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Densities and Viscosities for Binary Mixtures of *N*-Methyldiethanolamine + Triethylene Glycol Monomethyl Ether from 25 °C to 70 °C and *N*-Methyldiethanolamine + Ethanol Mixtures at 40 °C

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This paper reports the measured values of the density and viscosity of binary mixtures of *N*-methyldiethanolamine (MDEA) and triethylene glycol monomethyl ether (TEGMME) at five temperatures in the range 25 °C to 70 °C over the whole concentration range. We also report the density and viscosity of the binary mixture MDEA + ethanol at 40 °C. The results are compared with data for aqueous mixtures and other alkanolamines when these are available. The derived excess molar volumes and viscosity deviations were correlated as a function of composition. The Grunberg−Nissan interaction energy constants are also reported.

Introduction

Recent studies done on the absorption and desorption of acid gases (CO_2, H_2S) from natural gas, petroleum, and ammonia synthesis streams have shown that aqueous solutions of *N*-methyldiethanolamine (MDEA) can be used effectively for the selective removal of H_2S. MDEA has received increased use over other alkanolamines due to its slower reaction with CO_2, lower vapor pressure of solution (resulting in smaller losses of solvent in the regeneration section), and lower corrosivity. MDEA is also highly resistant to thermal and chemical degradation and as a tertiary amine has a high loading capacity (about 1.0 mol of CO_2/mol of amine). The main advantage of MDEA is its lower enthalpy of absorption, which leads to lower energy requirements for regeneration. Aqueous solutions of physical and chemical solvents have been proposed for selective absorption processes, since they possess the advantages of both physical solvents and chemical solvents.

Previously published data (Henni and Mather, 1995a) showed that mixtures of triethylene glycol monomethyl ether (50 mass %) + MDEA and methanol (50 mass %) + MDEA (Henni and Mather, 1995b) absorb more CO_2 than pure TEGMME (triethylene glycol monomethyl ether) or methanol alone for pressures <4000 kPa. The ternary systems could not be correlated by an equation of state (Peng and Robinson, 1976) even though the systems CO_2 + TEGMME and CO_2 + methanol were very well correlated by an equation of state. We were not able to give an explanation other than a possible formation of complexes or a presence of chemical reaction. Most researchers agree that MDEA does not react with CO_2 in nonaqueous solvents in the absence of water. Versteeg and van Swaaij (1988) state (without giving details) that the amount CO_2 absorbed in a mixture of MDEA + ethanol was nearly the same as the amount which can be physically dissolved in the solution and the difference can be completely accounted for by the presence of primary and secondary amine impurities. The densities and viscosities of aqueous solutions of TEGMME were recently published at temperatures from 25 °C to 80 °C (Henni et al., 1999). Excess volumes and viscosities at 35 °C were published by Pal and Singh (1996). Pal and Sharma (1999) recently published values of the excess volumes and viscosities of TEGMME and ethylene glycol dimethyl ether at 25 and 35 °C.

The present work was undertaken to measure the viscosities and densities of MDEA + TEGMME and MDEA + ethanol mixtures in order to find an explanation for the higher capacity of absorption of CO_2 in the absence of water. The measurement of the transport properties also provides the data necessary to model the rate of reaction and the mass-transfer rates in absorption and desorption operations. The density values are also needed in modeling gas absorption in binary and ternary systems (Wang et al., 1992).

Measurements of the density and viscosity of the MDEA + TEGMME system were performed at various temperatures (25, 40, 50, 60, and 70 °C) in order to cover the range of temperatures found in absorbers and regenerators. Measurements of the density and viscosity of the MDEA + ethanol system were performed at 40 °C in order to compare the values obtained with available data for MDEA + TEGMME and those for other alkanolamines (MEA + ethanol) available in the literature. The excess volumes, the deviation of the viscosity, and the Grunberg−Nissan constants (d_{12}) were derived.

Experimental Section

Triethylene glycol monomethyl ether (>97% pure) was purchased from Fluka. Anhydrous ethanol (>99.5% pure) and MDEA (>99% pure) were purchased from Aldrich. All chemicals were used without further purification.

