

Physicochemical Properties of 2-Piperidineethanol and Its Aqueous Solutions

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The physicochemical properties of 2-piperidineethanol (2-PE) and its aqueous solutions, such as the dissociation constant, density, and viscosity, as well as the solubility of N_2O in these solutions were determined over a wide range of temperatures and compositions. The dissociation constant of 2-PE is higher than those of the conventional alkanolamines. The standard-state enthalpy change of the dissociation reaction is 53.8 kJ/mol. The properties of 2-PE are correlated with temperature with good precision. The excess properties of aqueous 2-PE solutions are empirically correlated as functions of temperature and composition over the whole experimental range. The average deviations for the regression of the aqueous solutions are 0.031%, 2.72%, and 4.13% for density, solubility, and viscosity, respectively. The N_2O analogy is recommended for estimation of the solubility of CO_2 in aqueous 2-PE solutions.

1. Introduction

Sterically hindered alkanolamines have many advantages for the removal of the acid gas compounds H_2S and CO_2 from natural and refinery gases over conventional alkanolamines such as MEA, DEA, and MDEA (1-3). Sterically hindered alkanolamines have a low tendency to form carbamates with CO_2 due to the bulkiness of the substituent attached to the amino group. Also, their high loading for the acid gases and considerable reaction rate with CO_2 at the same time (2-6) cause an increased cyclic capacity and a decreased energy consumption for the industrial operation. Like 2-amino-2-methyl-1-propanol (AMP), 2-piperidineethanol (2-PE) is one of the sterically hindered amines, where a naphthenic ring group attached to the amino group in the structure of 2-PE makes 2-PE similar to a secondary amine. As a result, the different molecular structure will result in different physicochemical characteristics on which a kinetic study depends. The fundamental data are important for process design and optimization. Unfortunately, physicochemical data for 2-PE are very scarce, especially for the dissociation constant which is a significant quantity affecting both CO_2 reaction rate and capacity.

Shen et al. (7) recently investigated the kinetic rate of aqueous 2-PE solutions with CO_2 at 40 °C within the amine concentration range 0.2-1.0 kmol/m³. The density and viscosity of the aqueous solutions and the group $D^{1/2}/H$ of CO_2 and N_2O under these conditions were also presented in their paper. However, the concentration of amine (2-PE) was limited to 1.00 kmol/m³. These data are not sufficient for the kinetic study and industrial design.

In this study, the dissociation constant of pure 2-PE was determined between 15 and 60 °C. As well, the other physicochemical properties of 2-PE and its aqueous solutions, such as the density, viscosity, and solubility of N_2O , were measured over a wide range of temperatures and concentrations. Correlations that allow the estimation of the properties of aqueous 2-PE solutions in the experimental range were prepared.

2. Experimental Section

2.1. Chemicals. Analytical grade 2-PE with a purity of $\geq 95\%$, supplied by Aldrich Chemical (Milwaukee, WI), was

used in the determination of density, viscosity, and solubility. The 2-PE used for determining the dissociation constant was distilled at reduced pressure, and then it was kept under an atmosphere of nitrogen. The purity of the distilled 2-PE was higher than 99.5%, as analyzed by gas chromatography. The AMP (purity $\geq 99\%$) supplied by Aldrich Chemical was used as received.

The accuracies of the buffer solutions used to calibrate the pH meter were ± 0.01 and ± 0.02 at 25 °C, respectively, for pH 4.00, 7.00, and 10.00. The titration acid was 0.1000 ± 0.005 M hydrochloric acid. These chemicals were provided by Fisher Scientific Co.

All solutions were prepared with distilled water except the solutions for the measurement of the dissociation constant, for which reverse osmosis water obtained by passing triple-distilled water through anion-exchange, cation-exchange carbon and a 0.45- μm cartridge was used. The conductivity of the reverse osmosis water was higher than $0.06 \mu S cm^{-1}$.

The N_2O used for the measurement of the solubility had a minimum purity of 99.8%, and the nitrogen used for blanketing the titration of the dissociation constant had a purity of 99.99% without any oxygen. They all were supplied from Union Carbide Canada.

