Volumetric Properties and Viscosities for Aqueous Diglycolamine Solutions from 25 °C to 70 °C

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This paper reports the measured values of the density and viscosity of aqueous diglycolamine [2-(2-aminoethoxyethanol)] solutions at five temperatures in the range 25 °C to 70 °C over the whole concentration range. The results are compared with data published in the literature. The derived excess molar volumes, partial molar volumes, apparent molar volumes, and viscosity deviations were correlated as a function of composition.

Introduction

Commercially known as diglycolamine (DGA), 2-2-aminoethoxyethanol or β-hydroxyaminoethyl ether is a primary amine and was developed to compete with monoethanolamine (MEA). DGA $[OH(CH_3)_2O(CH_3)_2NH_2]$ has a slightly lower enthalpy of reaction with CO_2 than MEA and is noted for its fast reaction rate with carbon dioxide. The equilibrium partial pressure of CO_2 over DGA solution is lower than that over MEA solutions, creating a more favorable driving force for mass transfer for DGA.[1] Comparison with systems using MEA solution indicates some capital and operating savings as well as improved operation at relatively low pressures.[2] Additional advantages are the partial removal of mercaptans and COS by DGA solution and that a substantial portion of DGA can be reclaimed from degradation products by steam distillation. In 1955, Blohm and Riesenfeld obtained a patent to use DGA in acid gas removal from natural gas. The use of aqueous solutions of DGA was commercialized jointly by Fluor Corporation, the El Paso Natural Gas Company, and the Jefferson Chemical Company Inc. The process is named the Fluor Econamine process and is similar to that for MEA (monoethanolamine) except that its lower vapor pressure permits its use in relatively high concentration, typically 40 to 60 mass %. This results in lower circulation rates and steam consumption when compared to those of typical MEA solutions. Hikita et al.[3] and Alper[4] studied the kinetics for the $CO_2 + DGA$ reaction. In 1996, diglycolamine solutions were being used in more than 100 plants.[2] Recently there has been renewed interest in using DGA in blends of tertiary amines.[1] Measurements of the densities and viscosities of alkanolamines covering the whole concentration range are scarce. Even though DGA is an important alkanolamine, to our knowledge, there are no published data on the densities and viscosities of aqueous DGA solutions. Researchers have relied on data extracted from the graphs provided by the Jefferson Chemical Company.[5,6] Dingman et al.[7] presented an empirical relation that represents the experimental data published by the Jefferson Chemical Company. Hikita et al.[8] published a few data of the density and viscosity of aqueous DGA at 25 °C.

Measurements of the density and viscosity of the aqueous DGA solutions were performed at various temperatures, (25, 40, 50, 60, and 70) °C, in order to cover a wide range of temperatures found in industry. The excess volumes, the apparent molar volumes, the partial molar volumes, and the deviation of the viscosity were derived.

Experimental Section

Diglycolamine (>98% pure) was purchased from Aldrich and was used without further purification.

The solutions were prepared by mass on an analytical balance (model Ap 205 D, Ohaus, Florham Park, NJ) with ±0.01 mg accuracy. The possible error in the mole fraction is estimated to be around ±0.0001. Densities of the binary mixtures were measured with an Anton Paar DMA-4500 density meter. Accuracies of our densities are about $\pm 5 \times 10^{-5}$ g·cm^{-3}. In the viscosity measurement, the temperature was controlled by means of a digital controller (±0.004 °C) in a well-stirred water bath to better than ±0.01 °C as measured by a Cole-Parmer resistance thermometer (model H-01158-65, Anjou, Québec, Canada).

The density can be determined by the two-parameter equation

$$\rho = A + B\tau^2 \tag{1}$$

where ρ is the liquid density and τ is the period of oscillation. The values of the density calculated by the density meter were checked at each temperature using deionized water,[9] and an air equation for the determination of the density of dry air was taken from the annual book of ASTM standards.[10] Viscosities were determined with two different viscometers to cover, with precision,[10] the range of temperature from 25 °C to 70 °C. An Ubbelohde suspended level viscometer No. 10 (J. Toonen Glass, Edmonton, Canada) and a Cannon-Ubbelohde viscometer (Cole-Parmer, E-98934-11) were used. The efflux time was measured with a hand-held digital stopwatch capable of measuring time to within ±0.01 s. Experiments were repeated a minimum of four times at each temperature for

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Table 1. Densities of Water (1) + DGA (2) Mixtures at Various Temperatures

