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Physicochemical Properties of Aqueous Solutions of 2-(1-Piperazinyl)-ethylamine

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The density and viscosity of aqueous solutions of 2-(1-piperazinyl)-ethylamine (PZEA) as well as the Henry's law constants and diffusivity of N_2O in these solutions were measured at temperatures in the range (298 to 323) K at atmospheric pressure of 0.1 MPa. The mass fractions of PZEA used were (0.0108 to 0.158). The "N_2O analogy" was used to estimate the Henry's law constants and diffusivity of CO_2 in aqueous PZEA solutions. The experimental density, viscosity, Henry's law constants, and diffusivity data are correlated as a function of temperature and concentration of amine.

Introduction

In recent years, there has been growing interest in using improved and activated alkanolamine solvents, e.g., piperazine (PZ) activated *N*-methyldiethanolamine (MDEA) or 2-amino-2-methyl-1-propanol (AMP), for CO_2 removal to take advantage of the substantially high rate of reaction of CO_2 with the activating agent combined with the advantages of high loading capacity of MDEA or AMP and relatively low cost of regeneration of the activated solvent.[1–3] Recently, we have proposed a new such activator, 2-(1-piperazinyl)-ethylamine (PZEA), which has potential to act as an activator due to its very high reaction rate.[4,5] Knowledge of the physicochemical properties of the alkanolamines, for example, density, viscosity, and Henry's law constant, and diffusivity of CO_2 in these solutions is essential for the process design of gas treating units and the design of the gas treating equipments. This is also important for the mass transfer rate modeling of absorption and regeneration because these properties influence the values of the liquid side mass transfer coefficients, k_L.

The purpose of this work is mainly to present some new correlations which have been developed based on some experimentally measured data to express the density and viscosity of aqueous solutions of PZEA as well as the Henry's law constants and diffusivity of N_2O in these solutions as a function of temperature and concentration of amine over the temperature range from (298 to 323) K at atmospheric pressure. The mass fractions of PZEA used were (0.0108 to 0.158). Some of the experimentally measured data were already reported in our earlier publication.[4] The data for one more temperature (298 K) are only measured in the present study and used along with the published data to develop the correlations. N_2O analogy is applied to estimate the Henry's law constants and diffusivity of CO_2 in $(PZEA + H_2O)$ solutions.

Because CO_2 undergoes chemical reaction with these solvents, the Henry's law constant and diffusivity of CO_2 in amine solutions cannot be calculated directly. As a result, one must use a nonreacting gas such as N_2O as a surrogate to CO_2 in estimating the Henry's law constant and diffusivity of CO_2 in these solvents. The "N_2O analogy"[6] for estimating the Henry's law constant and diffusivity of CO_2 in amine solutions is given by eqs 1 and 2, respectively, as follows.

$$k_{H,CO_2\text{-Am}} = k_{H,N_2O\text{-Am}}\left(\frac{k_{H,CO_2\text{-water}}}{k_{H,N_2O\text{-water}}}\right) \quad (1)$$

$$D_{CO_2\text{-Am}} = D_{N_2O\text{-Am}}\left(\frac{D_{CO_2\text{-water}}}{D_{N_2O\text{-water}}}\right) \quad (2)$$

where $k_{H,CO_2\text{-Am}}$ and $D_{CO_2\text{-Am}}$ are the Henry's law constant and diffusivity of N_2O in amine solutions, respectively.

Experimental Section

Material. Reagent grade PZEA (mass fraction purity > 0.95) was obtained from Merck and used without further purification. Distilled water degassed by boiling was used for making the amine solutions. The amine contents of the solutions were determined by acidimetric titration with standard HCl using an autotitrator (DL 50, Mettler Toledo). The uncertainty in the composition of the amine solutions was estimated as ± 0.01

Table 1. Density ρ and Viscosity η of Aqueous Solutions of PZEA of Mass Fraction w

