

Solubility of Nitrous Oxide in Amine Aqueous Solutions

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Abstract

The solubility of nitrous oxide (N₂O) was measured in both pure water over the temperature range (5-80)°C, and in amine aqueous solutions over the temperature range (20-60)°C under atmospheric pressure. The systems studied are monoethanolamine (MEA), diethanolamine (DEA), and diisopropanolamine (DIPA) aqueous solutions. A new correlation was developed for the solubility of N₂O in water, while a semi-empirical model of the excess Henry's constant was used to correlate the solubility of N₂O in amine solutions. The parameters of the correlation were determined from the measured solubility data. Generally, comparisons with experimental results from the reported literature indicate that the obtained correlations are satisfactory for estimating the solubility of N₂O in amine solutions, which could be used to estimate the free-gas solubility of CO₂ in amines.

Keywords

Nitrous Oxide, Monoethanolamine, Diethanolamine, Diisopropanolamine, Aqueous Mixtures

1. Introduction

The question of acid gas removal has become increasingly significant in the treatment of natural gas, synthetic gas, ammonia production, Claus feed gases and landfill gases. A wide variety of alkanolamines such as monoethanolamine (MEA), diglycolamine (DGA), diethanolamine (DEA), diisopropanolamine (DIPA), triethanolamine (TEA), N-methyldiethanolamine (MDEA), 2-amino-2-methyl-1-propanol (AMP), and 2-piperidineethanol (2-PE) can be used as absorbents for acid gas removal processes [1].

Solubility measurements are essential to the design of the absorption process but also to the measurement of the kinetic rates.

The reactivity of CO₂ with alkanolamine solutions makes the direct measurements of the physicochemical properties impossible [2]. The N₂O analogy has been frequently used to estimate the solubility of CO₂ in amine solutions [3-10].

The relation that has been used to calculate the solubility of CO₂ in amine solutions based on the N₂O analogy is

$$H_{CO_2} = H_{N_2O} \left(H_{CO_2} / H_{N_2O} \right)_{in\ water} \quad (1.)$$

Where H_{N_2O} is the solubility of N₂O in amine solution.

Calculating the physical solubility of CO₂ in amines via the N₂O analogy requires three measurements: the physical solubility of CO₂ and N₂O in water, and the solubility of N₂O in amine.

The numerous solubility data of N₂O in water reported in the literature [5-9,11-21] and some of those summarized in Table 1 are in certain cases scattered. Reported solubility data, of N₂O in amine aqueous solutions [1,4-7,9-11,14,17,23-26] are also scattered and inconsistent, which results in scattered and inconsistent data of the Henry's constant of N₂O in water and amine that may contribute to the inconsistent results for the reaction kinetics reported in the literature [27].

Accordingly, the correct solubility of N₂O in water and amines is essential to estimate the correct

solubility of CO₂ in an amine, which in turn can be used in developing the correct reaction kinetic models. Therefore, the objective of this work is to measure the solubility of N₂O in water over the temperature range (5-80) °C; and in pure MEA, DEA, and DIPA and their aqueous solutions over the temperature range (20-60)°C. A semi empirical model proposed by Wang et al. [17] and used by Tsai et al [10] will be used to correlate the solubility of N₂O in amine solutions. The parameters of the correlation for each system would be determined from the solubility of N₂O measured in this study and compared with the available data in the open literature. The correlation may then be used to estimate the solubility of N₂O in amine aqueous solutions, (MEA, DEA, and DIPA) for wide temperature and concentration ranges. Using the N₂O analogy the solubility data for CO₂ in these systems can be estimated.

2. Experimental Section

2.1 Experimental Apparatus

The reaction cell, shown in Figure 1, with a volume of 275 cc has been used. It is composed of a double walled stainless steel cylinder closed at both ends by two metallic flanges. The upper flange is connected to a piston that can be adjusted to keep a constant pressure in the vessel. Pressure is measured using a digital pressure indicator with an accuracy of ±0.3 mbar. A thermo-well holds a thermocouple to measure the temperature inside the vessel. N₂O gas is introduced through a tube connected to the upper flange. The lower part of the cell is equipped with a needle to feed the cell with solvent.