	ρ/g·cm^{-3}				
x_2	25 °C	40 °C	50 °C	60 °C	70 °C
0.000	0.997 04	0.992 21	0.988 04	0.983 12	0.977 77
0.0552	1.021 77	1.014 62	1.009 19	1.003 32	0.997 02
0.1002	1.036 68	1.027 94	1.021 57	1.014 92	1.008 16
0.14466	1.047 46	1.037 31	1.030 22	1.022 88	1.015 31
0.2249	1.057 06	1.045 84	1.038 14	1.030 22	1.022 13
0.2997	1.060 56	1.048 88	1.040 87	1.032 78	1.024 52
0.3995	1.061 36	1.049 46	1.041 39	1.033 18	1.024 84
0.5392	1.060 31	1.048 22	1.040 19	1.032 01	1.023 67
0.5392	1.060 31	1.048 22	1.040 19	1.032 01	1.023 67
0.6075	1.058 50	1.046 45	1.038 30	1.030 11	1.021 78
0.7021	1.056 70	1.044 74	1.036 58	1.028 39	1.020 11
0.8071	1.054 90	1.042 80	1.034 68	1.026 56	1.018 28
0.9024	1.053 24	1.041 13	1.033 02	1.024 85	1.016 71
1.0000	1.051 56	1.039 53	1.031 37	1.023 21	1.015 09

all compositions. The equation for kinematic viscosity, according to Poiseuille's law, is

$$\nu = k_1 t - k_2/t \tag{2}$$

where t is the efflux time and k_1 and k_2 are the viscometer constants. The second term representing the correction due to the kinetic energy was found to be negligible.[11] The value of the absolute viscosity (η) was obtained by multiplying the measured kinematic viscosity (ν) by the measured density.

Calibration constants for the two viscometers were checked using high-purity ethylene glycol (EG), diethylene glycol (DEG), and water. A review of published values of the kinematic and dynamic viscosities of EG and DEG, with new experimental data, was published by Lee and Teja.[12] Values of the viscosity of water were taken from Stokes and Mills.[13] The values of the viscosities were reproducible to ±0.003 mPa·s. From the overall average percent deviation of the means of the average efflux time and the accuracy of the density measurement, we estimate the uncertainty of the absolute viscosity to be less than 0.5%.

Results and Discussion

Experimentally measured densities of the aqueous DGA solutions at (25, 40, 50, 60, and 70) °C throughout the whole concentration range are listed in Table 1. The values obtained are shown in Figure 1 and compare well with the data published by Hikita et al.[8] The density curves show a sharp increase as DGA is added to water, and a maximum occurs around 30 mol % (71 mass %) for all temperatures. In Figure 2, the values of the density are compared to data from the Jefferson Chemical Company,[5,6] as reported graphically by Kohl and Nielsen[2] and Pacheco.[1]

The density values of the binary mixtures were used to calculate the excess molar volume, V^E_m as

$$V^E_m = V_m - V°_1 x_1 - V°_2 x_2 \tag{3}$$

where V_m is the molar volume of the mixture and $V°_1$ and $V°_2$ are those of pure water and pure DGA, respectively; x_1 and x_2 are the mole fractions of the pure components. Figure 3 displays the dependence of V^E_m on the composition at various temperatures. In all cases, the V^E_m curves were negative, as is common for other completely miscible (water + polar organic) solvents with a minimum at around 40 mol %. These V^E_m values became less negative with increasing temperature, as is also common. Figures 1 and 3 show a sharp change in the density and V^E_m in the water-rich region. Negative values of V^E_m mean that there is a volume contraction and can be explained by the large difference in the molar specific volumes. Pal and Singh[14] concluded that the contraction in the volume is due to the ability of the OH group to form hydrogen bonds with water molecules and that the formation of the hydrogen bonds is enhanced by the presence of the ether oxygen (Buckley and Brochu[15]). The second interpretation given was that such a marked change in V^E_m might also be due to the accommodation of the nonaqueous molecules occupying in the structured water lattice a void space.

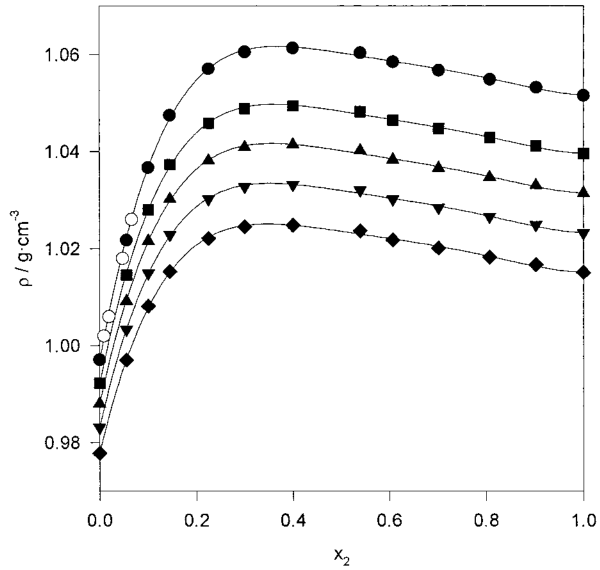

Figure 1. Densities of the water (1) + DGA (2) system at various temperatures: ●, 25 °C; ■, 40 °C; ▲, 50 °C; ▼, 60 °C; ◆, 70 °C; ○, 25 °C.[8]