T (K)	$100\ w_1$	ρ ($kg\cdot m^{-3}$)	η ($mPa\cdot s$)
298	1.08	998.0	0.92
	1.94	1000	0.95
	5.30	1002	1.10
	10.5	1006	1.38
	15.8	1010	1.80
303	1.08	996.0	0.87
	1.94	997.0	0.89
	5.30	999.0	1.00
	10.5	1003	1.25
	15.8	1009	1.66
313	1.08	992.0	0.74
	1.94	993.0	0.78
	5.30	996.0	0.85
	10.5	1000	1.04
	15.8	1005	1.32
323	1.08	988.0	0.60
	1.94	989.0	0.60
	5.30	990.0	0.69
	10.5	995.0	0.83
	15.8	1000	1.03

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Table 2. Binary Parameters (A_0, A_1, and A_2) of Equation 3 for the Excess Volume for PZEA (1) + H_2O (2)

parameters		binary pair PZEA + H_2O
$A_0/\mathrm{m^3 \cdot kmol^{-1}}$	$a/\mathrm{m^3 \cdot kmol^{-1}}$	−1210.0608
	$b/\mathrm{m^3 \cdot kmol^{-1} \cdot K^{-1}}$	7.6133615
	$c/\mathrm{m^3 \cdot kmol^{-1} \cdot K^{-2}}$	$-0.1196450 \cdot 10^{-1}$
$A_1/\mathrm{m^3 \cdot kmol^{-1}}$	$a/\mathrm{m^3 \cdot kmol^{-1}}$	−2544.2484
	$b/\mathrm{m^3 \cdot kmol^{-1} \cdot K^{-1}}$	16.008027
	$c/\mathrm{m^3 \cdot kmol^{-1} \cdot K^{-2}}$	$-0.2515630 \cdot 10^{-1}$
$A_2/\mathrm{m^3 \cdot kmol^{-1}}$	$a/\mathrm{m^3 \cdot kmol^{-1}}$	−1337.0887
	$b/\mathrm{m^3 \cdot kmol^{-1} \cdot K^{-1}}$	8.4128252
	$c/\mathrm{m^3 \cdot kmol^{-1} \cdot K^{-2}}$	$-0.1322028 \cdot 10^{-1}$

Table 3. Parameters of Equation 9 for the Viscosity of PZEA (1) + H_2O (2)

$a/\mathrm{mPa \cdot s}$	2.1867
$b/\mathrm{mPa \cdot s \cdot m^3 \cdot kmol^{-1}}$	−0.1657
$c/\mathrm{mPa \cdot s \cdot m^6 \cdot kmol^{-2}}$	0.6877
$h/\mathrm{T^{-1}}$	−1812.5

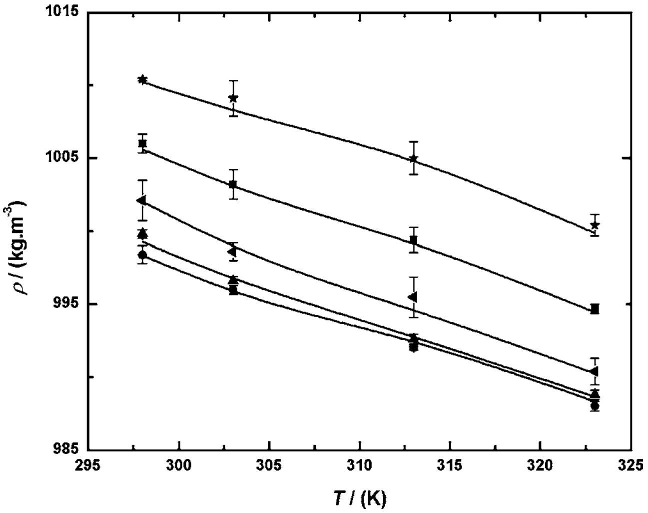

Figure 1. Density ρ of PZEA (1) + H_2O (2) solutions as a function of temperature T and mass fraction w: ●, $w_1 = 0.0108$; ▲, $w_1 = 0.0194$; solid triangle pointing left, $w_1 = 0.053$; ■, $w_1 = 0.105$; ★, $w_1 = 0.158$; —, calculated by eq 3.