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Table 1. Densities of TEGMME (1) + MDEA (2) Mixtures at Various Temperatures

	ρ/g·cm^{-3}				
x_2	25 °C	40 °C	50 °C	60 °C	70 °C
0.000	1.043 04	1.030 33	1.020 91	1.012 44	1.003 66
0.1082	1.041 95	1.029 49	1.020 17	1.011 77	1.003 08
0.1919	1.041 20	1.028 82	1.019 58	1.011 27	1.002 72
0.2870	1.040 36	1.028 07	1.018 99	1.010 77	1.002 33
0.3853	1.039 52	1.027 40	1.018 40	1.010 27	1.001 91
0.4584	1.038 86	1.026 90	1.018 00	1.010 06	1.001 71
0.5567	1.038 10	1.026 31	1.017 57	1.009 70	1.001 51
0.6728	1.037 35	1.025 73	1.017 15	1.009 39	1.001 30
0.7740	1.036 85	1.025 35	1.016 90	1.009 19	1.001 24
0.8860	1.036 41	1.025 10	1.016 74	1.009 20	1.001 35
1.0000	1.036 35	1.025 19	1.016 99	1.009 60	1.001 83

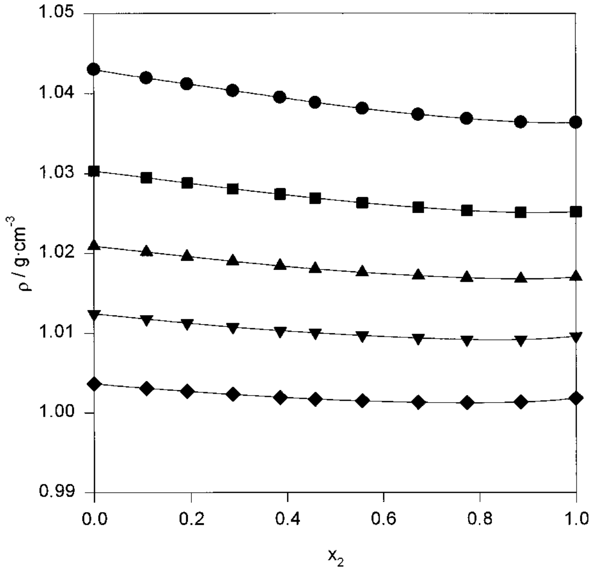

Figure 1. Densities of TEGMME (1) + MDEA (2) systems at various temperatures: (●) 25 °C; (■) 40 °C; (▲) 50 °C; (▼) 60 °C; (◆) 70 °C.

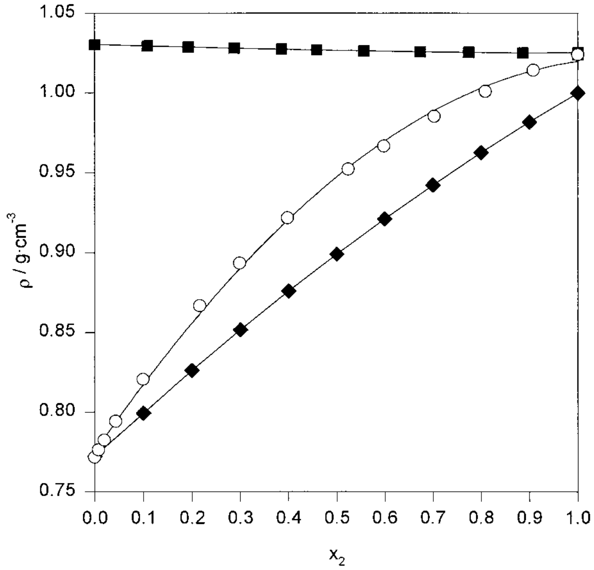

Figure 2. Densities of various systems at 40 °C: (◆) ethanol (1) + MEA (2)—Lee and Lin (1995); (■) TEGMME (1) + MDEA (2); (○) ethanol (1) + MDEA (2).

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. The densities of the binary mixtures were measured with an Anton Paar DMA-45 density meter. The accuracies of our densities are about ±(3 × 10^{-5} g·cm^{-3}). The temperature was controlled by means of a digital controller in a well-stirred water bath to better than ±0.01 °C as measured by a Guildline platinium resistance thermometer.

The density was 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 A and B were determined at each temperature using deionized water (Kell, 1975), and an air equation for the determination of the density of dry air was taken from the annual book of ASTM standards (ASTM, 1986). The values for pure ethanol at 40 °C were in good agreement (<0.08%) with the values reported by Lee and Lin (1995), who used a pycnometer and a water bath whose temperature was controlled at ±0.1 °C.