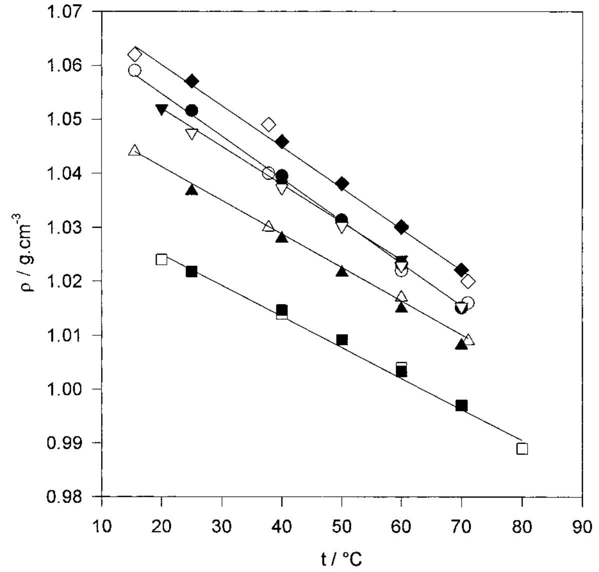

Figure 2. Comparison of the densities of the water (1) + DGA (2) system at various temperatures: ▼, 25.45 mass %; ●, 39.40 mass %; ◆, 50.06 mass. %; ■, 62.88 mass %; ▲, 100 mass % [▽, 25 mass %; ○, 40 mass %; ◇, 50 mass %; □, 60 mass %; △, 100 mass %].[5,6]

It is known that the number of cross-associated H bonds decreases with increasing temperature, which leads to a positive contribution to V^E_m. As a consequence, V^E_m values became more negative with a decrease in temperature.

In Figure 4, a comparison of the excess volumes of several alkanolamines shows that the addition of MDEA to water brings the largest increase in the excess volume,

Table 2. Redlich−Kister Equation Fitting Coefficients of the Excess Volumes (V^E_m/cm^3·mol^{-1}) for Water (1) + DGA (2) Mixtures at Various Temperatures

t/°C	a_0	a_1	a_2	a_3	a_4	a_5	a_6	σ
25	−5.9760	−0.5987	−1.8295	−0.4254	−1.4908	0.2886	0.1133	0.01
40	−5.7235	−0.5639	−1.7088	−0.3743	−1.3517	0.2954	0.1146	0.009
50	−5.6135	−0.5515	−1.6923	−0.3544	−1.3273	0.29617	0.1127	0.009
60	−6.3556	0.1963	−2.6087	0.35951	−2.2578	0.3123	0.1150	0.01
70	−3.7117	−0.6353	−1.5487	−0.1174	−1.0103	0.2397	0.0893	0.01

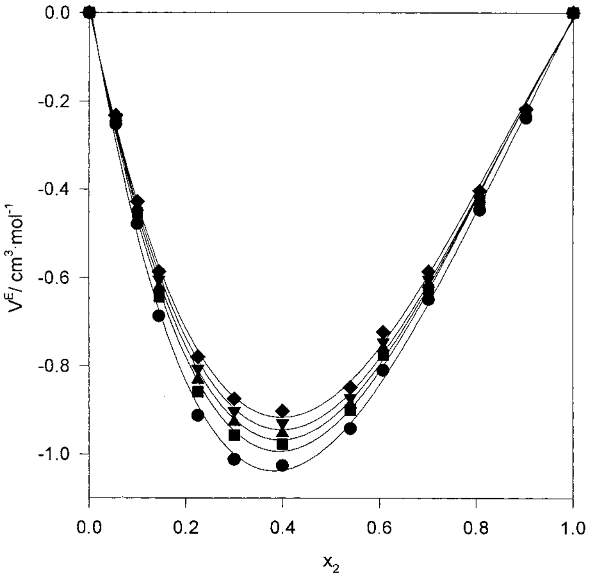

Figure 3. Excess molar volumes of the water (1) + DGA (2) system at various temperatures: ●, 25 °C; ■, 40 °C; ▲, 50 °C; ▼, 60 °C; ◆, 70 °C.