2.2. Density and Viscosity. Densities and viscosities were determined individually using calibrated 25-mL pycnometer bottles and Cannon-Fenske routine viscometers. The measurements were performed in a constant-temperature water bath, in which the temperature could be held constant to ± 0.05 K. The experimental errors were estimated to be equal to ± 0.01 kg/m³ and $\pm 1 \times 10^{-6}$ N-s/m², respectively.

2.3. Solubility. Solubilities were determined in a glass flask with a volume of 250 mL at the local pressure of about 94 kPa. The temperatures of the water bath and environment could be kept constant within ± 0.05 and ± 0.2 K, respectively. About 50 mL of solution weighed by a balance with precision $\pm 1 \times 10^{-6}$ kg was injected into the flask. After thermodynamic equilibrium was reached in the flask, the change of gas volume was measured with a cathetometer. The partial pressures of N_2O in the experiments were corrected for the vapor pressure of the solution. The maximum experimental error in the measured gas solubility was estimated to be $\pm 2\%$.

The vapor pressure of the aqueous 2-PE solution was approximately estimated by Raoult's law. The vapor pressure of the pure solvent 2-PE was correlated as an exponential

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Table I. Density of 2-PE and Its Aqueous Solutions

$t, ^\circ\text{C}$	2-PE concn, wt %	$\rho, \text{kg}\cdot\text{m}^{-3}$	$t, ^\circ\text{C}$	2-PE concn, wt %	$\rho, \text{kg}\cdot\text{m}^{-3}$
30.6	100.0	991.9	25.0	45.0	1018.7
40.7	100.0	984.2	35.7	45.0	1011.5
57.9	100.0	971.6	50.0	45.0	1001.5
68.7	100.0	962.9	68.3	45.0	988.4
84.2	100.0	950.5	83.5	45.0	976.4
25.0	10.0	1002.7	25.0	60.0	1021.2
35.7	10.0	998.9	35.7	60.0	1013.5
50.0	10.0	992.4	50.0	60.0	1002.6
68.3	10.0	982.5	68.3	60.0	988.8
83.5	10.0	971.3	83.5	60.0	976.4
25.0	30.0	1013.3	25.0	75.0	1020.4
35.7	30.0	1007.2	35.7	75.0	1012.0
50.0	30.0	998.5	50.0	75.0	1000.8
68.3	30.0	986.9	68.3	75.0	986.6
83.5	30.0	975.4	83.5	75.0	973.7

Table II. Viscosity of 2-PE and Its Aqueous Solutions

$t, ^\circ\text{C}$	$\eta, 10^3 \text{ Pa}\cdot\text{s}$					
	10.0 wt %	30.0 wt %	45.0 wt %	60.0 wt %	75.0 wt %	100.0 wt %
25.0	1.426	3.959	9.631	24.900	89.029	
40.0	1.025	2.371	5.150	11.327	35.529	77.171
53.1	0.753	1.719	3.341	6.644	16.374	33.483
65.2	0.610	1.255	2.343	4.299	9.400	17.182
75.3	0.527	1.016	1.815	3.134	6.301	10.715
85.2	0.459	0.858	1.442	2.371	4.412	7.139

function of the temperature on the basis of the data taken from the *CRC Handbook of Chemistry and Physics* (8).

2.4. Dissociation Constant. The dissociation constants, $\text{p}K_a$, of the amine solvent were determined by potentiometric titration of a 25-mL solution (<0.1 M 2-PE) with 0.1 M standard hydrochloric acid in a special jacketed beaker that could be kept at a constant temperature within $\pm 0.05 \text{ K}$. The solutions used in this experiment were protected by high-purity nitrogen. The pH meter, Model 520 digital pH/ion meter produced by the Fisher Scientific Co., was directly read with an accuracy of $\pm 0.002 \text{ pH}$ unit and reproducibility of $\pm 0.001 \text{ pH}$ unit. The pH meter, combined with glass and reference electrodes, was calibrated by the buffer solutions in the range of pH 4–10 before determining the dissociation constant. After the dilute amine solutions were made, they were stripped by nitrogen to remove trace amounts of gases, such as oxygen. Then, the solutions were stored under a nitrogen atmosphere.