Viscosities were determined with two different viscometers to cover with precision (ASTM, 1986) the range of temperature from 25 °C to 70 °C. A 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 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. 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 (1990). Values of the viscosity of water were taken from Stokes and Mills (1965). 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 accuracy of the absolute viscosity to be >0.3%. Measured values of the viscosity of ethanol at 40 °C compared well (0.24%) with the values of Lee and Lin (1995), who used a Haake falling-ball viscometer.

Results and Discussion

Experimentally measured densities of the binary solutions of TEGMME + MDEA (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. The density curves show a quasi-linear decrease in values from pure TEGMME to pure MDEA. Figure 2 shows the data at 40 °C for the system MDEA + ethanol compared to those for the systems MDEA + TEGMME and MEA + ethanol. The shapes of the curves for ethanol and TEGMME are quite different. Curves for MDEA + ethanol are closer to

Table 2. Densities of Ethanol (1) + MDEA (2) Mixtures at 40 °C

x_2	ρ/g·cm^{-3}	x_2	ρ/g·cm^{-3}
0.0000	0.771 53	0.3977	0.921 86
0.0079	0.775 80	0.5237	0.952 10
0.0200	0.782 39	0.5978	0.966 76
0.0433	0.794 09	0.7028	0.985 11
0.0995	0.820 45	0.8085	1.000 83
0.2160	0.866 56	0.9080	1.014 399
0.2986	0.893 82	1.0000	1.025 19

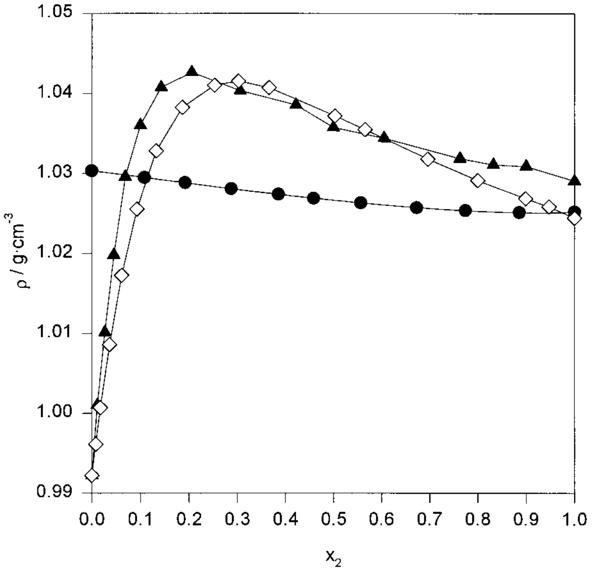

Figure 3. Densities of various systems at 40 °C: (●) TEGMME (1) + MDEA (2); (◇) water (1) + MDEA (2)—Teng et al. (1994); (▲) water (1) + TEGMME (2)—Henni et al. (1999).

the curves for the MEA + ethanol system than they are to those for the MDEA + TEGMME system. Density values for the mixture of ethanol + MDEA are presented in Table 2. Figure 3 shows the difference between the addition of MDEA or TEGMME to water and the addition of TEGMME or water to pure MDEA. The curves of MDEA + water and TEGMME + water are similar with the presence of a maximum while there is a linear change for the system MDEA + TEGMME. This may also be explained by the fact that there is almost no difference in the molar volumes of MDEA and TEGMME while that of ethanol is half that of MDEA or TEGMME.

The density values of the binary mixtures were used to calculate the excess molar volume V_m^E as

$$V_m^E = V_m - V_1^\circ x_1 - V_2^\circ x_2 \tag{3}$$

where V_m^E is the molar volume of the mixture and V_1° and V_2° are those of pure TEGMME and pure MDEA, respectively; x_1 and x_2 are the mole fractions of the pure components. Figure 4 displays the dependence of V_m^E on the composition at various temperatures. In all cases the V_m^E curves were positive with a maximum at 50 mol %. A Redlich−Kister (1948) relation was used to correlate the excess volume data.