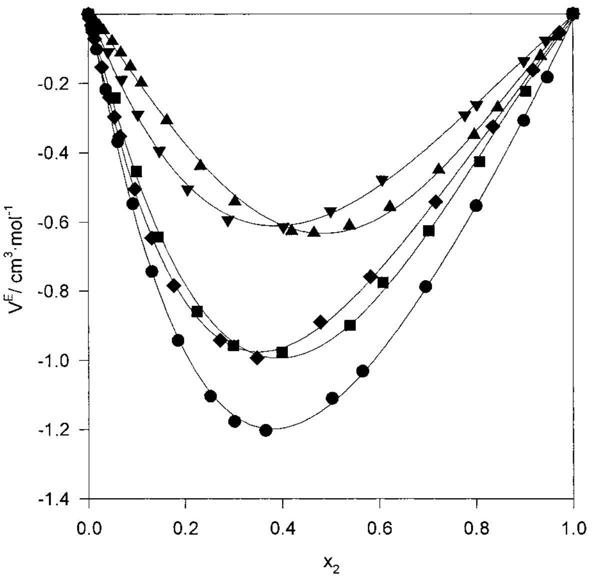

Figure 4. Excess molar volumes of various aqueous alkanolamine systems at 40 °C: ●, MDEA;[26] □, DGA; ◆, TEA;[20] ▲, MEA;[20] ▼, DEA.[20]

followed by DGA and triethanolamine (TEA) and then monoethanolamine (MEA) and diethanolamine (DEA). An interesting fact is that Chang et al.[16] using freezing point depression measurement and total pressure data found that, at 0 °C and 40 °C, MDEA and DGA solutions deviated the most from ideality.

A Redlich−Kister[17] relation was used to correlate the excess volume data.

$$V^E_m/\text{cm}^3\cdot\text{mol}^{-1} = x_1x_2\sum_{i=0}^{n} a_i(x_2 - x_1)^i \tag{4}$$

The coefficients and the standard deviation (σ) are presented in Table 2. It is well-known that the Redlich−Kister relation does not correlate well unsymmetrical curves of excess volumes (and viscosity deviations). The introduction of a number of skewing factors did not reduce the number of coefficients used. We thus present the correlation of the densities of the solutions at each temperature with the following polynomial:

$$\rho/\text{g}\cdot\text{cm}^{-3} = \sum_{k=0}^{n} a_k {x_2}^k \tag{5}$$

The coefficients and the standard deviation (σ) are presented in Table 3. Hepler[18] and Neal and Goring[19] recognized the usefulness of thermal expansion data for revealing correlations between the molecular structure of solutes and their effects on water structure. They suggested using the sign of the second derivative of the partial molar volume at infinite dilution of the solute with respect to the temperature ($d^2V^o_2/dT^2$) to classify solutes as "structure makers" or "structure breakers". A positive sign corresponds to a structure-making solute while a negative sign corresponds to a structure-breaking solute.

Both the partial molar volume of water at infinite dilution ($V^\infty{}_1$) in DGA and the partial molar volume of DGA at infinite dilution ($V^\infty{}_2$) in water were obtained using the method proposed by Maham et al.[19] The apparent molar volume of water ($V_{\phi,1}$) and the apparent molar volume of DGA in water ($V_{\phi,2}$) were first calculated as

$$V_{\phi,1} = V^o{}_1 + [V^E{}_m/(1 - x_2)] \tag{6}$$

and

$$V_{\phi,2} = V^o{}_2 + (V^E{}_m/x_2) \tag{7}$$

where $V^o{}_1$ and $V^o{}_2$ are the molar volumes of pure water and DGA, respectively. An analytical extrapolation of $V_{\phi,1}$ to $x_2 = 1$ led to $V^o{}_1$, and a similar extrapolation to $x_2 = 0$ led to $V^\infty{}_2$. Partial molar volumes at infinite dilution are listed in Table 4. $V^\infty{}_2$ values varied linearly with temperature (Figure 5). According to the criterion described above, the solute (DGA) would be considered as having no net effect on the structure of water. This is consistent with the conclusion of Maham et al.[20,21] concerning other alkanolamines (MEA, DEA, TEA, MDEA, and ethyldiethanolamine). All the values of the molar volumes at infinite dilution ($V^\infty{}_2$) were smaller than the corresponding molar values of pure DGA ($V^o{}_2$). The change in the excess volume can be explained by the DGA molecule fitting (partially) into the open, or empty, space in liquid water. This picture does not invoke either the structure-making or -breaking properties and is consistent with the observation that ($d^2V^\infty{}_2/dT^2$) values are approximately equal to zero.