The $\text{p}K_a$ values of the amines were calculated from the titration curve of the pair of pH values and acid volume titrated. The activity coefficients used in this calculation were obtained from the Debye–Hückel equation:

$$-\log \gamma_i = \frac{Az_i^2 I^{1/2}}{1 + B\tilde{a}_i I^{1/2}} \quad (1)$$

where the parameters A and B are functions of the temperature and the dielectric constant of the solution. The ionic radius parameters \tilde{a} were obtained from Barner and Scheuerman (9). The dissociation constant of water involved in the calculation was taken from the *CRC Handbook of Chemistry and Physics* (8). However, this activity coefficient equation could only be used for dilute solutions, in which the ionic strength was lower than 0.1 M.

3. Results and Discussion

3.1. Density and Viscosity. Densities and viscosities of the 2-PE solvent and aqueous 2-PE solutions over a wide temperature range are presented in Tables I and II. Also, the density and viscosity of 2-PE solvent are individually cor-

related with temperature, as shown in eqs 2 and 3. The average deviations of the regressions are 0.033% and 1.59%, respectively.

$$\rho_{2\text{-PE}} = 1015.57 - 0.767533t \quad (2)$$

$$\eta_{2\text{-PE}} = 8.0440 \times 10^{-4} \exp\left(\frac{1.9304 \times 10^3}{T - 128.287}\right) \quad (3)$$

Due to the polarity of 2-PE solvent, the density of its aqueous solution cannot be estimated by the ideal solution rule which is the sum of the product of the volume fraction and the density of solvent. In terms of the experimental results, the densities of aqueous 2-PE solutions should be regressed by

$$\rho_m = \phi_{2\text{-PE}}\rho_{2\text{-PE}} + \phi_w\rho_w + \Delta\rho \quad (4)$$

The excess density, $\Delta\rho$, is empirically correlated as follows:

$$\Delta\rho = \phi(1 - \phi)(1 + a_1\phi + a_2\phi^2 + a_3\phi^3)Tg \quad (5)$$

$$Tg = b_1 - b_2t + b_3t^2 - b_4t^3 \quad (6)$$

where ϕ_i is the volume fraction of species i in the solution, which is defined by eq 7. The parameters in eqs 5 and 6 are

$$\phi_i = x_i v_i / \sum x_i v_i \quad (7)$$

$a_1 = -7.062156 \times 10^{-2}$, $a_2 = 0.906124$, $a_3 = 0.924132$, $b_1 = 94.27849$, $b_2 = -1.369502$, $b_3 = 1.934676 \times 10^{-2}$, and $b_4 = -1.123692 \times 10^{-4}$. The average deviation for eq 4 is only 0.03%.

The viscosity of aqueous solutions, however, is not so easily correlated or estimated, since the aqueous 2-PE solution has not only polarity but also association effects between molecules. Furthermore, the structure of the constituent molecules in 2-PE can readily affect the viscosity of the aqueous solution. Although the Grunberg–Nissan equation (10) or Teja–Rice relation (11) is generally recommended to estimate or correlate the viscosities of liquid mixtures (12), both failed to be useful for aqueous 2-PE solutions. Besides the above-mentioned reasons, a large difference in viscosity between water and 2-PE at the same temperature can also cause the correlation methods to be unsuitable. Another method for correlating the viscosities of aqueous 2-PE solutions, therefore, has to be explored in this case.