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

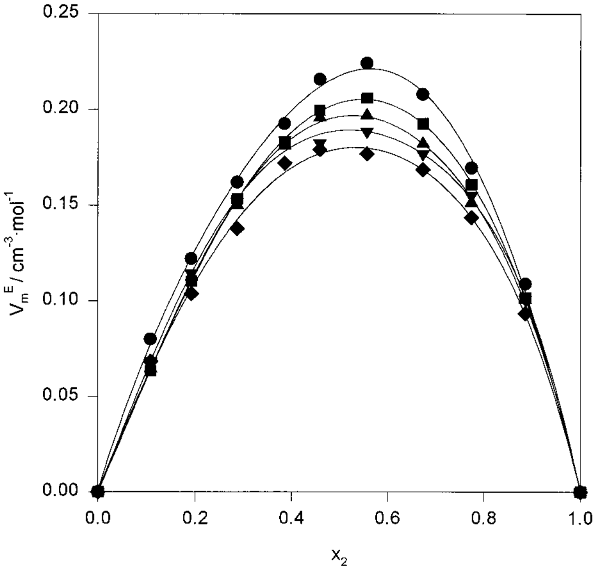

Figure 4. Excess molar volumes of TEGMME (1) + MDEA (2) systems at various temperatures: (●) 25 °C; (■) 40 °C; (▲) 50 °C; (▼) 60 °C; (◆) 70 °C.

Table 3. Redlich−Kister Equation Fitting Coefficients of the Excess Volumes (V_m^E/cm^3·mol^{-1}) for TEGMME (1) + MDEA (2) and Ethanol (1) + MDEA (2) Mixtures at Various Temperatures

t/°C	a_0	a_1	a_2	a_3	σ
		TEGMME + MDEA			
25	0.87098	0.2180	0.0786	−0.1103	0.004
40	0.8143	0.1513	0.0324	0.1103	0.001
50	0.7851	0.0778	0.0631	0.2019	0.007
60	0.7563	0.0452	0.1783	0.2514	0.003
70	0.7181	0.0785	0.1260	0.1395	0.008
		MDEA + Ethanol			
40	−2.81081	0.39085	0.25722	1.22918	0.008

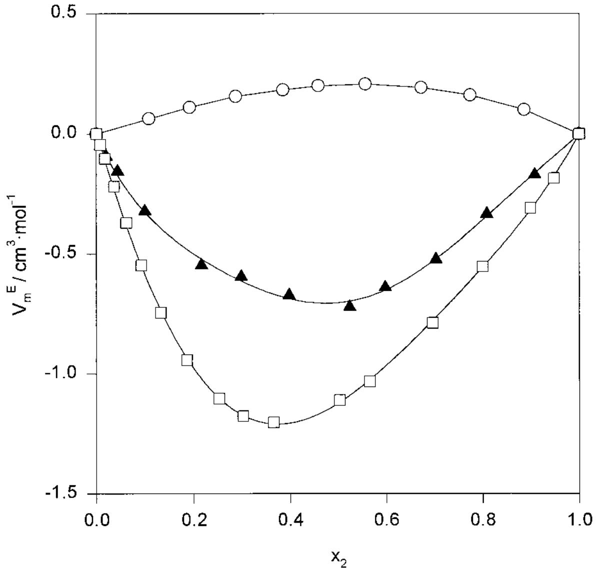

Figure 5. Comparison of the excess molar volumes of various systems at 40 °C: (○) TEGMME (1) + MDEA (2); (□) water (1) + MDEA (2)—Maham et al. (1995); (▲) ethanol (1) + MDEA (2).

The coefficients and the standard deviation s are presented in Table 3. Positive values of V_m^E mean that there is a volume expansion. Figure 5 shows the V_m^E values for ethanol + MDEA and water + MDEA (Maham et al., 1995) systems at 40 °C. This figure illustrates the effect of the addition of ethanol, water, and TEGMME to pure MDEA. The addition of MDEA to water brings the largest variation

Table 4. Experimental Viscosities for TEGMME (1) + MDEA (2) Mixtures at Various Temperatures