Table 3. Coefficients of the Polynomial ρ/g·cm^{-3} = $\sum_0^n a_k x_2^k$ and the Standard Deviation for the Aqueous DGA Solutions at Various Temperatures

t/°C	a_0	a_1	a_2	a_3	a_4	a_5	σ
25	0.996 86	0.550 46	−1.775 92	2.747 19	−2.091 49	0.624 54	0.0004
40	0.992 11	0.495 93	−1.618 23	2.528 46	−1.946 75	0.588 08	0.0003
50	0.988 00	0.465 22	−1.527 52	2.399 47	−1.858 21	0.564 49	0.0003
60	0.983 15	0.441 24	−1.466 16	2.330 35	−1.825 46	0.560 18	0.0003
70	0.977 85	0.420 17	−1.414 82	2.276 8	−1.803 97	0.559 11	0.0003

Table 4. Partial Molar Volumes of DGA at Infinite Dilution in Water (V^{∞}_2) and of Water at Infinite Dilution in DGA (V^{∞}_1)

t/°C	V^{∞}_2/cm^3·mol^{-1}	V^{∞}_1/cm^3·mol^{-1}
25	96.2	15.5
40	97.6	18.2
50	98.4	18.3
60	99.0	18.4
70	99.9	18.5

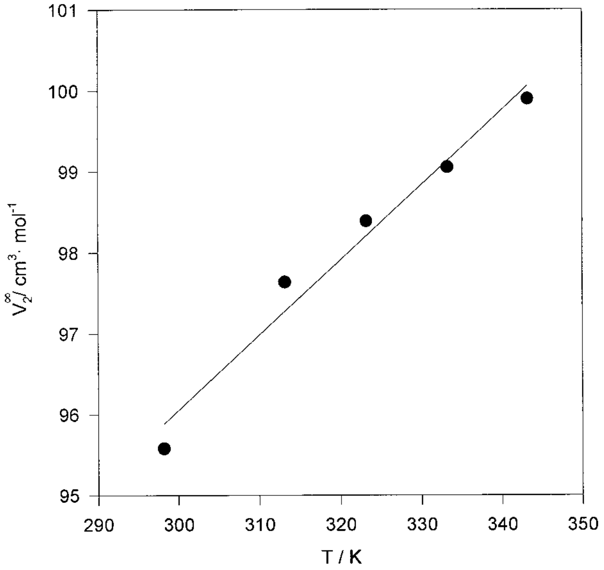

Figure 5. Partial molar volumes of DGA at infinite dilution in water at various temperatures: —, linear regression.

Having fewer values of V^E_m for x_2 near unity, accuracies of V^{∞}_1 may be lower. It is still clear that (dV^{∞}_1/dT) was positive. All values of V^{∞}_1 were smaller than the corresponding molar volumes of pure water (V^{o}_1) at the same temperature. As mentioned by Maham et al.,[19] this is consistent with the idea that the molar volume of pure water is the sum of the actual molecular volumes plus the "empty" volume that arises from the hydrogen-bonded open structure of water. Values of the apparent molar volumes of DGA in water ($V_{\phi,2}$) are shown in Figure 6 and increased with the increase in temperature. Each of the curves shown in Figure 6 has a minimum similar to that observed by Zhang et al.[22] for aqueous diethylethanolamine and dimethyldiethanolamine solutions.

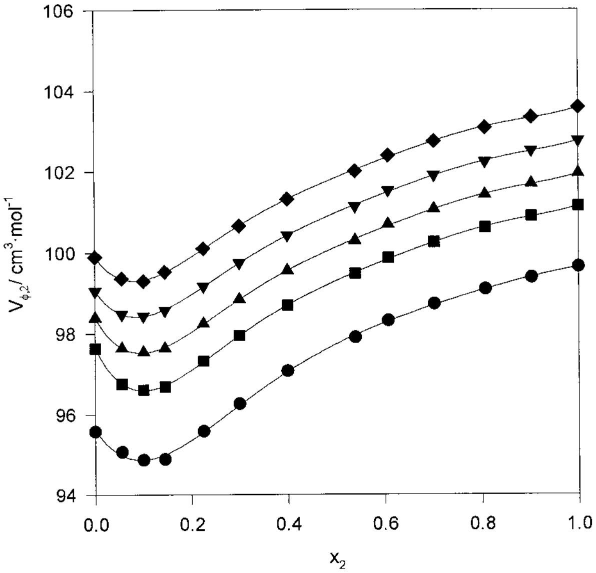

Figure 6. Apparent molar volumes of DGA in water at various temperatures: ●, 25 °C; ■, 40 °C; ▲, 50 °C; ▼, 60 °C; ◆, 70 °C.