According to the experimental data, the viscosities of aqueous 2-PE solutions can be expressed as eq 8. Moreover,

$$\ln(M_m \eta_m) = \sum x_i \ln(M_i \eta_i) + \eta_{\text{ex}} \quad (8)$$

$$M_m = \sum x_i M_i \quad (9)$$

the excess viscosity for the mixture has to be correlated as

$$\eta_{\text{ex}} = x_w x_{2\text{-PE}} \{a_1 + x_{2\text{-PE}}(a_2 + a_3 x_w + a_4 x_w^2 + a_5 T) + a_6 T^2 + (a_7 + a_8 T^{-1} + a_9 T^{-2})T^{-1}\} \quad (10)$$

where the parameters correlated from the experimental values in this work are listed as follows: $a_1 = 45.36349$, $a_2 = 10.29523$, $a_3 = -65.80022$, $a_4 = -30.00883$, $a_5 = 2.89443 \times 10^{-2}$, $a_6 = -1.90715 \times 10^{-4}$, $a_7 = -8.29877$, $a_8 = 4.79492 \times 10^4$, and $a_9 = 12.88693$. The average deviation of regression is only 4.13%. These equations meet the requirements of scientific research and practical purposes.

Figures 1 and 2 show that the excess values of both density and viscosity for the aqueous 2-PE solutions are positive. The plot of the excess density against volume fraction of 2-

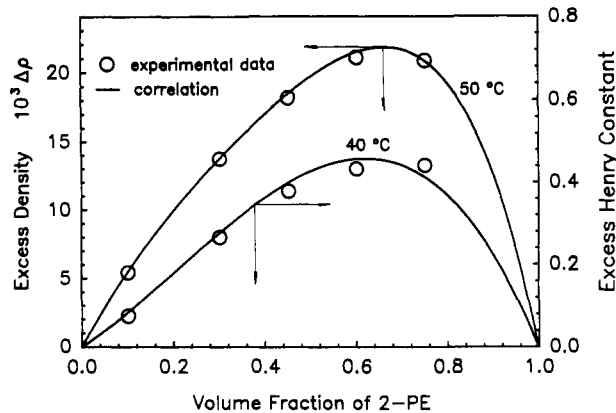


Figure 1. Excess property dependence of the volume fraction.

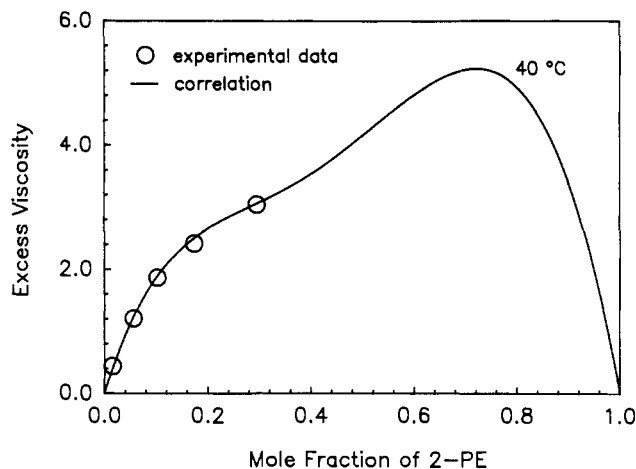


Figure 2. Excess viscosity dependence of the mole fraction.

Table III. Solubility of N₂O in 2-PE and Its Aqueous Solutions

<i>t</i> , °C	2-PE concn, wt %	<i>H</i> _{N₂O} , kPa·m ³ /kmol	<i>t</i> , °C	2-PE concn, wt %	<i>H</i> _{N₂O} , kPa·m ³ /kmol
31.4	100.0	1851.8	25.0	45.0	4286.8
45.7	100.0	2116.8	40.0	45.0	5279.6
59.1	100.0	2728.3	60.0	45.0	7092.7
72.1	100.0	3431.2	25.0	60.0	3811.3
83.1	100.0	4017.4	40.0	60.0	4650.5
25.0	10.0	4138.2	60.0	60.0	5642.9
40.0	10.0	5633.7	25.0	75.0	3226.2
60.0	10.0	8072.2	40.0	75.0	4198.1
25.0	30.0	4320.4	60.0	75.0	4794.8
40.0	30.0	5532.7			
60.0	30.0	7458.9			

PE represents a parabolic shape at a constant temperature, whereas the curve of the excess viscosity against mole fraction of 2-PE is strikingly different. In analyzing Figure 2, it is interesting to find that the excess viscosity increases dramatically with the increase of the concentration of 2-PE in the low mole fraction range, and then changes more gently with the increase of the mole fraction of 2-PE until the maximum excess viscosity is reached. After that, the excess viscosity decreases sharply with the increase of the concentration of 2-PE within the range of mole fractions from 0.8 to 1.0.