A thermostatic liquid is circulated inside the double-walled cylinder to control the temperature within ±0.1 K. The apparatus is installed over a vibrator that ensures good external agitation.

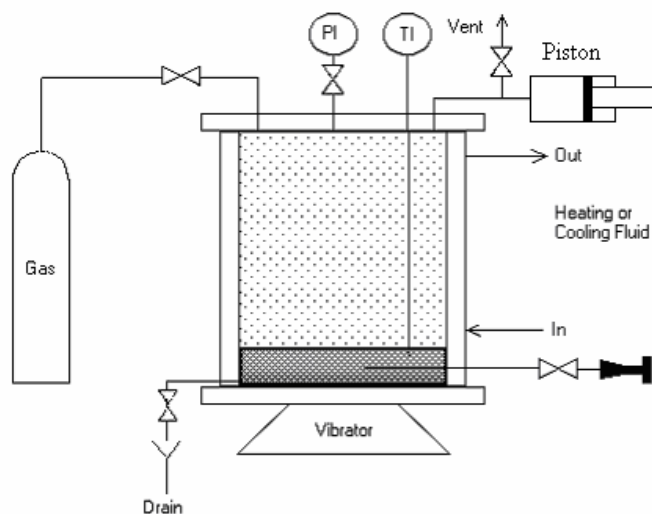


Figure 1. The reaction cell used for measuring N₂O solubility.

2.2 Experimental Procedure

All liquid solutions were prepared from distilled water and pure amines supplied by Sigma Aldrich. Medical grade N₂O with a purity of 99+% was used in all the solubility experiments. Water and amines are degassed independently, and aqueous solutions are prepared. The amounts of water and amines are known separately by differential weighing within 0.001 g. The flask containing the solution is kept inside the thermostatic water bath at the same temperature of the

experiment. The syringe is then connected to the reaction cell needle in order to transfer the solution by injection.

Accurate weighting of the syringe before and after the transfer yields the mass of solution present in the cell and then the liquid phase volume is calculated through the density correlation used by Glasscock [28].

2.2.1 Solubility

The solubility of N₂O in the aqueous amine solutions was determined by measuring the volume change in the constant pressure equilibrium cell. Initially, the cell was purged with N₂O at room temperature. The vent valve was then closed and heating started until the desired temperature was reached. A second purging with N₂O at that temperature was done before the cell was sealed. The closed system was allowed to reach constant pressure and temperature before a known mass (approximately 50 g) of degassed liquid was injected into the cell and pressure (P_i) and volume (V_i) were recorded. The vibrator was then started and the system was assumed to be at equilibrium when the temperature and volume stopped changing after a minimum of 80 minutes of continuous mixing. Moving the piston to the desired position allows the final pressure (P_f) to be maintained constant and equal to (P_i). Henry's law constant, H , was then calculated by the following equation

$$H = \frac{(P_f - P_w^V - P_A^V)}{[P_i V_i - (P_f - P_w^V - P_A^V) V_f]} \cdot V_l RT \quad (2.)$$

Where P_i and P_f are the initial and final pressures, respectively; P_w^V and P_A^V are the water and amine vapour pressures, respectively; V_i and V_f are the initial and final gas volumes, respectively; V_l the liquid volume; R is the ideal gas constant; and T is the absolute temperature.

3. Results and Discussion

Solubility was calculated in terms of Henry's law constant, H , and solubility C . The vapour pressure of the pure amines at different temperatures was neglected in all calculations [29], whilst the vapour pressure of pure water at different temperatures was calculated using

$$\ln(P_w^V / Pa) = 55.147 - \frac{6597.6}{T / K} - 4.3804 \ln(T / K) \text{ which is correlated from data given in Perry's}$$

Chemical Engineering Handbook [30], with the average regression error over the temperature range 0 to 120 °C being less than 0.1 %.

