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Physical Solubility and Diffusivity of N_2O and CO_2 in Aqueous Solutions of Piperazine and (*N*-Methyldiethanolamine + Piperazine)

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In this work, the physical solubility and diffusivity of N_2O in aqueous solutions of piperazine (PZ) and an aqueous blend of *N*-methyldiethanolamine (MDEA) and piperazine have been measured over a range of temperatures and at atmospheric pressure. For the binary mixtures, the mass percents of PZ used are (1.74 to 12) %. For the ternary mixtures, the total amine strength in the solution has been kept at 30 mass %. The "N_2O analogy" is then used to estimate the physical solubility and diffusivity of CO_2 in aqueous PZ and PZ-blended aqueous MDEA solutions. The experimental physical solubility and diffusivity data have been correlated as a function of temperature and concentration of amine and compared with the available data in the literature.

Introduction

Aqueous solutions of alkanolamines are widely used for the removal of acid gas impurities, such as CO_2 and H_2S, from natural gas and industrial gas streams by a regenerative chemical absorption process. Industrially important alkanolamines for such absorption processes are monoethanolamine (MEA), diethanolamine (DEA), di-isopropanolamine (DIPA), and *N*-methyldiethanolamine (MDEA).[1] Although MEA and DEA are widely used today for total CO_2 and H_2S removal from sour natural gas and industrial gas streams, aqueous MDEA is used very often for selective removal of H_2S from gas streams containing both CO_2 and H_2S. Recently, blended amine solvents, which consist of a mixture of a primary or secondary amine with a tertiary amine to combine the higher equilibrium capacity of the tertiary amine with the higher reaction rate of the primary or secondary amine, have been suggested for industrial gas-treating processes. The use of blended amines brings about a significant improvement in absorption capacity and absorption rate and consequently offers great savings in solvent regeneration energy requirements. More recently, there is a growing interest in the use of piperazine (PZ) activated aqueous MDEA for gas-treating processes. Being a cyclic symmetric diamine, each mole of piperazine can theoretically absorb two moles of CO_2, and it may favor rapid formation of the carbamates. The apparent second-order rate constant of PZ has been found to be an order higher than that of conventional alkanolamines such as MEA.[2] Because PZ is effective in enhancing the rate of CO_2 absorption at a much lower concentration in its blend with MDEA than the concentration of the primary and secondary amines in the conventional blended amines (MEA/MDEA or DEA/MDEA), PZ is better known as an activator or promoter in gas-treating processes.

For the analysis of the experimental absorption rate and for the rational design of the gas absorption units and equipment using alkanolamines, knowledge of the physical properties, for example, the solubility and diffusivity data of CO_2 in aqueous amine solvents and their blends at various amine concentrations and temperatures, is essential. However, because CO_2 undergoes chemical reaction with these solvents, the physical solubility and diffusivity of CO_2 in amine solutions cannot be measured directly. As a result, one must use a nonreacting gas such as N_2O as a surrogate to CO_2 in estimating the physical solubility and diffusivity of CO_2 in these solvents. The "N_2O analogy"[3] for estimating the physical solubility and diffusivity of CO_2 in amine solutions is given by eqs 1 and 2, respectively, as follows.

$$H_{CO_2-\text{amine}} = H_{N_2O-\text{amine}}\left(\frac{H_{CO_2,\text{water}}}{H_{N_2O,\text{water}}}\right) \tag{1}$$

$$D_{CO_2-\text{amine}} = D_{N_2O-\text{amine}}\left(\frac{D_{CO_2,\text{water}}}{D_{N_2O,\text{water}}}\right) \tag{2}$$

where $H_{N_2O-\text{amine}}$ and $D_{N_2O-\text{amine}}$ are the physical solubility and diffusivity of N_2O in the amine solution. This analogy has been frequently used to estimate the physical solubility of CO_2 in amine solvents.[4−9] Several properties of aqueous solutions of MDEA and its blends with primary and secondary alkanolamines have been reported previously in the literature.[5,7,10−12] However, in spite of the immense importance of these data, the PZ activated aqueous MDEA solvent system has not been thoroughly investigated so far.