Table 5. Experimental Viscosities for Water (1) + DGA (2) Mixtures at Various Temperatures

	η/mPa·s				
x_2	25 °C	40 °C	50 °C	60 °C	70 °C
0.0000	0.890	0.653	0.547	0.466	0.405
0.0552	2.400	1.452	1.160	0.948	0.789
0.1002	3.874	2.419	1.857	1.471	1.188
0.1447	6.344	3.715	2.737	2.098	1.661
0.2249	11.402	6.182	4.438	3.227	2.481
0.2997	16.210	8.343	5.773	4.197	3.149
0.3995	21.352	10.697	7.293	5.218	3.838
0.5392	25.203	12.276	8.273	5.849	4.308
0.6075	26.797	13.193	8.903	6.250	4.597
0.7021	27.716	13.654	9.182	6.493	4.781
0.8071	27.698	13.758	9.315	6.577	4.848
0.9024	27.403	13.654	9.257	6.592	4.866
1.0000	26.658	13.432	9.155	6.530	4.867

Experimentally measured viscosities of the binary solutions of aqueous DGA solutions at (25, 40, 50, 60, and 70) °C are listed in Table 5 and shown in Figure 7. Data published by Hikita et al.[8] were also added to Figure 7 for comparison. Curves for the system water + DGA are S-shaped with a maximum at 70 mol % (93 mass %). Aqueous MDEA solutions were also shown to have this S-shaped form.[23] Fort and Moore[24] and Liler and Kosanović[25] state that an S-shaped viscosity curve and the presence of a maximum indicate the formation of stable complexes. This might explain why, at 0 °C and 40 °C, Chang et al.[14] found that MDEA and DGA solutions deviated the most from ideality. Figure 7 shows a sharp increase in the viscosity of the mixture after the addition of DGA. The effect of the composition on viscosity decreases with an increase in temperature. The largest variation occurs at 25 °C. The viscosity at 70 mol % DGA was 31 times larger than that of pure water and 1.04 times that of pure DGA.

The viscosity of pure DGA is well represented by the Arrhenius equation

$$\eta/\text{mPa·s} = Ae^{E/RT} \tag{8}$$

where A is a system dependent constant, E the activation energy for viscous flow, R the gas constant, and T the temperature. The activation energy value regressed from the data was found to be equal to 33.7 kJ/mol. This value is higher than that of MEA (29.2 kJ/mol) and could be due to a stronger hydrogen bonding from the "free" hydrogen

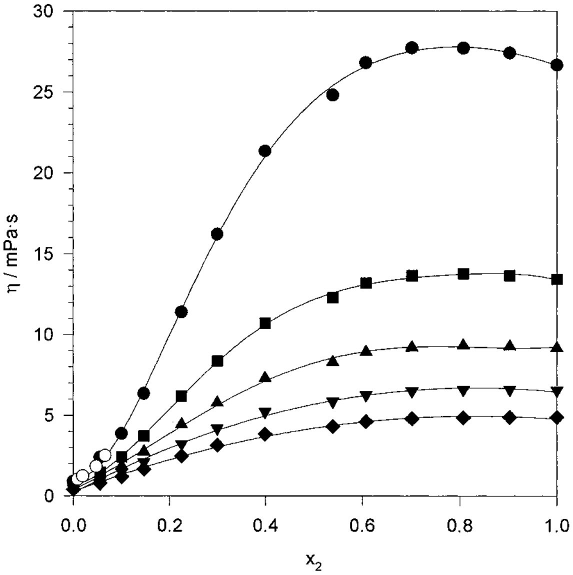

Figure 7. Viscosities of the water (1) + DGA (2) system at various temperatures: ●, 25 °C; ■, 40 °C; ▲, 50 °C; ▼, 60 °C; ◆, 70 °C; ○, 25 °C.[8]

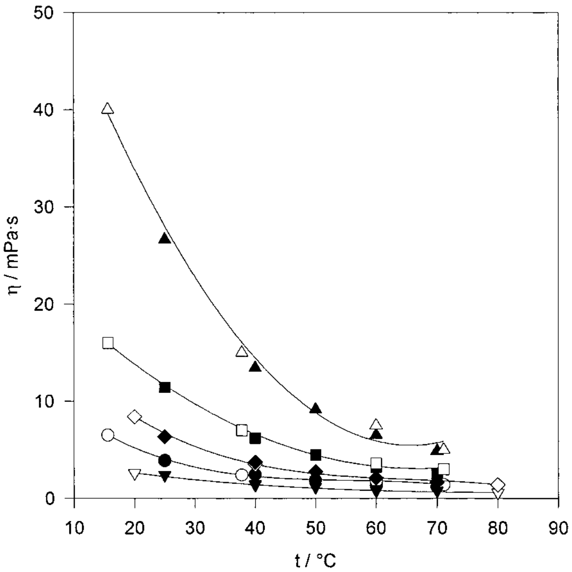

Figure 8. Comparison of the viscosities of the water (1) + DGA (2) system at various temperatures: ▼, 25.45 mass %; ●, 39.40 mass %; ◆, 50.06 mass %; ■, 62.88 mass. %; ▲, 100 mass %; [▽, 25 mass %; ○, 40 mass %; ◇, 50 mass %; □, 60 mass %; △, 100 mass %].[5,6]