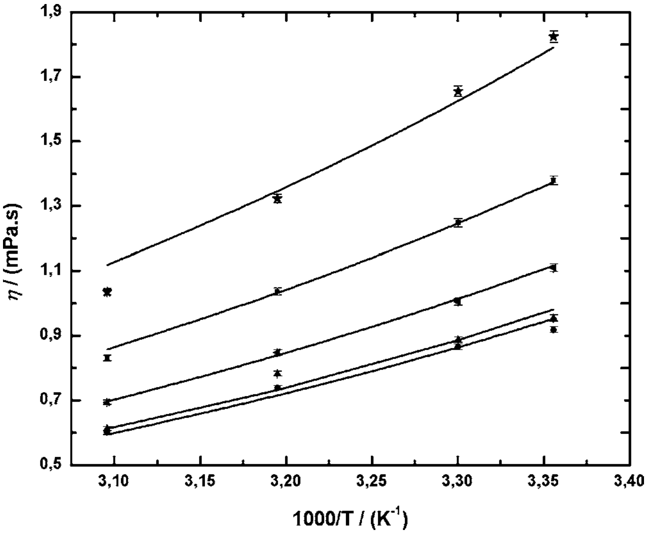

Figure 2. Viscosity η of PZEA (1) + H_2O (2) solutions as a function of temperature T and mass fraction w: ●, $w_1 = 0.0108$; ▲, $w_1 = 0.0194$; solid triangle pointing left, $w_1 = 0.053$; ■, $w_1 = 0.105$; ★, $w_1 = 0.158$; —, calculated by eq 9.

%. The nitrous oxide (mass fraction purity > 0.999) and carbon dioxide (mass fraction purity > 0.999) used for the Henry's law constant and diffusivity studies were obtained from Assam Air Products, India. The purity of CO_2 was checked by GC analysis. N_2O purity was reported as obtained from the same vendor with certified purity.

Density and Viscosity. The densities of the amine solutions were measured using a $26.76 \cdot 10^{-6}$ m^3 Gay-Lussac pycnometer. The pycnometer containing the amine solution was immersed in a constant-temperature bath. The bath temperature was controlled within ± 0.3 K of the temperature range using a circulator temperature controller (RW 2025G, Jeio Tech). Once the solution reached the desired temperature, it was weighed to within ± 0.0002 g with an analytical balance (AND GR-200). Each reported density data was the average of three measurements. The experimental uncertainty in the measured density was estimated to be ± 0.1 %.

The viscosity was measured using an Ostwald viscometer. The viscometer was immersed in a thermostatted bath. The bath temperature was controlled within ± 0.3 K of the desired level using a circulator temperature controller (RW 2025G, Jeio Tech). Each reported value was the average of three measurements. The experimental uncertainty was estimated to be ± 1 %. The validity of the procedure and apparatus for density and viscosity measurements was reported in our previous publications.[7–9]

Henry's Law Constant. The method for calculating Henry's law constants of N_2O is to measure the absorption of N_2O which is carried out by bringing a known volume of liquid into contact with gas in a closed system at constant temperature and pressure. A $6.5 \cdot 10^{-4}$ m^3 glass flask was used as the equilibrium absorption cell. A magnetic stirrer was used for the liquid phase, and two four-bladed impellers mounted on a shaft, passing through a mercury seal and driven by a dc motor, were used for the gas phase. The temperature of the equilibrium cell was controlled within ± 0.3 K of the desired level with a circulating temperature controller operated on external control mode (RW 2025G, Jeio Tech). All measurements were done at atmospheric pressure. The equilibrium cell is equipped with a precise U-tube manometric device with an adjustable limb, which can be moved up and down to maintain the atmospheric pressure throughout each experimental run. The total pressure was measured for each run within an accuracy of ± 0.2 kPa.