Therefore, in the present work, the physical solubility and diffusivity of N_2O in aqueous PZ and PZ activated MDEA solutions at various temperatures and various relative compositions of amine blends are measured, and the N_2O analogy is then applied to estimate the solubilities of CO_2 in (PZ + water) and in (MDEA + PZ + water) solutions. The experimental results are correlated as a function of the temperature and concentration of amine.

Experimental

Reagent grade piperazine (>99 % pure) and MDEA (>98 % pure) were obtained from E. Merck, FRG, and were used without further purification. Distilled water degassed by boiling was used for preparing the amine solutions. The total amine contents of the solutions were determined by titration against standard 0.5 mol·L^{-1} HCl using a methyl orange indicator. The

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Table 1. Measured Solubility of N_2O and CO_2 in Water

T / K	$H_{N_2O-\text{water}}$ / $kPa \cdot m^3 \cdot kmol^{-1}$	$H_{CO_2-\text{water}}$ / $kPa \cdot m^3 \cdot kmol^{-1}$
293	3530	2619
298	3932	2949
303	4497	3358
308	5120	3850
313	5535	4264

nitrous oxide (>99.99 % pure) and carbon dioxide (>99.9 % pure) used for the physical solubility and diffusivity study were obtained from BOC, India, and Chemtron Science Pvt. Ltd., India, respectively.

Physical Solubility. The physical solubility measurements were carried out in a corning glass equilibrium cell, similar to one used by Saha et al.[8] The temperature of the cell and jacketed eudiometer was controlled within ± 0.1 K of the desired level with a circulator temperature controller (JULABO FP 55, FRG). Also, the experimental setup was housed inside an enclosure in which the environmental temperature could be controlled within ± 1 K of the desired level. A precise manometric device was used to maintain the atmospheric pressure in the cell throughout the experiment. All solubility measurements were carried out at atmospheric pressure.

For each run, the cell was purged with saturated N_2O or CO_2 gas when the system reached thermal equilibrium with respect to the desired temperature for solubility measurement. The gas-phase stirrer was put on at 65−70 rpm during purging to ensure proper purging and a uniform gas-phase temperature throughout the cell. When it was purged properly, the gas phase stirrer was stopped for the time being. A measured volume of 10 mL of freshly prepared aqueous amine solution of desired concentration was quickly transferred into the cell, and the cell was fully sealed. The liquid was agitated with a magnetic stirrer until there was no further change in the gas volume at least for 1 h. 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 experimental uncertainty in the measured physical solubility was estimated to be ± 2 %. The reproducibility among the various experiments was within ± 2 %.

Diffusivity. A $2.81 \cdot 10^{-2}$ m o.d. stainless steel wetted wall absorber was used to measure the diffusivity of CO_2 in water and of N_2O in water and aqueous amine solutions. The apparatus and procedure are the same as those described by Saha et al.[8] The gas−liquid contact time could be varied from 0.3 to 0.85 s by varying the absorption length but keeping the liquid flow rate constant. The rate of absorption of the gas was measured by the volume uptake method using a soap film meter. The flow rate of the solvent was measured with a rotameter that was calibrated at various experimental temperatures and concentrations of the solutions. The temperature of absorption was controlled within ± 0.1 K of the desired level with the circulator temperature controllers (FP 55 and F 32 Julabo, FRG). The pressure in the absorption chamber was about 100 kPa. The experimental uncertainty was estimated to be ± 4 %. The reproducibility among the various experiments was always within ± 2 %.