	η/mPa·s				
x_2	25 °C	40 °C	50 °C	60 °C	70 °C
0.0000	6.240	4.080	3.200	2.610	2.110
0.1004	6.741	4.491	3.445	2.749	2.265
0.2133	8.012	5.038	3.861	3.033	2.463
0.2964	9.103	5.882	4.307	3.304	2.648
0.3993	10.853	6.508	4.930	3.782	3.004
0.4991	13.350	7.732	5.674	4.421	3.366
0.5993	17.082	9.412	6.845	5.054	3.874
0.7001	22.811	12.027	8.383	6.156	4.626
0.8006	31.152	15.988	10.779	7.589	5.589
0.9002	50.170	22.231	14.355	9.852	7.054
1.0000	77.190	34.110	21.973	14.300	9.850

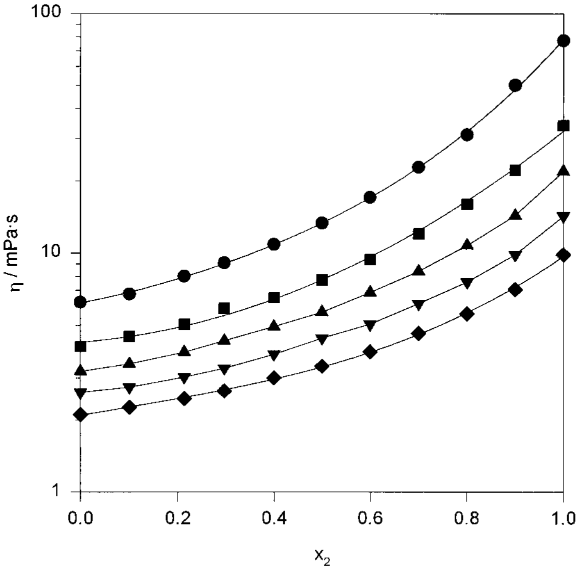

Figure 6. Viscosities of TEGMME (1) + MDEA (2) systems at various temperatures: (●) 25 °C; (■) 40 °C; (▲) 50 °C; (▼) 60 °C; (◆) 70 °C.

Table 5. Experimental Viscosities for Ethanol (1) + MDEA (2) Mixtures at 40 °C

x_2	η/mPa·s	x_2	η/mPa·s
0.0000	0.832	0.3977	4.196
0.0079	0.832	0.5237	7.050
0.0200	0.888	0.5978	9.003
0.0434	0.998	0.7028	12.919
0.0995	1.274	0.8085	17.788
0.2160	2.132	0.9080	23.875
0.2986	2.967	1.0000	34.109

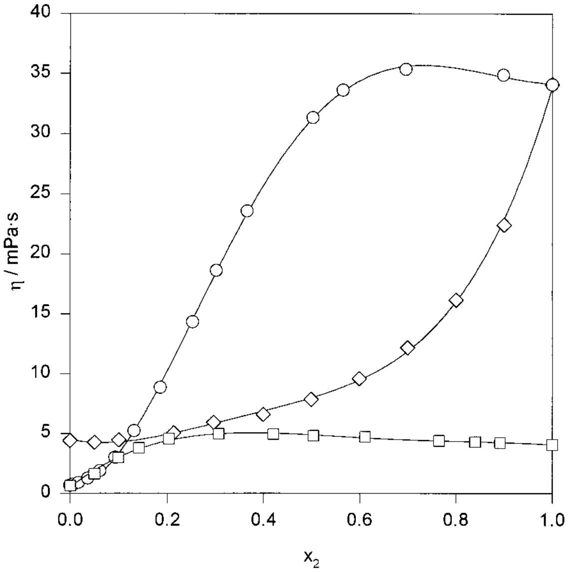

Figure 7. Comparison of the viscosities of various systems at 40 °C: (○) water (1) + MDEA (2)—Teng et al. (1994); (□) TEGMME (2) + Water (1)—Henni et al. (1999); (◇) TEGMME (1) + MDEA (2).

