Density and Viscosity of Aqueous Blends of Three Alkanolamines: *N*-Methyldiethanolamine, Diethanolamine, and 2-Amino-2-methyl-1-propanol in the Range of (303 to 343) K

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Experimental values of the density and viscosity for aqueous solutions of three alkanolamines composed of 32.5 mass % *N*-methyldiethanolamine (MDEA) and 12.5 mass % diethanolamine (DEA) with 2, 4, 6, 8, and 10 mass % 2-amino-2-methyl-1-propanol (AMP) have been determined in the temperature range of (303.15 to 343.15) K. The experimental results of the density and viscosity are given as a function of both temperature and AMP concentration. In the range of temperature studied, the experimental density values of the aqueous blends of alkanolamines decrease as the AMP concentration increases while the viscosity values increase as the AMP concentration increases. Correlation equations were obtained to allow the calculation of density and viscosity for aqueous solutions of MDEA and DEA as a function of AMP concentration and temperature.

Introduction

Aqueous solutions of alkanolamines are widely used in industrial processes for sour gas purification (e.g., natural, refinery, and synthesis gas streams) primarily to eliminate acid gases, such as CO_2 and H_2S. The alkanolamines that have been widely utilized in such sweetening processes are monoethanolamine (MEA), diethanolamine (DEA), and *N*-methyldiethanolamine (MDEA) in aqueous solution of a single amine. An important advance in gas-treating technologies has been the availability of sterically hindered amines, such as 2-amino-2-methyl-1propanol (AMP), for CO_2 removal from industrial hydrocarbon-rich streams.[1,2]

In the past few years, aqueous solutions of mixed alkanolamines (a primary or secondary alkanolamine with a tertiary alkanolamine) have received increasing attention for the simultaneous removal of CO_2 in the presence of H_2S from different gas streams since those solutions combine the advantages of each individual amine with the aim to produce a considerable improvement in absorption capacity[2] as well as selective reactions and easier regeneration of the acid gas-loaded mixed amines solutions. Overall, their use results in great savings in energy requirements in the gas sweetening processes.[3]

Recent results from our laboratory on acid gas solubility have shown that selected aqueous solutions of blends of three alkanolamines: a secondary amine (DEA), a tertiary amine (MDEA), and a sterically hindered amine (AMP) are highly efficient for the absorption of CO_2 and H_2S.[4,5] Furthermore, such systems present high absorption selectivity toward H_2S in a large range of pressure and temperature values, which is a result of great interest for the removal of the latter from industrial and natural hydrocarbon gas streams.

It is well-known that, in order to select the most adequate concentration of the mixture of two or three alkanolamines that will efficiently eliminate the acid gases from a given hydrocarbon-rich gas stream, it is of the utmost importance to have a reliable body of solubility data for the acid gases of interest in a relative large range of temperature and pressure. To further know the absorption capacity and selectivity of the aqueous blends of amines of fixed concentration, it is also very important to have experimental results for their different physical properties in order to carry out the efficient design, simulation, and operation of acid gas treatment equipment.

The period 1992−2003 has been very rewarding for the experimental study of different thermophysical properties of aqueous solutions of alkanolamines blends that are of great interest for the purification of sour hydrocarbon gas streams. Hence, density data of aqueous solutions of mixtures of two alkanolamines have been reported in the literature as a function of concentration and temperature: MDEA with MEA,[6−8,12,14] MDEA with DEA,[9,10,12−14] MDEA with AMP,[11,13] MEA with AMP,[7,12,14] and DEA with AMP.[12−14] Viscosity data of some aqueous solutions of mixtures of two alkanolamines have also been reported in the literature, as a function of concentration and temperature: MDEA with MEA,[7,8,14,15] MDEA with DEA,[9,14,15] MDEA with AMP,[11] MEA with AMP,[7,14−16] DEA with AMP,[14−16] and DEA with MEA.[16]

Since the reports on the physicochemical behavior of aqueous blends of three alkanolamines are still scarce, neither density nor viscosity data are available in the open literature for the aqueous blends of the following three alkanolamines: MDEA, DEA, and AMP. Aqueous blends of these amines have been studied to determine their absorption capacity toward CO_2 and H_2S[4,5] in ranges of temperature and pressure that are of industrial importance. Considering that the concentration ratio at which these three amines may be studied is practically infinite, the aqueous blends of amines that were studied in the above-mentioned works are composed of 32.5 mass % MDEA + 12.5 mass % DEA with 4, 6, and 10 mass % of AMP.