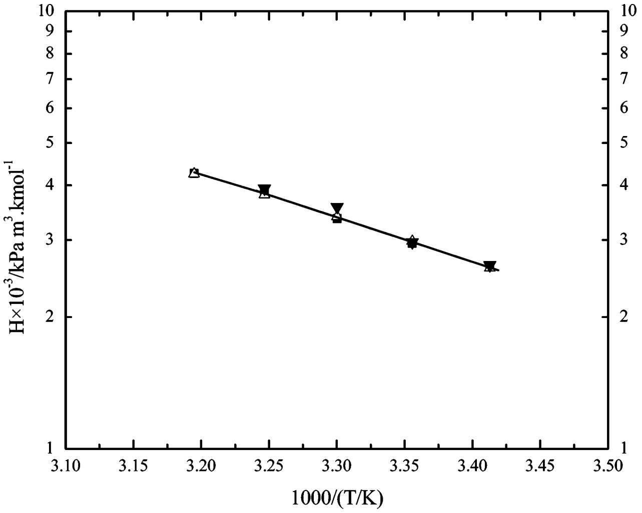

Figure 1. Henry's constant of CO_2 in water as a function of temperature: ▼, Versteeg et al.;[6] Δ, Al-Ghawas et al.;[10] ■, this study; —, calculated by eq 3.

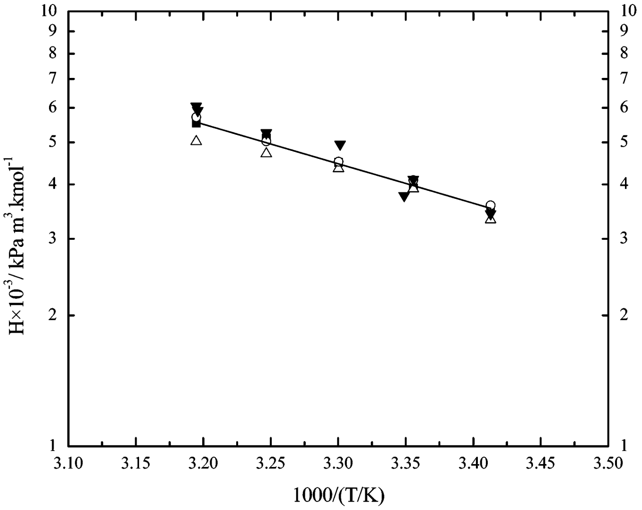

Figure 2. Henry's constant of N_2O in water as a function of temperature: ▼, Versteeg et al.;[6] Δ, Al-Ghawas et al.;[10] ○, Mandal et al.;[12] ■, this study; —, calculated by eq 4.

Table 2. Estimated Solubility of CO_2, H_{CO_2}, in PZ (2) + Water (3) Using the N_2O Analogy

	H_{N_2O}/$kPa \cdot m^3 \cdot kmol^{-1}$					H_{CO_2}/$kPa \cdot m^3 \cdot kmol^{-1}$				
100 w_2	T = 293.0 K	T = 298.0 K	T = 303.0 K	T = 308.0 K	T = 313.0 K	T = 293.0 K	T = 298.0 K	T = 303.0 K	T = 308.0 K	T = 313.0 K
1.74	3581	4094	4664	5278	5981	2657	3044	3483	4047	4607
5.16	3690	4198	4843	5565	6194	2737	3149	3616	4267	4772
12.0	3866	4423	5037	5753	6345	2868	3318	3761	4411	4888

Table 3. Estimated Solubility of CO_2, H_{CO_2}, in MDEA (1) + PZ (2) + Water (3) Using the N_2O Analogy

	H_{N_2O}/$kPa \cdot m^3 \cdot kmol^{-1}$					H_{CO_2}/$kPa \cdot m^3 \cdot kmol^{-1}$				
w_1/w_2	T = 293.0 K	T = 298.0 K	T = 303.0 K	T = 308.0 K	T = 313.0 K	T = 293.0 K	T = 298.0 K	T = 303.0 K	T = 308.0 K	T = 313.0 K
28/2	3992	4587	5140	5565	6050	2961	3441	3838	4267	4661
22/8	3866	4346	4890	5333	5781	2867	3259	3651	4090	4454
18/12	4199	4673	5302	5753	6269	3115	3505	3959	4411	4829