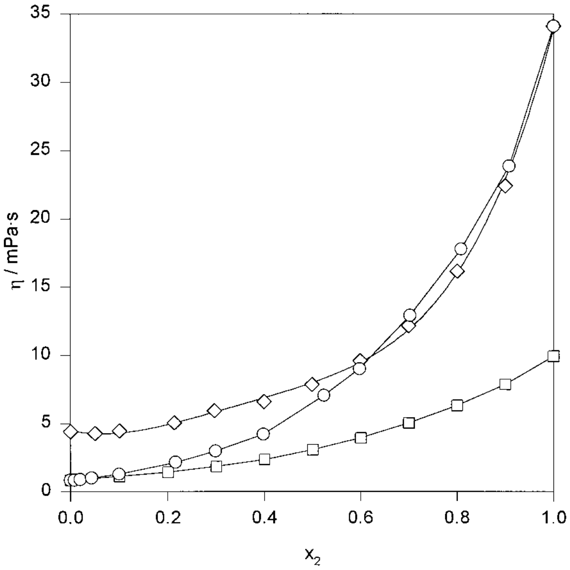

Figure 8. Comparison of the viscosities of various systems at 40 °C: (○) ethanol (1) + MDEA (2); (◇) TEGMME (1) + MDEA (2); (□) ethanol (1) + MEA (2)—Lee and Lin (1994).

in excess volume values followed by the variation due to the addition of MDEA in ethanol. Both water + MDEA and ethanol + MDEA mixtures have negative excess volumes while the mixtures TEGMME + MDEA have positive excess volumes. The negative values of the excess volumes at all mole fractions for ethanol + MDEA and water + MDEA systems can be explained by the large difference in the molar specific volumes. The MDEA + ethanol and MDEA + TEGMME systems are thus not expected to have among other properties the same capacity for gas absorption.

Experimentally measured viscosities of the binary solutions of TEGMME + MDEA at 25, 40, 50, 60, and 70 °C are listed in Table 4 and shown in Figure 6. The values for the ethanol + MDEA system are presented in Table 5. Figure 7 shows the changes in the value of η starting from pure MDEA and adding either water or TEGMME. We can also have an appreciation for the changes in viscosity due to the addition of TEGMME or MDEA to pure water. Both curves for the system TEGMME + water and MDEA + water systems are s-shaped with a maximum. Fort and Moore (1966) and Liler and Kosanović (1959) state that the presence of a maximum in the viscosity deviation curve indicates the formation of stable complexes. We have already suggested in a previous paper that TEGMME and water form complexes (Henni et al., 1999). The same conclusion should then be valid for the MDEA + water system; that is, there is formation of a stable complex. This statement was not previously mentioned in the literature and should be valuable in the thermodynamic and kinetic modeling of aqueous MDEA solutions.

Figure 8 illustrates the effect on the viscosity of the addition of MEA or MDEA to pure ethanol and the addition to pure MDEA of TEGMME or ethanol. Experimental viscosity values of the binary mixtures were used to

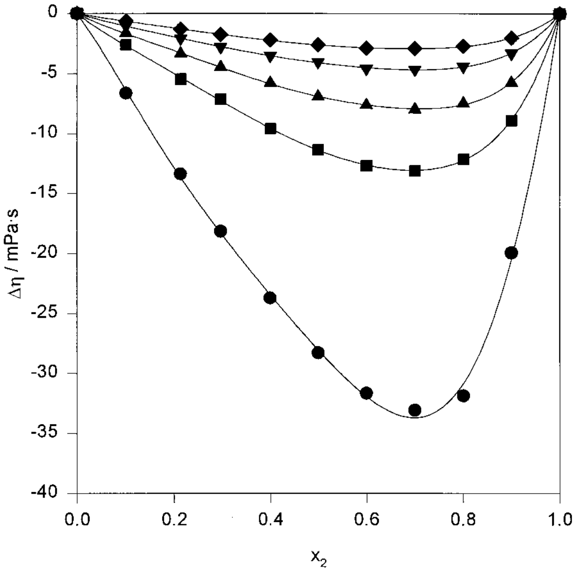

Figure 9. Viscosity deviations for TEGMME (1) + MDEA (2) systems at various temperatures: (●) 25 °C; (■) 40 °C; (▲) 50 °C; (▼) 60 °C; (◆) 70 °C.