For each run, the equilibrium cell was allowed to reach thermal equilibrium with respect to the desired temperature for solubility measurement. The cell was then purged with the desired gas. The gas streams at the outlet of the respective flow meters were passed through water vapor saturators, maintained at the measurement temperature, before being introduced into the cell. The gas-phase stirrer was kept on at 70 rpm during purging to ensure uniform gas-phase concentration throughout the cell. After completion of purging, 10 mL of freshly prepared amine solution of desired concentration was quickly transferred into the cell, and the cell was fully sealed. The liquid-phase and gas-phase stirrers were turned on to commence absorption. The attainment of equilibrium was indicated when there was no absorption for at least 1 h, while the temperature was maintained constant at the desired level. It took about (5 to 6) h to reach equilibrium for each run. The measured volume change is equal to the volume of gas absorbed. The partial pressure of N_2O in the experiments was corrected for the vapor pressure of the solution.

The procedure of Henry's law constant calculation for CO_2 is the same as those described by Paul et al.[10] The uncertainties in the measured Henry's law constant for N_2O were estimated

Table 4. Estimated Solubility of CO_2, k_{H,CO_2}, in PZEA (1) + H_2O (2) at Temperatures T from (298 to 323) K Using the N_2O Analogy as a Function of Mass Fraction w

	k_{H,N_2O}/(kPa·m^3·kmol^{-1})				k_{H,CO_2}/(kPa·m^3·kmol^{-1})			
100 w_1	T = 298 K	T = 303 K	T = 313 K	T = 323 K	T = 298 K	T = 303 K	T = 313 K	T = 323 K
1.08	4120	4706	5957	7518	3123	3474	4295	5338
1.94	4170	4763	6027	7674	3161	3515	4345	5448
5.30	4324	4935	6221	7977	3277	3685	4485	5664
10.5	4528	5080	6444	8172	3432	3792	4646	5802
15.8	4590	5202	6531	8317	3479	3882	4709	5905

Table 5. Parameters of Equation 10 and AAD for N_2O Solubility of PZEA (1) + H_2O (2)

a/kPa·m^3·kmol^{-1}	9.8183·10^6
b/kPa·m^6·kmol^{-2}	1.7221·10^6
c/kPa·m^9·kmol^{-3}	−6.868·10^5
h/T^{-1}	2320.8
100 AAD[a]	0.5

[a] $\mathrm{AAD} = (1/N)\sum_{i=1}^{N}(|k_{\mathrm{H,exptl},i} - k_{\mathrm{H,calcd},i}|)/(k_{\mathrm{H,exptl},i})$

to be ± 1 %. The repeatability between the various experiments was within ± 1.0 %.

Diffusivity. The diffusivity of N_2O in aqueous PZEA solutions was measured using a wetted-wall column absorber with the outside diameter of $2.81 \cdot 10^{-2}$ m. The length of the absorption column was maintained at $8 \cdot 10^{-2}$ m. The gas absorption rate was measured by the volume uptake method using a soap film meter. The liquid flow rate was measured with a rotameter that was calibrated at various experimental temperatures and concentrations. The temperature of absorption was controlled within ± 0.2 K of the desired level with a circulating temperature controller (Jeio Tech, RW-2025G). It is a PID controller, and the uncertainty in the measurement of temperature was ± 0.1 K. A jacketed glass soap film storage was used to store the saturated gas at constant temperature. The liquid film and the gas-phase temperature were controlled by a circulating water thermostat. The pressure in the absorption chamber was about 100 kPa. This was measured using a Schaevitz sensor with an accuracy of 0.1 %. The experimental uncertainty in the measured diffusivity was estimated to be ± 2 %. Repeatability between the various experiments was within ± 1 %.

Results and Discussion

Density and Viscosity. The measured densities and viscosities of solutions of (PZEA + H_2O) are presented in Table 1. w_1 is the mass fraction of amine present in the solutions. The densities and viscosities of the solutions increase with mass fraction of PZEA in the solutions.