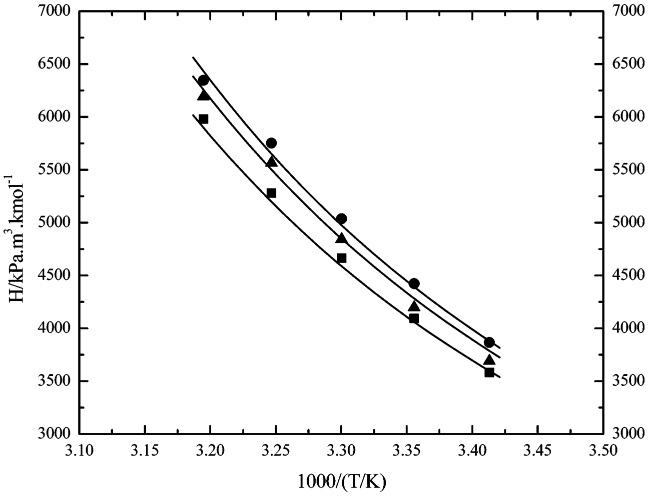

Figure 3. Henry's constant of N_2O in the PZ (2) + water (3) system at various temperatures: ■, $w_2 = 1.74$; ▲, $w_2 = 5.16$; ●, $w_2 = 12.0$; —, calculated by eq 5.

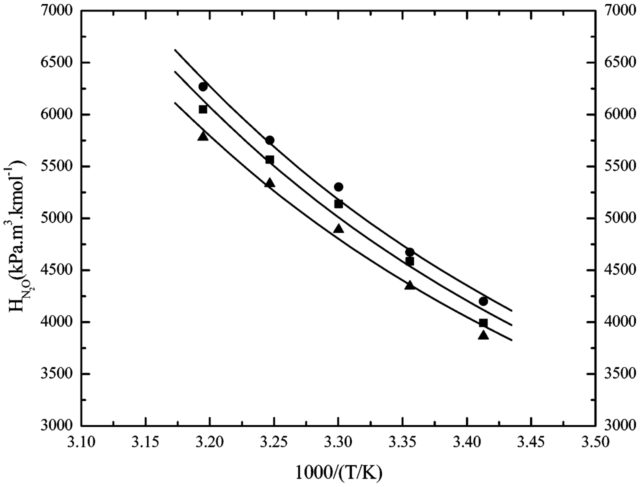

Figure 4. Henry's constant of N_2O in the MDEA (1) + PZ (2) + water (3) system at various temperatures: ■, $w_1/w_2 = 28/2$; ▲, $w_1/w_2 = 22/8$; ●, $w_1/w_2 = 18/12$; —, calculated by eq 5.

Table 4. Parameters and AADs for N_2O Solubility Correlations for the Binary and Ternary Solvent Systems

		a_i	b_i	c_i	AAD[a]/%
	$i = 1$	4.69	$-2.39 \cdot 10^{-5}$	$2.42 \cdot 10^{-4}$	
(PZ + H_2O)	$i = 2$	$1.05 \cdot 10^{-5}$	$1.89 \cdot 10^{-4}$	$7.38 \cdot 10^{-5}$	1.32
	$i = 3$	$4.05 \cdot 10^{-5}$	$1.68 \cdot 10^{-5}$	$-5.96 \cdot 10^{-5}$	
	$i = 1$	8.05	−3.85	−16.58	
(MDEA + PZ + H_2O)	$i = 2$	17.52	4.08	−10.38	1.47
	$i = 3$	0.026	−0.175	0.294	
	α		0.969		

[a] $$\mathrm{AAD} = \frac{1}{N}\sum_{i=1}^{N}\frac{|H_{\mathrm{calcd},i} - H_{\mathrm{exptl},i}|}{H_{\mathrm{exptl},i}} \cdot 100$$