in the presence of the ether oxygen in DGA. Figure 8 is a comparison between the values presented in this study and data published by the Jefferson Chemical Company and reported in Kohl and Nielsen[2] and Pacheco.[1] The activation energy for flow for DGA was closer to the activation energy of MDEA (38.0 kJ/ mol) than those of DEA (51.5 kJ/ mol) and TEA (50.2 kJ/ mol). The activation energies of flow for the mentioned alkanolamines were reported by Maham et al.[20] from different sources. Figure 9 displays the different viscosity curves of aqueous solutions of two primary alkanolamines (MEA and DGA), a secondary alkanolamine (DEA), and a tertiary alkanolamine (MDEA).

Experimental viscosity values of the binary mixtures were used to calculate the viscosity deviation, defined by

$$\Delta\eta = \eta - \eta_1 x_1 - \eta_2 x_2 \tag{9}$$

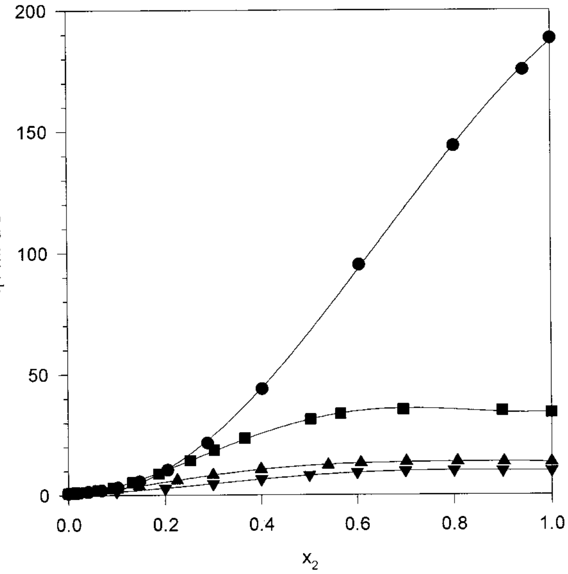

Figure 9. Comparison of the viscosities of various aqueous alkanolamine solutions at 40 °C: ●, DEA;[20] ■, MDEA;[25] ▲, DGA; ▼, MEA.[26]

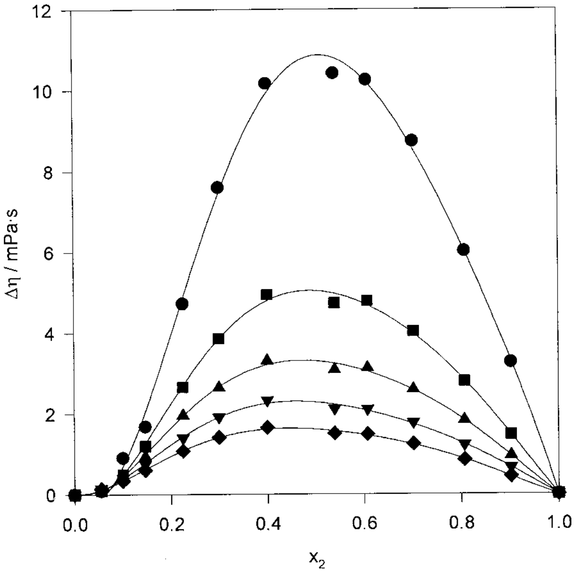

Figure 10. Viscosity deviations for the water (1) + DGA (2) system at various temperatures: ●, 25 °C; ■, 40 °C; ▲, 50 °C; ▼, 60 °C; ◆, 70 °C.

where η is the viscosity of the mixture and η_1 and η_2 are those of pure water and pure DGA, respectively; x_1 and x_2 are the mole fractions of the pure components. The viscosity deviation represents deviations from a rectilinear dependence of viscosity on mole fraction. Figure 10 displays the dependence of $\Delta\eta$ on the composition and temperature. Values of $\Delta\eta$ were all positive throughout the whole concentration range for all temperatures. The results are compared with those of aqueous MDEA[26] and aqueous MEA solutions[27] at 40 °C. Note that the viscosity deviations of dilute DGA have a value of zero up to a mole fraction of 0.1. Both MDEA and MEA viscosity deviations are negative in the water-rich region (<0.2 mole fraction) at all temperatures and then become positive. DEA viscosity deviations calculated from Teng et al.[26] were negative for all compositions at all temperatures except at 70 °C and 80 °C, where they became positive in the DEA-rich region.