Therefore, in the present work, we have studied the same blends of alkanolamines as above with 2, 4, 6, 8, and 10 mass

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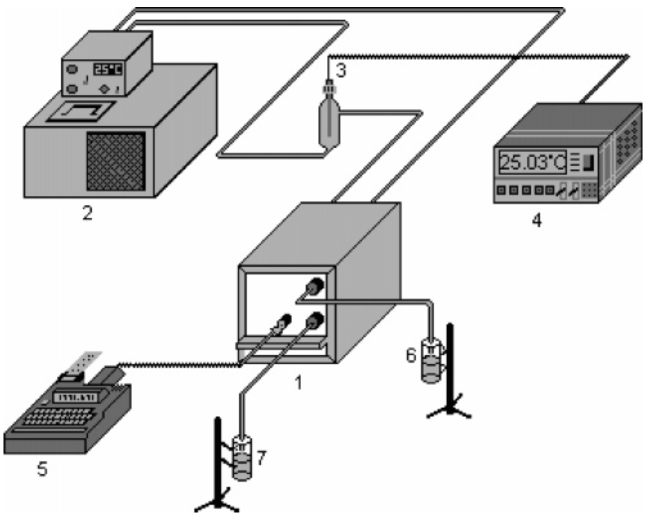

Figure 1. Diagram of the experimental setup to measure density values: 1, densimeter; 2, constant-temperature bath; 3, platinum resistance probe; 4, digital temperature indicator; 5, integrator and printer; 6, sample inlet; 7, sample outlet.

% of AMP to obtain experimental results of density and viscosity in the range (303.15 to 343.15) K under atmospheric pressure.

Experimental Section

***Materials*.** The aqueous solutions of alkanolamines were prepared with doubly distilled and deionized water with a conductivity of 17 $\mu\Omega$/cm. The samples of the alkanolamines were the same as those used in previous work.[4,5] MDEA and AMP were obtained from Aldrich Chemical Co. with a purity of 99+ mol % and 95 mol %, respectively; whereas DEA was obtained from J. T. Baker with a purity of 99.9 mol %. The alkanolamines were distilled under vacuum with a stream of nitrogen in order to remove any possible traces of moisture and other impurities. The ethylene glycol used in the calibration of the density meter was obtained from Merck with a purity of 99.5 mol %.

Chromatographic analyses were carried out on each sample of amine after distillation, and no impurities were found using a thermal conductivity detector with a capillary column Supelcowax 10 with a limit of detection of 0.05 mol %. The aqueous solutions of known concentration were prepared by weight in a Sartorius 2006 MP analytical balance with a precision and accuracy of ± 0.0001 g. Although the concentration values for the studied systems are reported throughout to the first decimal place, it should be noted that they are accurate within ± 0.002 mass %.

***Density*.** The density of the alkanolamine solutions was measured using a Sodev 03D vibrating tube density meter, which has been used in previous work.[17−20] The temperature was controlled by means of a Julabo F26 digital circulating bath within ± 0.002 K of the reported values and was measured with a digital thermometer Systemteknik, model AB, with a platinum resistance probe with a precision of ± 0.001 K. The readings from this thermometer were compared with those from a calibrated thermometer of Automatic System Laboratories (AΣL) whose accuracy is ± 0.005 K, traceable to the U.S. National Institute of Standards and Technology. The schematic diagram of the experimental arrangement is shown in Figure 1.

The density meter was calibrated at each studied temperature with doubly distilled and deionized water, whose density as a function of temperature was obtained from the literature with an accuracy[21] of $\pm 1 \times 10^{-3}$ kg·m^{-3} and dried ethylene glycol whose density as a function of temperature was obtained with an accuracy[22] of $\pm 1.3 \times 10^{-2}$ kg·m^{-3}. The working equation of the density meter is

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

where ρ is the density in kg·m^{-3} of the system under study, A and B are constants obtained from the calibration at a given temperature, and τ is the vibrating period of the density meter corresponding to the system under study.

For each temperature studied, the reported density results were obtained from the average period of vibration (τ), which was in turn obtained from at least 20 stable measurements. The final or combined uncertainty of the density results was evaluated through a complete statistical analysis on the propagation of uncertainties for all the known variables involved in the experimental work that considered the use of the so-called Student's *t* distribution. Therefore, the uncertainty reported in this work was always determined with a 95 % confidence for the best values of density experimentally determined. The estimated combined uncertainty for the experimental density values of this work is ± 0.01 % for the range of values obtained.

Viscosity. The kinematic viscosity was measured using three different Cannon−Fenske-type viscosimeters (sizes 50, 75, and 100) according to the specifications and operating instructions of the ASTM D 445 standard test method.[23] The viscosimeters were acquired from ACE Glass Inc., and they were calibrated and verified along this study using Cannon certified standard viscosity liquids S6 and N10. The standard fluids have kinematic viscosity values in the range 7.8 to 2.5×10^{-6} m^2·s^{-1} and 14.7 to 3.3×10^{-6} m^2·s^{-1}, respectively, in the studied range of temperature. All the viscosimeters were connected through short segments of flexible hose with glass tubes containing anhydrous calcium sulfate in order to avoid contact between the studied systems with CO_2 as well as humidity from the air. The viscosimeters were immersed in a Tamson TV4000 windowed constant-temperature bath, and the temperature was controlled within ± 0.005 K with the additional help for some temperatures of a Julabo FT401 coldfinger. Temperature was measured with the same digital thermometer used in the density study. Figure 2 shows the experimental arrangement used.