3.1 Solubility of N₂O in water

The measured solubilities of N₂O in water reported in the literature and those obtained in this study are summarized in Table 1. In Figure 2, a comparison between the literature values [5-8,11-14,18,22] and those obtained in this study for N₂O solubility in water are shown. The solid line represents calculated values using the following equation.

$$H_{N_2O,W} = 10.86 \times 10^6 \exp\left(\frac{-2372}{T}\right) \quad (3.)$$

Where $H_{N_2O,W}$ is the Henry's constant in Pa.m³.mol⁻¹ and T the absolute temperature.

The above equation is the correlation of the solubility of N₂O in water as a function of temperature from experimental data obtained in this work. The standard deviation was found to be 0.24.

Table 1. Solubility of N₂O in water

Solubility of N ₂ O in Water (Pa.m ³ .mol ⁻¹)											
T/K	Ref. 4	Ref. 5	Ref. 6	Ref. 7	Ref. 11	Ref. 12	Ref. 13	Ref. 17	Ref. 20	Ref. 21	This work
278											2 026
283											2 433
288	2 992		2 897		3 172						2 887
291.2		3 344									
292		3 484									
292.9		3 333	2 589.9								
293	3 482	3 425	3 321		3 506		3 694				3 306
298	4 169	4 132	3 911	4 176	3 982	4 101		4 179		4 314	3 821
298.6		3 774	3 809.8								
303		4 950	4 350		4 408				4 406		4 315
306		4 900				4 982				4 975	
308	5 284	5 263	4 710								4 899
312.9		5 917	4 249.6								
313		6 061	5 020				6 339		5 725	5 900	5 541
318		6 993	4 689.3								6 243
322.6		7 143	5 166.6								
322.9		7 407									
323			5 371	7 254.2		7 214		7 260	7 264		7 007
328											7837
333							9 105				8 737
338											9 708
340		10 309									
343											10 754
348				12 348							11 878
353		12 821					11 220				13 083
355.4		14 085									

From Figure 2, it is seen that the measured solubilities of N₂O in water are in good agreement with the values reported by Al-Ghawwas et al [7] over the temperature range (15-40) °C, and with the values reported by Versteeg et al [6] over the temperature range (45-80) °C.

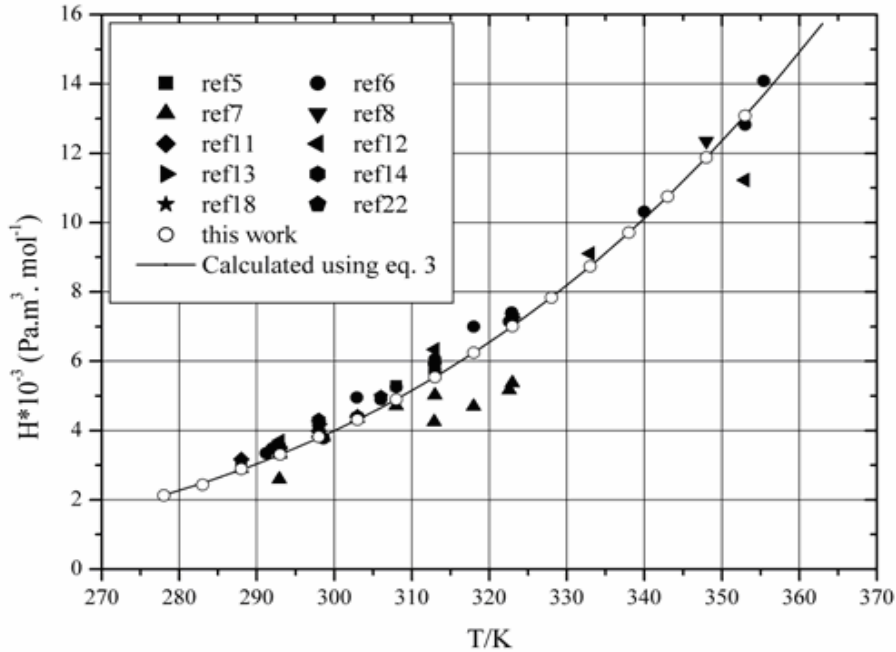


Figure 2. Comparison between literature values on solubility of N₂O in water and experimental values of this work.