Table 6. Redlich−Kister Equation Fitting Coefficients of the Viscosity Deviations ($\Delta\eta$/mPa·s) for Water (1) + DGA (2) Mixtures at Various Temperatures

t/°C	a_0	a_1	a_2	a_3	a_4	a_5	σ
25	43.468	−1.834	−30.983	60.124	−4.008	−46.496	0.21
40	20.139	−2.8504	−6.833	24.734	−11.870	−15.284	0.11
50	13.210	−2.713	−2.616	14.968	−9.192	−8.458	0.08
60	9.122	−2.764	−1.547	11.865	−5.682	−7.477	0.06
70	6.428	−2.001	−0.114	6.301	−5.065	−3.367	0.03

Table 7. Coefficients of the Polynomial ln η/mPa·s = ln η_0 + $\Sigma_1{}^n a_k x_2{}^k$and the Standard Deviation for the Water (1) + DGA (2) Mixtures at Various Temperatures[a]

t/°C	a_1	a_2	a_3	a_4	a_5	σ
25	19.148	−46.401	60.351	−40.877	11.178	0.02
40	16.559	−38.525	46.986	−29.189	7.190	0.009
50	15.523	−36.949	46.494	−29.908	7.655	0.010
60	14.603	−35.5447	46.154	−30.754	8.181	0.08
70	13.780	−34.399	46.251	−32.044	8.888	0.0002

[a] η_0 is the viscosity of pure water

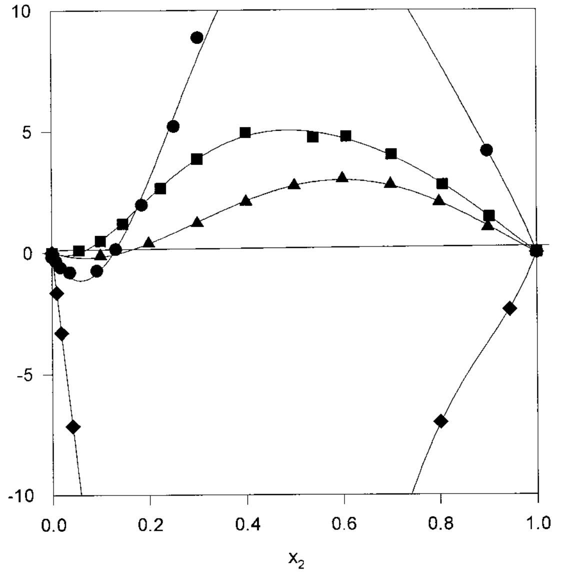

Figure 11. Comparison of the viscosity deviations of various aqueous alkanolamine solutions at 40 °C: ●, MDEA;[25] ■, DGA; ▲, MEA;[26] ◆, DEA.[25]

The calculated values of $\Delta\eta$ were correlated with a Redlich−Kister[16] relation:

$$\Delta\eta/\text{mPa}\cdot\text{s} = x_1 x_2 \sum_{i=0}^{n} a_i (x_2 - x_1)^i \tag{10}$$

The coefficients and the standard deviation (s) are presented in Table 6. We also present the correlation of the viscosities of the solutions at each temperature with the following polynomial:

$$\ln \eta/\text{mPa}\cdot\text{s} = \ln \eta_0 + \sum_{k=1}^{4} a_k x_2{}^k \tag{11}$$

where η is the viscosity of the binary solution, η_0 is the viscosity of pure water, and x is the mole fraction of DGA. The values of the polynomial coefficients a_k are listed in Table 7.

When shown in Figure 11, the viscosity deviations at 40 °C for aqueous MEA, DGA, and MDEA solutions seem to have a change in the sign of the viscosity deviation from negative to positive at low alkanolamine concentration. This is in sharp contrast to the appearance of the viscosity deviation curve for the secondary alkanolamine, DEA.

Conclusions

This paper reports experimental data for the densities and viscosities of the aqueous DGA solutions over a range of temperature from 25 °C to 70 °C. The calculated $V^E{}_m$ values for the aqueous DGA solutions were all negative at all temperatures and compositions. The criterion proposed by Hepler[17] suggests that the addition of DGA to water had no effect on its structure, a conclusion similar to that of Maham et al.[19,20] for MEA, DEA, TEA, MEA, and EDEA. The viscosity deviations $\Delta\eta$ for DGA + water were positive at all temperatures with few values close to zero in the water-rich region.

Aqueous DGA, MDEA,[24] and MEA solutions exhibited S-shaped viscosity curves and a change in the sign of the viscosity deviations from negative to positive in the water-rich region. Aqueous DEA viscosities[25] did not have an S-shaped curve but still exhibited a change in the sign of the viscosity deviations from negative to positive in the DEA-rich region.

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