To correlate the densities of the liquid mixtures, a Redlich−Kister type equation for the excess molar volume is applied. For a binary system, the Redlich−Kister equation has the following expression[7,11]

$$V_{jk}^{\mathrm{E}}/\mathrm{m^3{\cdot}kmol^{-1}} = x_j x_k \sum_{i=0}^{n} A_i (x_j - x_k)^i \tag{3}$$

where x is the mole fraction and A_i are pair parameters and are assumed to be temperature dependent as follows

$$A_i = a + b(T/\mathrm{K}) + c(T/\mathrm{K})^2 \tag{4}$$

The excess volume of liquid mixtures for the binary system is assumed to be

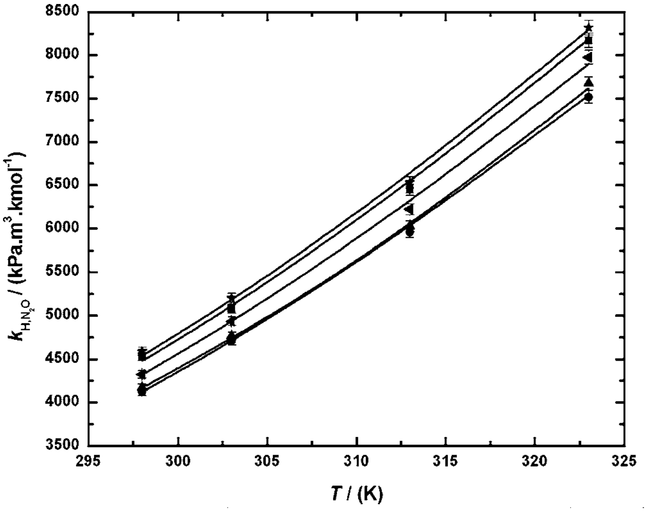

Figure 3. Henry's constant k of N_2O in PZEA (1) + H_2O (2) solutions as a function of temperature T and mass fraction w: ●, w_1 = 0.0108; ▲, w_1 = 0.0194; solid triangle pointing left, w_1 = 0.053; ■, w_1 = 0.105; ★, w_1 = 0.158; −, calculated by eq 10.

$$V^{\mathrm{E}} = V_{12}^{\mathrm{E}} \tag{5}$$

The excess volume of liquid mixtures can be calculated from the measured density of the fluids

$$V^{\mathrm{E}} = V_{\mathrm{m}} - \sum x_i V_i^0 \tag{6}$$

where V_{m} is the molar volume of the liquid mixture and V_i^0 is the molar volume of the pure fluids at the system temperature. The molar volume of the liquid mixtures is calculated by

$$V_{\mathrm{m}} = \frac{\sum x_i M_i}{\rho_{\mathrm{m}}} \tag{7}$$

where M_i is the molar mass of pure component i, ρ_{m} is the measured liquid density, and x_i is the mol fraction of the pure component i.

A general set of temperature-dependent parameters has been developed using experimental data. The determined parameters are presented in Table 2. The density of pure PZEA has been measured and correlated as a function of temperature using the following correlation

$$\rho/\mathrm{kg \cdot m^{-3}} = 1159.7 - 0.41280(T/\mathrm{K}) - 5.9431 \cdot 10^{-4}(T/\mathrm{K})^2 \tag{8}$$

The experimental viscosity data are correlated as a function of amine concentration and temperature using eq 9.[10]

$$\eta \cdot 10^3/\mathrm{mPa \cdot s} = (a + bM + cM^2)\exp(-h/(T/\mathrm{K})) \tag{9}$$

where M is the molarity of amine in the solutions. The determined parameters for viscosity are obtained by regression

Table 6. Estimated Diffusivity of CO_2, D_{CO_2}, in PZEA (1) + H_2O (2) at Temperatures T from (298 to 323) K Using the N_2O Analogy as a Function of Mass Fraction w