3.2 Solubility of N₂O in pure amines

Experimental solubility data for N₂O in pure MEA, DEA, and DIPA was determined over the temperature range (20-60) °C by the above-mentioned method. The experimental values for each of the amines have been correlated as a quadratic function of temperature using

$$H_{N_2O, \text{Amine}} = a + bT + cT^2 \quad (4.)$$

Where $H_{N_2O, \text{amine}}$ is the Henry's constant in Pa.m³.mol⁻¹ and T the absolute temperature.

Parameters a, b, and c for the three pure amines (MEA, DEA, and DIPA) were calculated and tabulated in Table 2. The average regression deviations, for temperatures between 20 and 60 °C, between the calculated solubilities of N₂O in pure amines and experimental data is <0.65 %, which is satisfactory for estimating the solubilities of N₂O in pure amines. Figure 3 shows the experimental and calculated solubility data of pure MEA, DEA, and DIPA.

Table 2. Parameters in equation 4 for the solubility of N₂O in pure alkanolamines.

	a	b	c	% Error
N ₂ O-MEA	-12 922	63	-0.0362	0.22
N ₂ O-DEA	47 103	-305	0.5337	0.4
N ₂ O-DIPA	-24 129	128.6	-0.1423	0.65

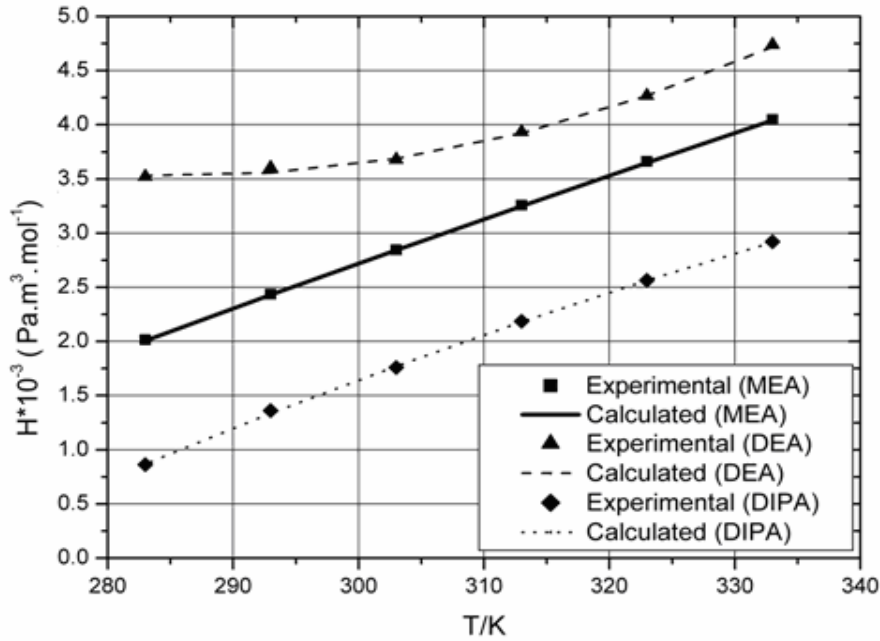


Figure 3. Experimental and calculated solubility of N₂O in pure MEA, DEA and DIPA.

3.3 Solubility of N₂O in amine aqueous solutions

The measured solubilities of N₂O in amine aqueous solutions over the temperature range (20 to 60) °C are presented in Table 3. The concentrations of amine vary between 5% and 30% (w/w).

A semi empirical model proposed by Wang et al [17] was used to correlate the solubility of N₂O in amine solutions. In this method, the excess Henry's coefficient for the binary system has the following form

$$R = \ln(H_{N_2O,m}) - \Phi_A \ln(H_{N_2O,A}) - \Phi_W \ln(H_{N_2O,W}) \quad (5.)$$

Where $H_{N_2O,m}$, $H_{N_2O,A}$, and $H_{N_2O,W}$ are Henry's constant of N₂O in the amine aqueous solution in pure solvent A and in water, respectively. Φ_A , and Φ_W are the volume fractions of

solvent *A* and water, respectively. From eq. 5, the excess Henry's quantity *R* can be calculated from the measured $H_{N_2O,m}$ and the estimated $H_{N_2O,A}$, and $H_{N_2O,W}$.