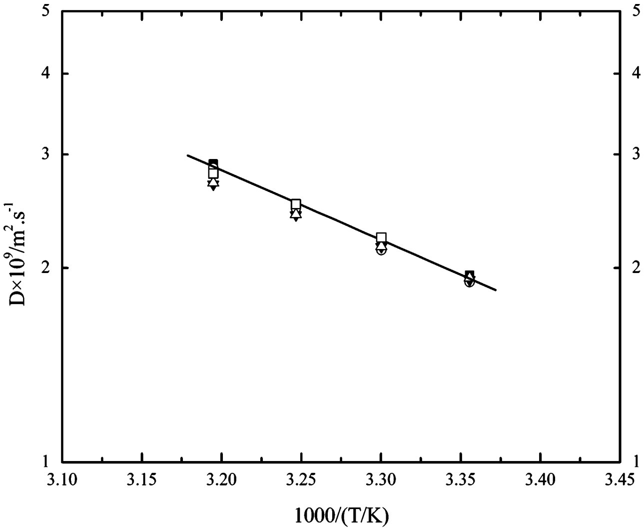

Figure 5. Diffusivity of CO_2 in water as a function of temperature: ▼, Versteeg et al.;[6] Δ, Al-Ghawas et al.;[10] □, Li and Lee;[11] ○, Mandal et al.;[12] ■, this study; —, calculated by eq 7.

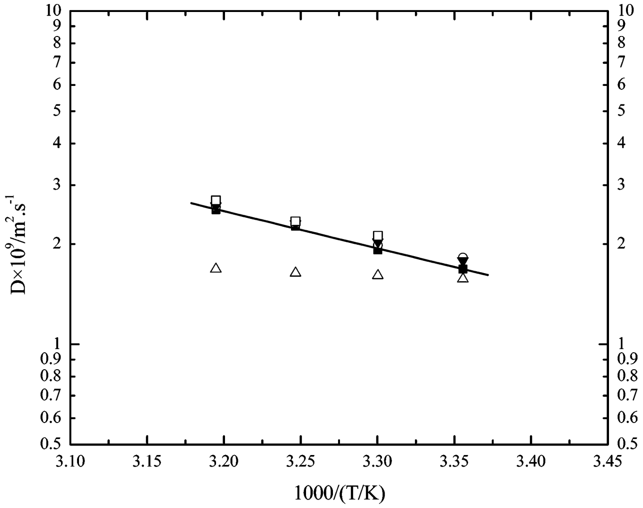

Figure 6. Diffusivity of N_2O in water as a function of temperature: ▼, Versteeg et al.;[6] Δ, Al-Ghawas et al.;[10] □, Li and Lee;[11] ○, Mandal et al.;[12] ■, this study; —, calculated by eq 8.

Table 5. Measured Diffusivity of N_2O and CO_2 in Water

$\frac{T}{\mathrm{K}}$	$\frac{D_{N_2O-\mathrm{water}} \cdot 10^9}{\mathrm{m^2 \cdot s^{-1}}}$	$\frac{D_{CO_2-\mathrm{water}} \cdot 10^9}{\mathrm{m^2 \cdot s^{-1}}}$
298	1.68	1.95
303	1.92	2.20
308	2.26	2.48
313	2.53	2.90

Results and Discussion

Solubility. To confirm the validity of the experimental method and the apparatus used for the present study, the solubilities of CO_2 and N_2O in water were measured at (293, 298, 303, 308, and 313) K. The results are presented in Table 1 and compared with the literature data[6,10] in Figures 1 and 2. There is a good agreement between the literature results and the results of the present study, thus supporting the applicability of the present experimental method. The results of the solubility of N_2O and CO_2 in water have been correlated as a function of temperature by eqs 3 and 4, respectively, as follows.

$$H_{CO_2-\mathrm{water}}/\mathrm{kPa \cdot m^3 \cdot kmol^{-1}} = 6.17 \cdot 10^6 \exp(-2276/(T/\mathrm{K})) \quad (3)$$

$$H_{N_2O-\mathrm{water}}/\mathrm{kPa \cdot m^3 \cdot kmol^{-1}} = 4.86 \cdot 10^6 \exp(-2117/(T/\mathrm{K})) \quad (4)$$

The data for the solubility of N_2O and the estimated solubility of CO_2 using the N_2O analogy in aqueous solutions of PZ and PZ activated MDEA blends for the temperatures (293, 298, 303, 308, and 313) K are presented in Tables 2 and 3 and shown in Figures 3 and 4. The experimental solubility data of N_2O in

Table 6. Estimated Diffusivity of CO_2, D_{CO_2}, in PZ (2) + Water (3) Using the N_2O Analogy