The comparison of viscosities of the aqueous 2-PE solutions at 40 °C calculated by the method mentioned above with those proposed by Shen et al. (7) is listed in Table IV. The results of this work are slightly lower than those of Shen et al. The difference in viscosities between Shen et al. and this work mainly results from impurities in the amine solution in addition to the deviations of measurement and regression,

Table IV. Comparison of the Properties of Aqueous 2-PE Solutions at 40 °C^a

concn, kmol/m ³	ρ , kg/m ³	η , 10 ³ Pa·s	$(D^{1/2}/H)_{N_2O} \times 10^8$	H_{N_2O} , kPa·m ³ /kmol	D_{N_2O} , 10 ⁹ m ² /s
0.00	992.3	0.720	0.788		
0.107	992.6	0.742	0.770		
0.218	993.1	0.780	0.710		
0.320	995.1	0.816	0.613		
1.00	996.9	1.133	0.529		
0.00	992.1	0.653		5810.7	2.08 ^b
0.107	992.9	0.690		5786.6	1.99 ^b
0.218	993.7	0.730		5764.3	1.68 ^b
0.320	994.4	0.769		5746.2	1.24 ^b
1.00	998.4	1.108		5669.7	0.90 ^b

^a The top set of data were obtained from ref 7. The bottom set were calculated in this work. ^b Obtained from the group $(D^{1/2}/H)_{N_2O}$ of ref 7.

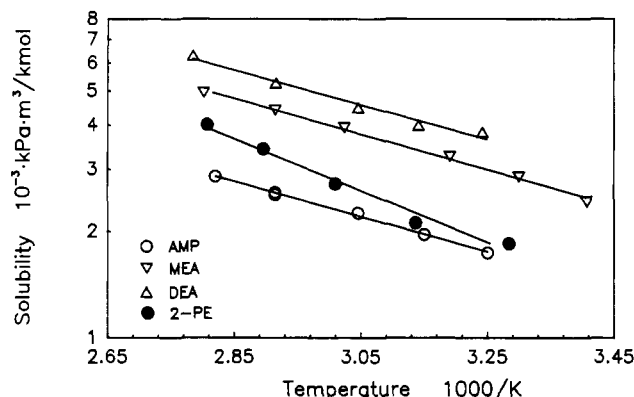


Figure 3. Solubility of N₂O in pure amines.

since they added a small amount of surface-active agent Tween 80 to suppress the formation of ripples on the liquid face of the wetted-wall column (13). Although the concentration of surface-active agent is very low, it can still cause a change in the viscosity of aqueous 2-PE solutions, especially in dilute solutions. Table IV also indicates that the effect of the surface agent on the viscosity of aqueous 2-PE solutions decreases with the increase of the concentration of 2-PE.

3.2. Solubility. The solubilities of N₂O in 2-PE solvent and in aqueous 2-PE solutions measured from 25 to 85 °C are listed in Table III. The solubility of N₂O in 2-PE solvent is correlated with temperature by eq 11. The average deviation

$$H_{N_2O} = 4.65 \times 10^5 \exp(-1700/T) \quad (11)$$

is 3.49%. Figure 3 shows that the solubilities of N₂O in sterically hindered amine solvents are generally higher than those of the conventional alkanolamines (14) due to the amino groups attached to a tertiary or secondary carbon atom. Also, comparison of the solubility values of the sterically hindered amines AMP and 2-PE indicates that Henry's coefficient of N₂O in 2-PE is higher than that in AMP. This means that the amount of N₂O absorbed per cubic meter of AMP solution is larger than that in 2-PE solvent. If the unit of Henry's coefficient is changed to per mole of alkanolamine solvent, however, it is just the reverse, which is shown in Figure 4, since the number of carbon groups for 2-PE is greater than that for AMP, and the structure of 2-PE is a cyclic compound. This special structure of 2-PE looks like a cage that can increase the attractive force between carbon groups and gas molecules.