The calculated excess Henry's quantity is then correlated as a function of volume fraction,

$$R_{ij} = \Phi_i \Phi_j \alpha_{ij} \quad (6.)$$

Where the two-body interaction parameter, α_{ij} , is temperature dependent. It has assumed the expression

$$\alpha_{ij} = c_1 + c_2 T + c_3 T^2 + c_4 \Phi_w \quad (7.)$$

Where c_1 , c_2 , c_3 , and c_4 are parameters for each binary system and determined from corresponding solubility data of N₂O in (H₂O+MEA), (H₂O+DEA), and (H₂O+DIPA) solutions.

Solubility of N₂O in pure water, $H_{N_2O,W}$, is calculated using eq. 3. The solubility of N₂O in pure amines, $H_{N_2O,A}$, is calculated using eq. 4 and the parameters in Table 2.

Table 3. Solubility of N₂O in alkanolamine aqueous solutions

C _{amine} (Weight %)	H _{N₂O} (Pa.m ³ .mol ⁻¹)		
	MEA	DEA	DIPA
20 °C			
5	3 782	3 722	3 612
10	3 826	3 778	3 827
15	3 869	3 834	4 041
20	3 912	3 890	4 256
25	3 956	3 946	4 471
30	4 001	4 002	4 686
30 °C			
5	4 698	4 676	4 762
10	4 752	4 732	4 977
15	4 794	4 788	5 191
20	4 828	4 844	5 406
25	4 891	4 900	5 621
30	4 975	4 956	5 836

Table 3 continues

	H_{N_2O} (Pa.m ³ .mol ⁻¹)		
C_{amine} (Weight %)	MEA	DEA	DIPA
	40 °C		
5	5 624	5 630	5 912
10	5 687	5 686	6 127
15	5 723	5 742	6 341
20	5 748	5 798	6 556
25	5 787	5 854	6 771
30	5 821	5 910	6 986
	50 °C		
5	7 140	7 084	7 062
10	7 283	7 140	7 277
15	7 326	7 196	7 491
20	7 467	7 252	7 706
25	7 508	7 308	7 921
30	7 553	7 364	8 136
	60 °C		
5	8 845	8 738	8 212
10	8 889	8 794	8 427
15	8 932	8 850	8 641
20	8 976	8 906	8 856
25	9 019	8 962	9 071
30	8 863	9 018	9 286

Using the solubility data obtained in this study, i.e. Table 3, the parameters, C_1 , C_2 , C_3 , and C_4 in eq. 7 are determined for each amine solution system; the results are presented in Table 4. Comparisons of the calculated and experimental solubilities of N_2O in amine solutions are shown in Figures 4, 6 and 8.

For the MEA + H₂O system, the experimental values are shown in Figure 4 along with the results of the solubility calculation using eq. 7.

Figure 5 shows the application of the correlation obtained in this work, the correlation of Wang et al [17] and that of Tsai et al [10] to solubility data published by Little et al [24] over the temperature range of 30 to 75 °C.

Table 4. Parameters in eq. 7 for water-amine systems.

	C ₁	C ₂	C ₃	C ₄
H ₂ O-MEA	89.2	-5.54E-01	8.670E-04	0.443
H ₂ O-DEA	56.8	-3.53E-01	5.490E-04	0.948
H ₂ O-DIPA	-40.5	3.17E-01	-5.57E-04	-2.59

The figure shows that the correlation of Wang yields poor results for the solubility calculations at temperatures above 30 °C. The dotted lines in Figure 5 show the calculated values from the correlation of Tsai et al. It is clear in Figure 5 that the calculated values from the present correlation as well as those obtained from Tsai et al's approach experimental values up to 60 °C.