	$D_{N_2O}\cdot10^9/m^2\cdot s^{-1}$				$D_{CO_2}\cdot10^9/m^2\cdot s^{-1}$			
100 w_2	$T = 298.0$ K	$T = 303.0$ K	$T = 308.0$ K	$T = 313.0$ K	$T = 298.0$ K	$T = 303.0$ K	$T = 308.0$ K	$T = 313.0$ K
1.74	1.69	1.89	2.12	2.28	1.94	2.15	2.40	2.57
3.45	1.58	1.80	2.03	2.18	1.81	2.05	2.30	2.46
6.88	1.53	1.67	1.94	2.14	1.75	1.90	2.20	2.41

Table 7. Estimated Diffusivity of CO_2, D_{CO_2}, in MDEA (1) + PZ (2) + Water (3) Using the N_2O Analogy

	$D_{N_2O}\cdot10^9/m^2\cdot s^{-1}$				$D_{CO_2}\cdot10^9/m^2\cdot s^{-1}$			
w_1/w_2	$T = 298.0$ K	$T = 303.0$ K	$T = 308.0$ K	$T = 313.0$ K	$T = 298.0$ K	$T = 303.0$ K	$T = 308.0$ K	$T = 313.0$ K
28/2	0.73	0.91	1.02	1.15	0.84	1.04	1.16	1.30
25/5	0.61	0.77	0.83	0.97	0.70	0.88	0.94	1.09
22/8	0.54	0.65	0.75	0.84	0.62	0.74	0.85	0.95

Table 8. Test of Validity of the Modified Stokes−Einstein Equation (eq 9) for the Diffusivity of N_2O, D_{N_2O}, in PZ (2) + Water (3)

T		$D_{N_2O}\cdot10^9$	$\eta\cdot10^3$	$D\eta^{0.79}/T\cdot10^{14}$
K	100 w_2	$m^2\cdot s^{-1}$	$m\cdot Pa\cdot s$	$m^3\cdot Pa\cdot K^{-1}$
298	1.74	1.69	0.96	2.35
	3.68	1.58	1.02	2.31
	6.88	1.53	1.17	2.49
313	1.74	2.28	0.68	2.31
	3.68	2.18	0.72	2.30
	6.88	2.14	0.82	2.51

Table 9. Test of Validity of the Modified Stokes−Einstein Equation (eq 9) for the Diffusivity of N_2O, D_{N_2O}, in MDEA (1) + PZ (2) + Water (3)

T		$D_{N_2O}\cdot10^9$	$\eta\cdot10^3/$	$D\eta^{0.79}/T\cdot10^{14}$
K	w_1/w_2	$m^2\cdot s^{-1}$	$m\cdot Pa\cdot s$	$m^3\cdot Pa\cdot K^{-1}$
298	28/2	0.73	3.18	2.61
	25/5	0.61	3.34	2.28
	22/8	0.54	3.48	2.08
313	28/2	1.15	1.93	2.64
	25/5	0.97	1.97	2.27
	22/8	0.84	2.03	2.02

binary and ternary mixtures are correlated as a function of temperature and concentration of amine according to eq 5.

$$\ln H_{N_2O} = K_1 + \frac{K_2}{T} + K_3T \tag{5}$$

where the parameters, K_i's, are expressed as

$$K_i = a_i + b_i(w_1 + \alpha w_2) + c_i(w_1 + \alpha w_2)^2 \tag{6}$$

where a_i, b_i, c_i, and α are the correlation parameters and w_1 and w_2 are the mass percent of individual amine.

The calculated parameters and average absolute deviations (AADs) are listed in Table 4 for the (PZ + H_2O) and (MDEA + PZ + H_2O) systems. The calculated solubilities from eq 5 are in good agreement with the experimental results of this study. The AAD values between the correlated and experimental data for (PZ + H_2O) and (MDEA + PZ + H_2O) systems are about 1.32 % and 1.47 %, respectively. The measured and calculated solubilities from the correlation (eq 5) are compared in Figures 3 and 4. The solubility data of Sun et al.[13] and Derks et al.[14] for N_2O in aqueous PZ solutions for the temperature range (298 to 313) K show good agreement with the values found using eq 5, and the average absolute deviations are 3.3 % and 1.0 %, respectively. As shown in Figures 3 and 4, Henry's constant of N_2O in aqueous PZ increases with an increase in temperature and increases with the mass percent of PZ in the mixture, although the effect of PZ mass fraction on N_2O solubility in amines is less pronounced.