The solubilities of N₂O in aqueous 2-PE solutions are correlated over a wide range of temperatures and concentrations of amine on the basis of the method proposed by Wang et al. (14) because of the effect of a nonideal positive

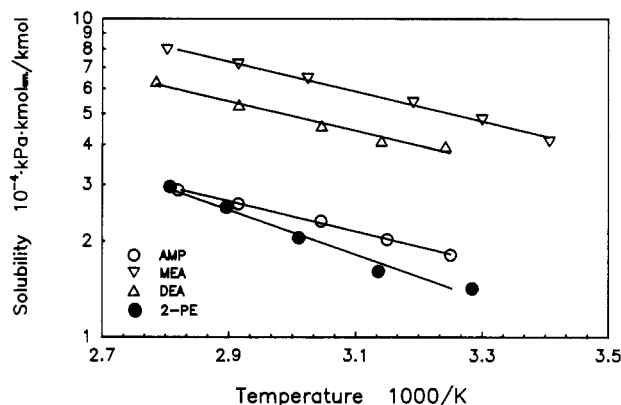


Figure 4. Solubility of N_2O in pure amines.

deviation solution. The solubility model for aqueous 2-PE solutions obtained in this case, described by eq 12, can be

$$\ln H_m = \phi_{2-PE} \ln H_{2-PE} + \phi_w \ln H_w + R_{23} \quad (12)$$

reliably used to estimate the solubility of N_2O in aqueous 2-PE solutions within the experimental range. ϕ_i is the volume fraction for species i , and R_{23} is a binary excess property of solubility for mixed solutions, and is presented by the following equation:

$$R_{23} = \phi_{2-PE} \phi_w (a_1 + a_2 t + a_3 t^2 + a_4 \phi_w) \quad (13)$$

Here $a_1 = 3.300753$, $a_2 = -2.302352 \times 10^{-3}$, $a_3 = -3.282063 \times 10^{-4}$, and $a_4 = -1.950792$. The parameters were obtained from the experimental values of Table III. The average deviation of the regression is 2.72%. The solubility of N_2O in water is obtained from eq 14 which was presented by Versteeg and van Swaaij (15).

$$H_w = 8.55 \times 10^6 \exp(-2284/T) \quad (14)$$

Figure 1 clearly shows that the excess solubility has a parabolic profile as well as a positive deviation. It decreases with the increase of the temperature for a constant concentration of the amine, and first increases with the increase of the volume fraction of the amine and then decreases at a constant temperature. Nevertheless, the solubility of CO_2 in aqueous 2-PE solutions can be estimated readily according to the analogy method, in which the analogy parameter of water was given by Versteeg and van Swaaij (15).

According to the regression equations of the density, viscosity, and solubility of N_2O for aqueous 2-PE solutions, it is easy to obtain the properties of the solutions at 40 °C, which are compared with the data of Shen et al. (7) listed in Table IV. The diffusivities of N_2O in the aqueous 2-PE solutions are also calculated on the basis of the values presented by Shen et al. However, either the value of diffusivity of N_2O in water from Table IV or Henry's constant of N_2O in water measured by Shen et al. (7) is lower than that presented by Versteeg and van Swaaij (15), whose data for the diffusivity of N_2O in water is $2.61 \times 10^{-9} \text{ m}^2/\text{s}$ at 40 °C.

3.3. Dissociation Constants. The dissociation constants of amines were measured in the temperature range from 15.0 to 60.0 °C, since the highest temperature is limited by the standard buffer solutions. In order to check the measurements of this work, the dissociation constants of AMP were measured before determination of the pK_a of 2-PE. Table V shows that the dissociation constants of AMP obtained in this experiment are in good agreement with the published data of Perrin (16) and Littel et al. (17). It verifies that this experimental method is reliable. This experimental accuracy is within ± 0.02 pK_a unit.