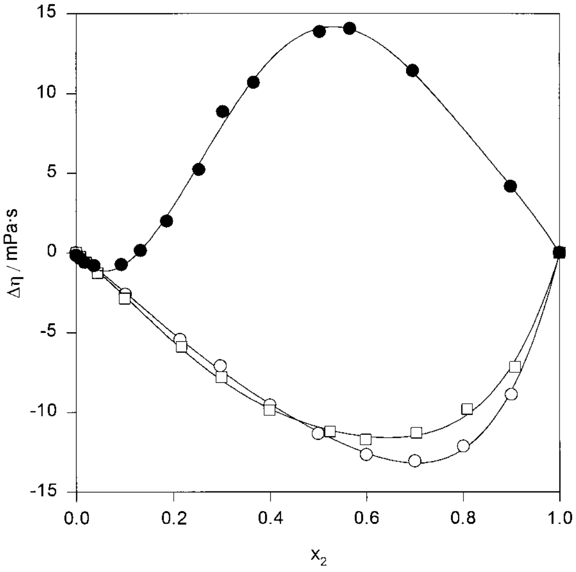

Figure 10. Comparison of the viscosity deviations for various systems at 40 °C: (●) water (1) + MDEA (2)—calculated from Teng et al. (1994); (□) ethanol (1) + MDEA (2); (○) TEGMME (1) + MDEA (2).

calculate the viscosity deviation, defined by

$$\Delta\eta = \eta - \eta_1 x_1 - \eta_2 x_2 \tag{5}$$

where η is the viscosity of the mixture and η_1 and η_2 are those of pure TEGMME and pure MDEA, 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 9 displays the dependence of $\Delta\eta$ on the composition and temperature. Values of $\Delta\eta$ were all negative throughout the whole concentration range for all temperatures. Figure 10 shows the values of the viscosity deviation ($\Delta\eta$) for the MDEA + ethanol system at 40 °C, where all values were negative for all compositions. The results are compared with those of TEGMME + MDEA and water + MDEA mixtures. Ethanol and TEGMME systems were negative in contrast; when MDEA is added to water, $\Delta\eta$ changes signs from negative to positive. At all temperatures (25 °C to 80 °C) presented by Teng et al. (1994) the viscosity deviations ($\Delta\eta$) of aqueous MDEA solutions were positive and changed sign around 0.15 mole fraction (54 mass %) for 40 °C and at a lower mole fraction for higher temperature. It is interesting to note that this range of concentration is of great industrial importance (20−50 mass %).

This fact may explain the limitation of the "N_2O analogy" as applied to the MDEA + water system (Kreulen et al., 1993). Absorption of N_2O in MDEA + water mixtures deviates the most from ideal mixing precisely in the same range of composition previously mentioned (i.e., 0.15 mole fraction at 25 °C, Kreulen et al., 1993). The measurements of the viscosity can be used as a tool to predict the limit of the validity of the "N_2O analogy" and other solubility models.

We can also explain with this observation the contradiction in the conclusions made by Chang et al. (1993) and Austgen et al. (1989a,b). Austgen et al. (1989) measured the total pressure of concentrated aqueous MDEA solutions at high temperatures, and Chang et al. measured the freezing point depression for dilute aqueous MDEA solutions at low temperature. The freezing point measurements implied totally different behavior than measurements of total pressures. It would be of interest to check if this trend is present in other aqueous alkanolamine systems.

The calculated values of $\Delta\eta$ were correlated with a Redlich−Kister (1948) relation:

$$\Delta\eta/\text{mPa·s} = x_1 x_2 \sum_{i=0}^{n} a_i (x_2 - x_1)^i \tag{6}$$

The coefficients and the standard deviation *s* are presented in Table 6. As is well-known, the Redlich−Kister relation does not correlate well unsymmetrical curves of excess volumes or viscosity deviations. As, in general, we needed eight coefficients to get a standard deviation as close as possible to the experimental error (0.003 mPa·s), we also present the correlation of the viscosities of the solutions

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

t/°C	a_0	a_1	a_2	a_3	a_4	a_5	a_6	a_7	σ
				TEGMME + MDEA					
25	−113.685	−82.668	−30.8874	11.0552	−202.195	−339.0863	261.533	463.799	0.08
40	−45.651	−30.5663	−10.202	−47.577	−47.598	147.551	300.0691	−164.715	0.06
50	−27.505	−18.411	−11.541	−19.419	−8.689	47.144	−9.710	−65.091	0.03
60	−16.260	−11.475	−10.664	11.681	9.797	−59.524	−18.799	49.093	0.04
70	−10.427	−7.387	−3.833	10.579	−2.970	−46.304	−2.2892	37.465	0.003
				Ethanol + MDEA					
40	−44.3806	−18.105	−10.816	−19.794	20.4365	33.641	−55.504	−56.468	0.08