	$D_{N_2O}\cdot 10^9/(m^2\cdot s^{-1})$				$D_{CO_2}\cdot 10^9/(m^2\cdot s^{-1})$			
$100\ w_1$	$T = 298$ K	$T = 303$ K	$T = 313$ K	$T = 323$ K	$T = 298$ K	$T = 303$ K	$T = 313$ K	$T = 323$ K
1.08	1.60	1.80	2.30	2.87	1.72	1.94	2.72	3.36
1.94	1.57	1.79	2.26	2.80	1.68	1.90	2.65	3.32
5.30	1.47	1.70	2.19	2.75	1.57	1.80	2.57	3.22
10.5	1.38	1.60	2.08	2.60	1.47	1.70	2.44	3.08
15.8	1.30	1.54	2.00	2.57	1.40	1.63	2.38	3.01

Table 7. Parameters of Equation [11] and AAD for N_2O Diffusivity for PZEA (1) + H_2O (2)

A_0	−35.268	B_0	−110.78	C_0	1567.2
A_1/K	5321.7	B_1/K	2493.0	C_1/K	216.08
A_2/K^{-1}	0.0582	B_2/K^{-1}	0.2943	C_2/K^{-1}	−4.4217
100 AAD[a]	0.8				

[a] $\text{AAD} = (1/N)\sum_{i=1}^{N}(|D_{\text{exptl},i} - D_{\text{calcd},i}|)/(D_{\text{exptl},i})$

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analysis of the experimental data of this work and are presented in Table 3. The calculated densities and viscosities from the correlation are in excellent agreement with the experimental data, the average absolute deviation between the correlated and the experimental density and viscosity data for the (PZEA + H_2O) systems being about 0.03 % and 2 %, respectively, for 20 data points. The measured and calculated densities and viscosities from the correlations are also compared in Figures 1 and 2. These figures show different agreement for different compositions.

Solubility. The experimental method and the apparatus used for the solubility measurements are validated, and the same has been discussed in our recent publication.[10] The data for the solubility of N_2O and the estimated solubility of CO_2 using the N_2O analogy in aqueous solutions of PZEA for the temperatures (298, 303, 313, and 323) K are presented in Table 4. The calculated Henry's constant of N_2O in aqueous PZEA from the experimental measurements increases with an increase in temperature and increases with the mass percent of PZEA in the mixture.

The experimental solubility data of N_2O in (PZEA + H_2O) are correlated as a function of amine concentration and temperature according to eq 10.[10]

$$k_{H,N_2O}/\text{kPa}\cdot\text{m}^3\cdot\text{kmol}^{-1} = (a + bM + cM^2)\exp(-h/(T/\text{K})) \tag{10}$$

The calculated parameters and average absolute deviation (AAD) are listed in Table 5 for the (PZEA + H_2O) systems. The calculated solubilities from eq 10 are in good agreement with the experimental results of this study. The average absolute deviation between the correlated and experimental data for (PZEA + H_2O) is 0.5 % for 20 data points. The measured and calculated solubilities from the correlation (eq 10) are compared in Figure 3.

Diffusivity. The validity for the experimental method and the apparatus used for the diffusivity measurements has been discussed in our recent publication.[10] The data for the diffusivity of N_2O and the estimated diffusivity of CO_2 using the N_2O analogy in aqueous solutions of PZEA for the temperatures (298, 303, 313, and 323) K are presented in Table 6. Diffusivity of N_2O in aqueous PZEA increases with an increase in temperature and decrease in the mass percent of PZEA in the mixture. The experimental diffusivity data are correlated as a function of amine concentration and temperature using the following polynomial equation.