Table V. Dissociation Constants of Amines

$t, ^\circ\text{C}$	AMP			2-PE this work
	ref 16	ref 17	this work	
10.0	10.194			
15.0	10.022 ^a		10.04	10.47
20.0	9.853	9.88		
25.0	9.694 ^a		9.64	10.14
30.0	9.535	9.52		
35.0	9.387 ^a		9.40	9.82
40.0	9.240			
45.0	9.100 ^a	9.20	9.13	9.52
50.0	8.963			
60.0	8.705	8.78	8.81	9.16

^a Calculated by the regression equation.

Table VI. pK_a and Standard-State Enthalpies of Alkanolamines at 25 °C

alkanolamine	pK_a	$\Delta H^\circ, \text{kJ/mol}$	ref
MEA	9.500	50.55	19
DEA	8.883	41.93	19
TEA	7.762	33.91	19
DIPA	8.88	42.7	19
MDEA	8.52	35.2	19
AMP	9.694	53.93	16
2-PE	10.14	53.8	this work

The experimental results of the dissociation constants of 2-PE are listed in Table V, and correlated with temperature as eq 15, in which the average deviation is only 0.13%. The

$$pK_{a,2-PE} = 0.7050 + 2811.37/T \quad (15)$$

values of pK_a for 2-PE are higher than those of AMP at the same temperature. This means that the alkalinity of 2-PE is slightly stronger than that of AMP. Its reaction rate constants with CO_2 may be higher than that of all alkanolamines. Therefore, 2-PE is one of the best sterically hindered amines that should be considered for use in the removal of CO_2 , since it was supposed that the second-order rate constant of CO_2 in the aqueous amine solution depends on the dissociation constant of amine (18). In terms of thermodynamics, the van't Hoff equation can be used to calculate the desired standard-state enthalpy change (ΔH°) of reaction of amine dissociated in water. The standard-state enthalpies of AMP and 2-PE are 53.93 and 53.8 kJ/mol, respectively. In Table VI, it is readily seen that the sterically hindered amines have higher ΔH° values than those of conventional alkanolamines.

4. Conclusions

The physicochemical properties of 2-PE solvent and aqueous 2-PE solutions over a wide range of temperature and composition were investigated in this work. Also, they were empirically correlated with the temperature and composition under the experimental conditions. These equations should be very useful for kinetic research and industrial design as well as optimization.

The experimental results also show that the solubility of N_2O in 2-PE solvent is lower than that in AMP solvent, while it is larger than in other alkanolamine solvents. The solubility of CO_2 in this aqueous solution can be obtained using the analogy method. For the aqueous solutions, the excess properties of density, viscosity, and solubility have positive deviations with the concentration and temperature. Empirical equations have to be used for correlating those excess properties in order to get a high precision. The dissociation constant of 2-PE is slightly higher than that of AMP. It

implies that the absorption rate of CO₂ in the aqueous 2-PE solution is higher. This should be studied in the future.

Glossary

<i>a</i>	parameter of eqs 4, 10, and 13
<i>A</i>	parameter of eq 1
<i>b</i>	parameter of eq 5
<i>B</i>	parameter of eq 1
<i>H</i>	Henry's coefficient, kPa·m ³ /kmol
ΔH°	standard-state enthalpy change, kJ/mol
<i>I</i>	ionic strength, kmol/m ³
<i>K_a</i>	dissociation constant of amine
<i>M</i>	molecular weight, kg/kmol
<i>R</i>	gas constant
<i>t</i>	temperature, °C
<i>T</i>	temperature, K
<i>x</i>	mole fraction
<i>z</i>	charge number
γ	activity coefficient
η	viscosity, 10 ⁻³ Pa·s
ρ	density, kg/m ³
ϕ	volume fraction

Subscripts

am	alkanolamine
CO ₂	carbon dioxide
<i>i</i>	composition <i>i</i>
<i>m</i>	mixture
N ₂ O	nitrous oxide
w	pure water

Amine Abbreviations

AMP	2-amino-2-methyl-1-propanol
DEA	diethanolamine

MEA	monethanolamine
2-PE	2-piperidineethanol
MDEA	<i>N</i> -methyldiethanolamine

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