Table 10. Parameters and AADs for N_2O Diffusivity Correlations for the Binary and Ternary Solvent Systems

		a_i	b_i	c_i	AAD/%
	$i = 1$	−23.48	−0.122	−0.054	
(PZ + H_2O)	$i = 2$	0.21	$-7.72\cdot10^{-4}$	0.109	1.34
	$i = 3$	$3.78\cdot10^{-5}$	$-5.89\cdot10^{-5}$	$4.77\cdot10^{-4}$	
	$i = 1$	−26.41	−11.10	52.83	
(MDEA + PZ + H_2O)	$i = 2$	−29.81	−15.71	−2.86	3.27
	$i = 3$	2.70	−19.14	33.85	
	α		0.0999		

Diffusivity. To validate the diffusivity apparatus and the experimental method of the measurement, the diffusivities of CO_2 and N_2O in water were measured at (298, 303, 308, and 313) K. The results are presented in Table 5. The comparisons between the literature results[6,10−12] and results obtained in this study for the diffusivities of CO_2 and N_2O in water are shown in Figures 5 and 6. As shown in Figures 5 and 6, the diffusivities of CO_2 and N_2O in water obtained in this study are in good agreement with the literature values. However, the diffusivity data of N_2O in water reported by Al-Ghawas et al.[10] are somewhat different than those of this study. On the basis of the results, the diffusivities of N_2O and CO_2 in water can be correlated as a function of temperature by eqs 7 and 8, respectively.

$$D_{CO_2-water}/m^2\cdot s^{-1} = 8.13\cdot10^{-6}\exp(-2488/(T/K)) \tag{7}$$

$$D_{N_2O-water}/m^2\cdot s^{-1} = 9.72\cdot10^{-6}\exp(-2582/(T/K)) \tag{8}$$

The measured diffusivities of N_2O as well as the estimated diffusivity of CO_2 in (PZ + H_2O) and (MDEA + PZ + H_2O) for the temperatures (298, 303, 308, and 313) K are presented in Tables 6 and 7 and shown in Figures 7 and 8. The experimental diffusivities of N_2O in (PZ + H_2O) and (MDEA + PZ + H_2O) as presented in Tables 8 and 9 are correlated by a modified Stokes−Einstein equation (eq 9). The viscosity of (PZ + H_2O) has been taken from Samanta and Bandyopadhyay,[15] and the viscosity of (MDEA + PZ + H_2O) has been measured using the standard procedure described elsewhere.[15] Equation 9 predicts the experimental diffusivities of N_2O in (PZ + H_2O) and (MDEA + PZ + H_2O) with an average absolute deviation of 6.4 %.

$$\frac{D_{N_2O}\eta^{0.79}}{T} = \text{constant} \tag{9}$$

The prediction of diffusivity of N_2O from eq 9 also requires the knowledge of the solution viscosity. For convenience, however, the same data are correlated by a simpler equation

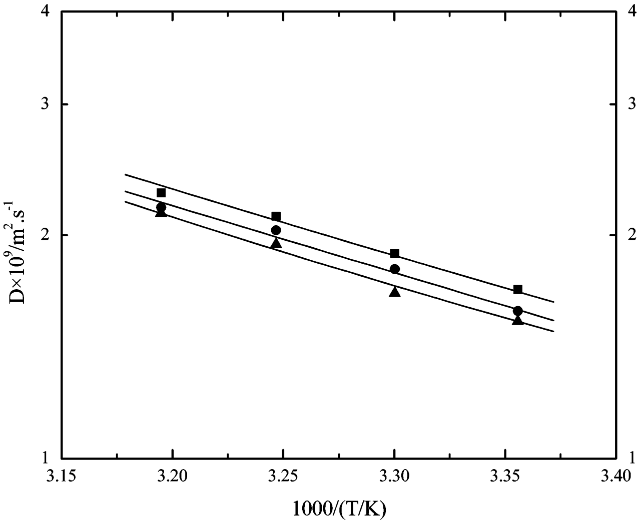

Figure 7. Diffusivity of N_2O in the PZ (2) + water (3) system at various temperatures: ■, $w_2 = 1.74$; ●, $w_2 = 3.45$; ▲, $w_2 = 6.88$; —, calculated by eq 10.