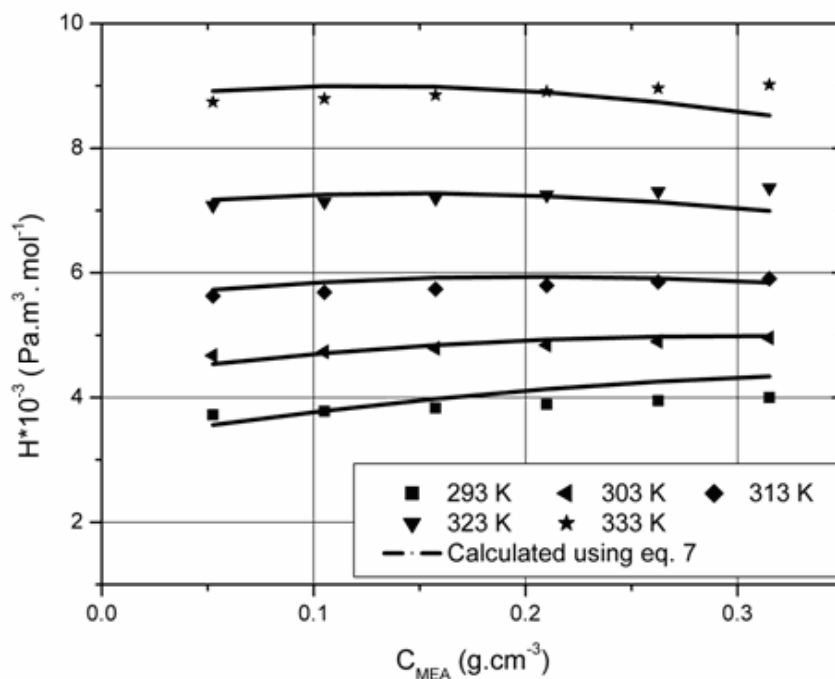


Figure 4. Calculated solubility of N₂O in aqueous MEA solutions.

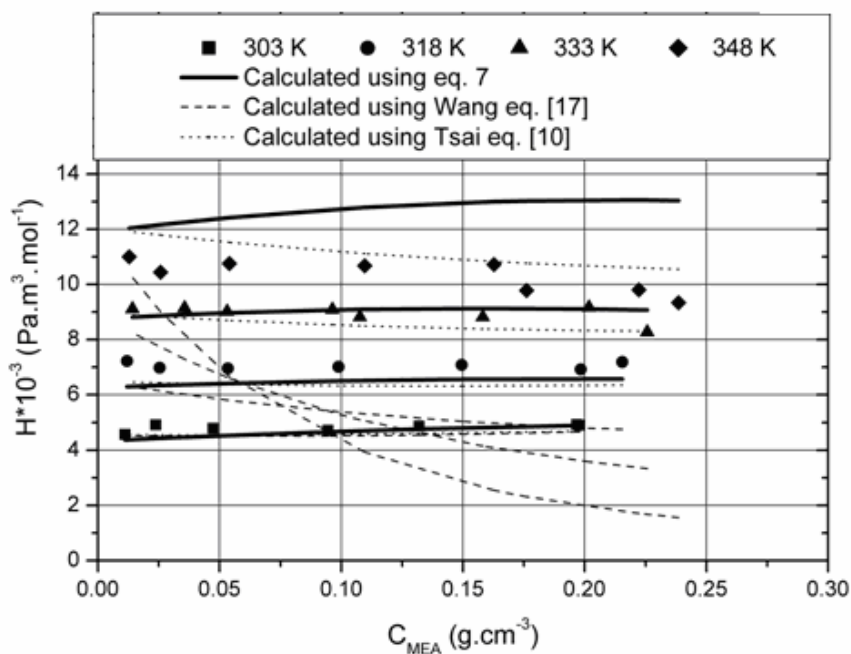


Figure 5. Comparison between calculated and experimental literature solubility data of N_2O in aqueous MEA solutions.

Experimental solubilities of N_2O in $DEA + H_2O$ are shown in Figure 6. There is a satisfactory agreement between experimental and correlation data at all temperatures and concentrations. The results of calculations, using equation 7, of the present work (solid lines), Tsai et al [10] (dotted lines), and Wang et al. [17] (broken lines) correlations when applied to experimental data of Little et al [24] are shown in Figure 7. The correlation of Wang et al [17] shows very poor predictions at low-temperature (15, 20, 25, and 30 °C) solubility data. On the other hand, results obtained by Tsai et al [10] correlation deviate from experimental values when the temperature is increased above 45 °C. However, the calculated Henry's constant values using equation 7 are consistent with most of the data of Little et al [24] at all temperatures and concentrations of DEA.

