.0	17.732	0.561
31.	313	0.02	2.0	17.732	0.563
32.	313	0.25	2.0	17.732	0.574
33.	313	0.10	2.0	17.732	0.595
34.	313	0.20	2.0	17.732	0.676
35.	313	0.01	2.0	20.265	0.569
36.	313	0.02	2.0	20.265	0.576
37.	313	0.25	2.0	20.265	0.589
38.	313	0.10	2.0	20.265	0.609
39.	313	0.20	2.0	20.265	0.685
40.	303	0.20	2.0	15.199	0.678
41.	323	0.20	2.0	15.199	0.587
42.	333	0.20	2.0	15.199	0.530
43.	343	0.20	2.0	15.199	0.475
44.	353	0.20	2.0	15.199	0.442

There is an acceptable degree of agreement between the experimental solubility data of the present work and calculated values from the model as depicted in Figure 6. The average absolute deviations for all data points were less than 4.5 %.

Conclusions

In this work, the solubility of CO₂ has been determined in aqueous solutions of (DEA + PZ) for molar ratio of PZ from 0.01 to 0.20 in total amines, temperature range between (303.14 and 353.14) K, partial pressure of CO₂ from (10.133 to 20.265) kPa, and total amine concentration range of (1 to 4) mol·dm⁻³. The solubility of CO₂ decreases with an increase in temperature and increases with an increase in the partial pressure of CO₂ at the given temperature and at a given total amine concentration studied. A solubility model has also been developed to correlate and predict the solubility data of CO₂ in aqueous solutions of (DEA + PZ). The model-predicted data over aqueous blends of (DEA + PZ) have been found to be in good agreement with the experimental results of the present work over the range of temperatures and CO₂ partial pressures and molar ratio of PZ in total amines studied.

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