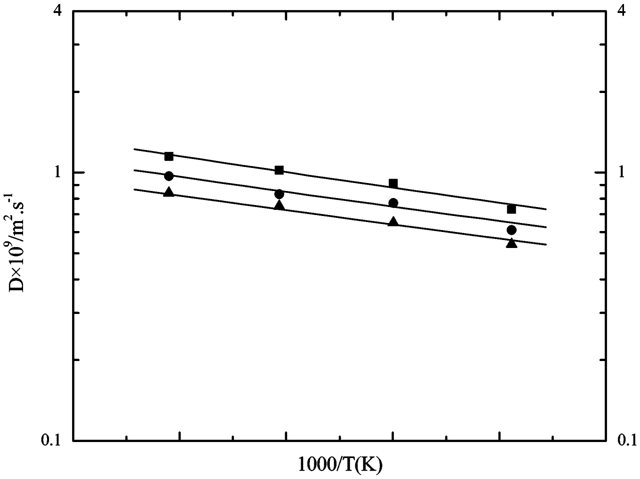

Figure 8. Diffusivity of N_2O in the MDEA (1) + PZ (2) + water (3) system at various temperatures: ■, $w_1/w_2 = 28/2$; ●, $w_1/w_2 = 25/5$; ▲, $w_1/w_2 = 22/8$; —, calculated by eq 10.

that takes into account the effect of solution viscosity implicitly. This equation has the following form

$$\ln D_{N_2O} = K_1 + \frac{K_2}{T} + K_3 T \qquad (10)$$

where the parameters, K_i's, are expressed as

$$K_i = a_i + b_i(w_1 + \alpha w_2) + c_i(w_1 + \alpha w_2)^2 \qquad (11)$$

Table 10 includes the determined parameters and AADs between the measured and calculated values for both (PZ + H_2O) and (MDEA + PZ + H_2O) solutions. The AAD values for (PZ + H_2O) and (MDEA + PZ + H_2O) solutions are 1.34 % and 3.27 %, respectively. The small AADs indicate that the calculated diffusivities from eq 10 are in good agreement with the experimental results of this study. The measured and calculated diffusivities from the correlation (eq 10) are also compared in Figures 7 and 8. The diffusivity data of Sun et al.[13] for N_2O in aqueous PZ solutions for the temperature range (303 to 313) K and for the PZ concentration range between 0.23 and 0.92 kmol·m^{-3} show good agreement with the values calculated using eq 10, the average absolute deviation being 4.41 %. The data reported by Sun et al.[13] for the estimated CO_2 diffusivities in (PZ+ H_2O) appear to be smaller than the measured diffusivities of N_2O, which is contradictory to the N_2O analogy. As shown in Figures 7 and 8, diffusivities of N_2O in aqueous PZ and blends of (MDEA + PZ) increase with an increase in temperature and increase with the decrease in mass fraction of PZ in the mixture, though the effect of PZ mass fraction in solution is less pronounced on the N_2O diffusivity in amines.

Conclusion

The physical solubility and diffusivity of N_2O in aqueous piperazine and piperazine activated aqueous MDEA solvents have been measured and correlated. The solubility measurements were done using a corning glass equilibrium cell over the temperature range (293 to 313) K, and the diffusivity measurements were done using a laboratory wetted wall contactor over the temperature range (298 to 313) K. The N_2O analogy was used to estimate the solubility as well as the diffusivity of CO_2 in (PZ + water) and in (MDEA + PZ + water). Calculated results using the correlations (eqs 5 and 10) for solubility and diffusivity of N_2O in binary and ternary mixtures are in good agreement with the experimental data of this study and literature results over the temperature and amine concentration ranges studied.

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