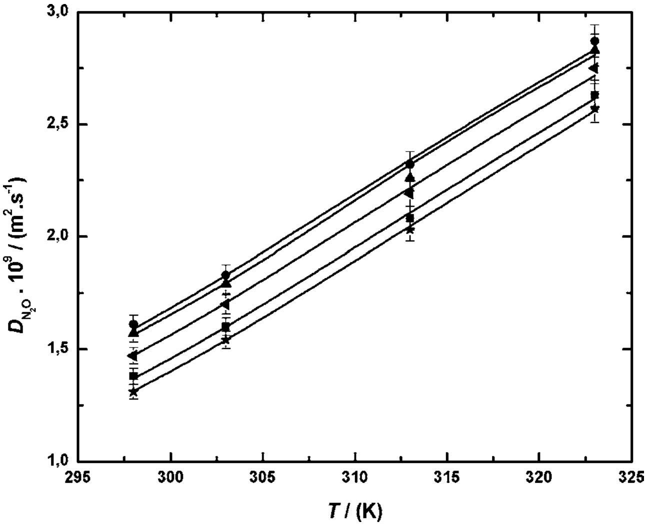

Figure 4. Diffusivity D of N_2O in PZEA (1) + H_2O (2) solutions as a function of temperature T and mass fraction w: ●, $w_1 = 0.0108$; ▲, $w_1 = 0.0194$; solid triangle pointing left, $w_1 = 0.053$; ■, $w_1 = 0.105$; ★, $w_1 = 0.158$; —, calculated by eq 11.

Table 8. Diffusivity D of N_2O in PZEA (1) + H_2O (2) and Verification of the Stokes−Einstein Relation as a Function of Temperature T and Mass Fraction w

T/K	$100\ w_1$	$D_{N_2O}\cdot 10^9$/m$^2\cdot$s^{-1}	η/mPa$\cdot$s	$(D_{N_2O}\eta)/(T)\cdot 10^{15}$/m$^2\cdot$Pa$\cdot$K^{-1}
298	1.08	1.60	0.92	5.30
	1.94	1.57	0.95	5.37
	5.30	1.47	1.10	5.85
	10.5	1.38	1.38	6.82
	15.8	1.30	1.80	8.57
303	1.08	1.80	0.87	5.54
	1.94	1.79	0.89	5.55
	5.30	1.70	1.00	5.97
	10.5	1.60	1.25	6.98
	15.8	1.54	1.66	8.91
313	1.08	2.30	0.74	6.43
	1.94	2.26	0.78	6.36
	5.30	2.19	0.85	6.96
	10.5	2.08	1.04	8.08
	15.8	2.00	1.32	10.1
323	1.08	2.87	0.60	6.26
	1.94	2.80	0.60	6.28
	5.30	2.80	0.69	6.92
	10.5	2.60	0.83	7.93
	15.8	2.57	1.03	9.65

$$\ln(D_{N_2O}/D_{N_2O-\text{water}}) = (A_0 + B_0x + C_0x^2) + ((A_1 + B_1x + C_1x^2)/(T/\text{K})) + ((A_2 + B_2x + C_2x^2)(T/\text{K})) \tag{11}$$

The calculated parameters and average absolute deviation are listed in Table 7 for the (PZEA + H_2O) systems. The calculated diffusivities from eq 11 are in good agreement with the experimental results of this study. The average absolute deviation between the correlated and experimental data for (PZEA + H_2O) is 0.8 % for 20 data points. The measured and calculated diffusivities from the correlation (eq 11) are compared in Figure 4.

The Stokes−Einstein relation ($D_{N_2O}\eta/T$ = constant) has often been used to correlate the diffusivity of N_2O in aqueous solutions of alkanolamines.[12,13] The experimentally measured η and D_{N_2O} values were used to evaluate the applicability of the Stokes−Einstein relation for the present system. The results reported in Table 8 show a wide variation between minimum and maximum constants: $5.30 \cdot 10^{-15}$ ($m^2 \cdot Pa \cdot K^{-1}$) and $10.1 \cdot 10^{-15}$ ($m^2 \cdot Pa \cdot K^{-1}$). Thus, this equation provides a poor adjustment for the present case.

Conclusions

The density and viscosity of aqueous PZEA solutions and the solubility and diffusivity of N_2O in these solutions were measured at 298 K at atmospheric pressure. The mass fractions of PZEA used were (0.0108 to 0.158). N_2O analogy was applied to estimate the physical solubility and diffusivity of CO_2 in (PZEA + H_2O) solutions. The experimental data at 298 K along with data published in our previous literature for the temperature range from (303 to 323) K were correlated as a function of temperature and amine concentrations. The calculated results from the correlations were found in good agreement with the experimental data.

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