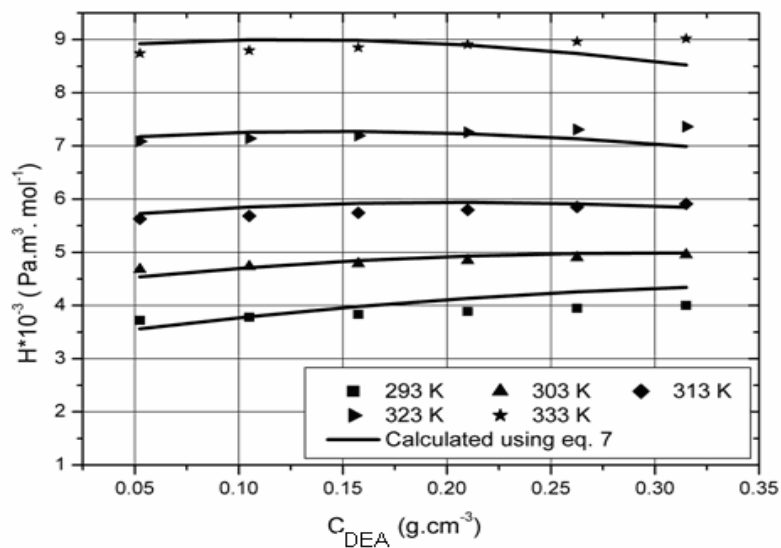


Figure 6. Calculated solubility of N₂O in aqueous DEA solutions.

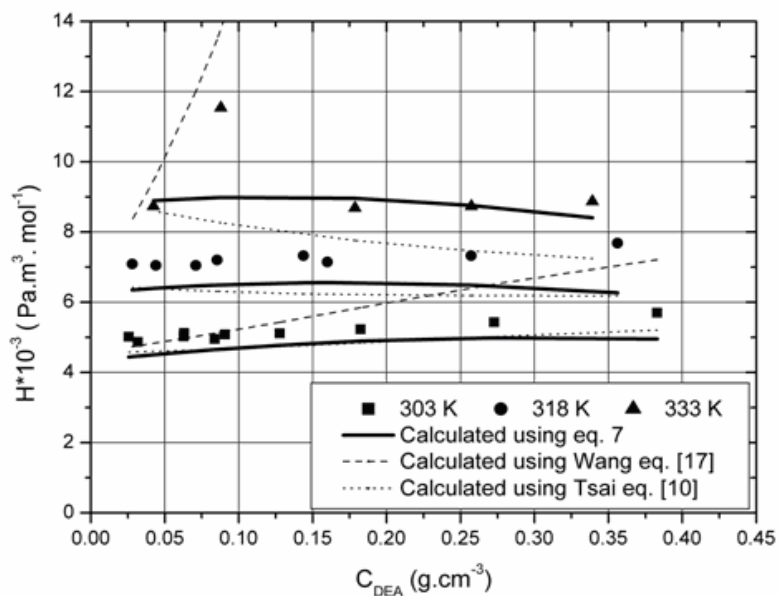


Figure 7. Comparison between calculated and experimental literature solubility data of N₂O in aqueous DEA solutions.

For the DIPA + H₂O system, the experimental values are presented in Figure 8 along with the results of the solubility calculation using eq. 7. There is a good agreement between experimental data and correlation for temperatures lower than 50 °C, while slightly lower values at high concentrations are shown when compared with the experimental values obtained in this study.

Figure 9 shows the application of the correlation to data taken from solubility data of Versteeg et al [6] over the temperature range from 25 to 60 °C. The solid lines, the broken lines, and the dotted lines in Figure 9 are calculated values from the use of equation 7, Wang et al. [17] correlation, and of Tsai et al. [10] correlation, respectively. There is satisfactory agreement between experimental data and the three correlations results for all temperatures and concentrations. Though, the correlation of Tsai et al. [10] gives high-calculated values at temperature of 60 °C and concentration above 2g/cm³.