Table 7. Coefficients of the Polynomial $\ln \eta/\mathrm{mPa{\cdot}s} = \ln \eta_0 + \sum_1^n a_k x_2^k$ and the Standard Deviation for the Binary Solutions of TEGMME (1) + MDEA (2) and Ethanol (1) + MDEA (2) Mixtures at Various Temperatures[a]

t/°C	a_1	a_2	a_3	a_4	σ
		TEGMME + MDEA			
25	0.835	1.531	−0.757	0.920	0.021
40	0.932	0.635	−0.211	0.766	0.016
40*	4.155	1.195	−3.721	2.073	0.023
50	0.503	2.360	−3.161	2.218	0.009
60	0.172	3.250	−4.371	2.646	0.00005
70	0.464	1.590	−2.004	1.485	0.00004
		Ethanol + MDEA			
40	4.155	1.195	−3.721	2.073	0.023

[a] η_0 is the viscosity of pure TEGMME or ethanol.

at each temperature with the following polynomial:

$$\ln \eta/\mathrm{mPa{\cdot}s} = \ln \eta_0 + \sum_{k=1}^{4} a_k x^k \tag{7}$$

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

According to Grunberg and Nissan (1949), the value of the binary mixture of components can be described in terms of the viscosities of the pure components and an interaction term as

$$\ln \eta = x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_1 x_2 d_{12} \tag{8}$$

where d_{12} is regarded as a measure of the strength of interactions between the mixing species. Values of d_{12} were negative for all temperatures and compositions and varied from −1.09 to −37.80 at 25 °C, from −0.98 to −20.22 at 70 °C for TEGMME + MDEA, and from −3.92 to −35.40 for ethanol + MDEA. There is a large variation in the value of d_{12} depending on the mole fraction. In the four systems studied (MDEA + water, MDEA + ethanol, MDEA + TEGMME, TEGMME + water), whenever the viscosity curves had an s-shape, the Grunberg and Nissan constants d_{12} were positive, and they were negative in the cases were the viscosity curves did not have an s-shape.

Conclusions

This paper reports experimental data for the densities and viscosities of the TEGMME + MDEA system over a range of temperature from 25 °C to 70 °C and for the system MDEA + ethanol at 40 °C. The calculated V_m^E values for the TEGMME + MDEA system were all positive at all temperatures and compositions. The system MDEA + ethanol did not show a similar trend, as the excess volumes for all compositions at 40 °C were all negative. Mixtures of ethanol + MDEA and TEGMME + MDEA are not thus expected to have similar gas absorption capacities. We should not expect mixtures of MDEA and any physical solvent to have the same behavior when it comes to absorption of gases (CO_2 for example).

The viscosity deviations $\Delta\eta$ for MDEA + TEGMME and MDEA + ethanol systems were negative. The viscosity deviations for the system MDEA + water at all temperatures (25 °C to 80 °C) were found to be negative at low MDEA mole fractions (up to 0.25) and positive at higher mole fractions.

The calculated Grunberg−Nissan constants for both systems were found to be all negative, but their values varied greatly with changes in mole fraction. When mixed with MDEA, ethanol and TEGMME lead to completely different density curve shapes while their viscosity curves were similar.

Finally, according to Fort and Moore's observation, the shape of the viscosity curves for TEGMME + water and MDEA + water systems and the presence of maxima indicate the probable formation of stable complexes in these mixtures (0.3 mole fraction for TEGMME + water and 0.7 mole fraction for MDEA + water). This remark and the change in sign of the viscosity deviation may be helpful in explaining the "anomalies" found in the measurement of the mass-transfer and kinetics rate of CO_2 in highly concentrated aqueous MDEA solutions. It may also explain the limitation of the "N_2O analogy" in the case of the MDEA + water system as the absorption of N_2O in MDEA + water mixtures deviates the most from ideal mixing precisely in this range of composition (Kreulen et al., 1993). It may also explain the contradiction in the conclusions reached by Austgen et al. (1989a,b) and Chang et al. (1993) in studying the behavior of aqueous MDEA at high and low MDEA concentration. The measurements of the viscosities of other alkanolamines can be used as a tool to predict the limit of the validity of the "N_2O analogy" and solubility models. According to Fort and Moore, the mixtures TEGMME + MDEA would not form a complex.

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