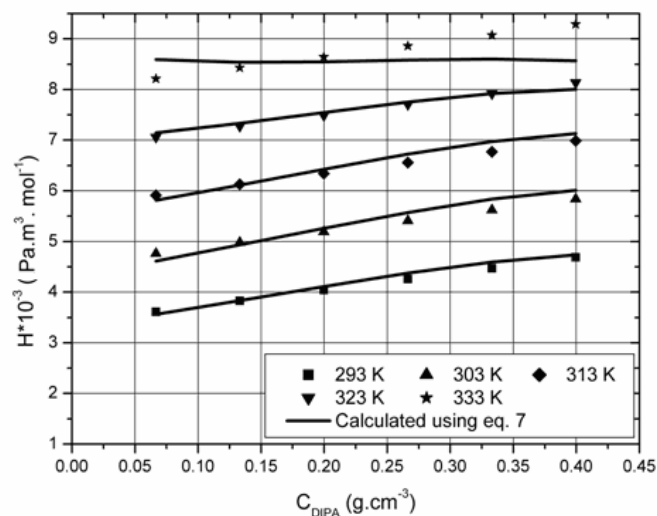


Figure 8. Calculated solubility data of N₂O in aqueous DIPA solutions.

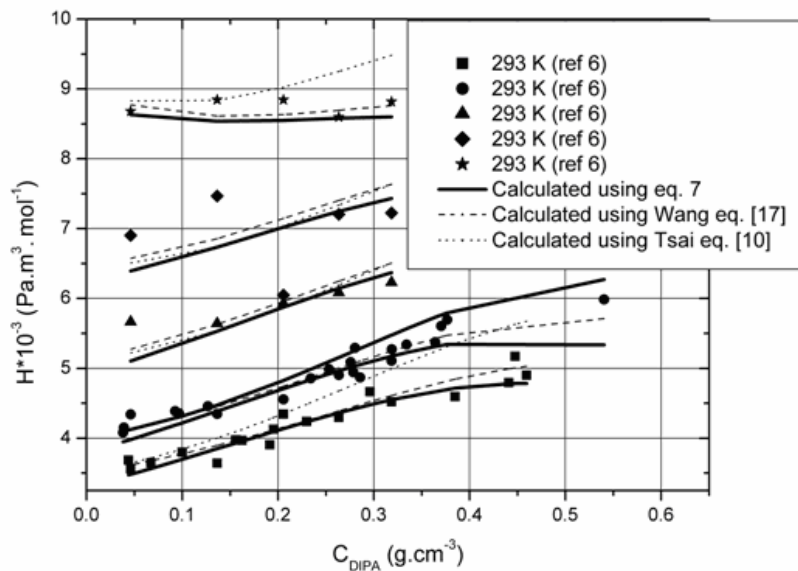


Figure 9. Comparison between calculated and experimental literature solubility data of N₂O in aqueous DIPA solutions.

4. Conclusions

The solubility of nitrous oxide in pure water over the temperature range (5 to 80) °C was measured and a new correlation was developed. Solubility data of N₂O in three pure amines MEA, DEA, and DIPA within the temperature range (20 to 60) °C shows that the solubility of N₂O in these amines could be represented by a quadratic function of temperature. Solubility of N₂O in the above-mentioned amine solutions was measured over the temperature range (20 to 60) °C. The concentration of amine ranges from (5 to 30) % mass. A semi-empirical model of the excess Henry's constant was used to correlate the solubility of N₂O in these amine solutions. The parameters of the correlation were determined from the solubility of N₂O obtained in this study. The obtained correlation has been shown to represent reasonably well the solubility of N₂O in the three amine aqueous solutions. In process design, the obtained correlations are in general satisfactory for estimating the solubility of N₂O in amine solutions that could be used to estimate the correct free-gas solubility of CO₂ in amines.

Acknowledgments

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