The efflux time was measured manually with a digital stopwatch whose precision is ± 0.01 s. With the efflux time, the kinematic viscosity was calculated from the equation

$$\nu = Ct \tag{2}$$

where ν is the kinematic viscosity in m^2·s^{-1}, C is a constant specific to each viscosimeter in m^2·s^{-2}, and t is the efflux time in s.

Table 1 presents the main working characteristics of the different viscosimeters used together with the calibration constants obtained in this work. For each studied temperature, the reported results were obtained from the average efflux time of at least 15 measurements taken on each freshly prepared aqueous blend of alkanolamines of known concentration. Replicate measurements of both density and viscosity were carried out on freshly prepared independent samples, and the results agreed within the estimated uncertainty of each property.

The absolute viscosity of the studied systems was calculated by multiplying the kinematic viscosity by the corresponding density value obtained in this work for each system at each

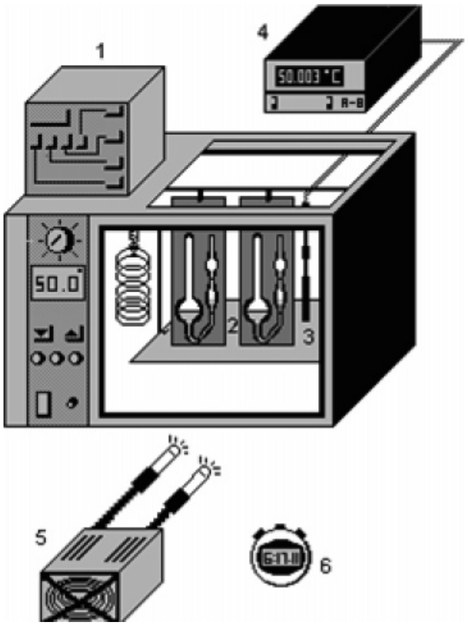

Figure 2. Diagram of the experimental setup to measure viscosity values: 1, constant-temperature bath; 2, viscosimeters; 3, platinum resistance probe; 4, digital temperature indicator; 5, light source; 6, stopwatch.

Table 1. Main Characteristics of the Viscosimeters Used in This Work

size	efflux time range/s	kinematic viscosity range/10^6·m^2·s^{-1}	$C/10^6$·m^2·s^{-2} (313.15 K)	$C/10^6$·m^2·s^{-2} (373.15 K)
50	200−1000	0.8−4.0	0.00354	0.00352
75	208−1044	1.6−8.0	0.00769	0.00766
100	212−1062	3.0−15.0	0.01413	0.01406

temperature considered. Considering the uncertainty in both the density and the kinematic viscosity values, the absolute viscosity values presents an uncertainty of ± 0.3 %.

Results and Discussion

To establish the accuracy of the experimental results obtained in this work, we determined the density for the three pure alkanolamines considered here and for the aqueous solutions of 30 mass % MDEA and 30 mass % DEA, all at 313.15, 323.15, and 333.15 K. Tables 2 and 3 contain the density results for the pure amines and the aqueous solutions, respectively. The tables also include data previously reported in the literature for comparison purposes. It can be observed that our results for MDEA present an absolute mean deviation with respect the reported data from four different authors[6,24−26] that guarantee an accuracy of ± 2 × 10^{-1} kg·m^{-3}; for DEA the comparison gives an absolute mean deviation with respect the data from three authors[24,25,27] that ensures an accuracy of ± 6 × 10^{-1} kg·m^{-3}, and for AMP the comparison gives for the lower deviation[7] an accuracy of ± 5 × 10^{-1} kg·m^{-3} in the studied temperature range. These values are of the same order of magnitude as the estimated combined uncertainty of the experimental density results given above. From the comparison of density results given in Table 3 we obtain similar results as above for each of the two aqueous solutions studied considering the reported data from three different authors for each comparison.

For the viscosity measurements, we have obtained results for an aqueous solution of 50 mass % MDEA at 303.15, 313.15, 323.15, and 333.15 K. This system has also been reported in the literature. Table 4 shows the comparison between our results and two different sets of dynamic viscosity data from the literature.[11,29] The comparison of our results with those from Al-Ghawas et al.[29] shows an absolute mean percent deviation of 0.06 % or ± 0.03 mPa·s, which is very close to the value of the combined uncertainty determined above for our experimental viscosity results.

Table 2. Comparison of Density (ρ/kg·m^{-3}) Values of Three Pure Alkanolamines at Different Temperatures

N-Methyldiethanolamine (MDEA)

T/K	this work	Wang et al.[24]	DiGuilio et al.[25]	Maham et al.[26]	Li and Shen[6]
313.15	1025.0	1024.6	1024.7	1024.45	1024.9
323.15	1017.4	1017.0	1017.3	1016.66	1017.4
333.15	1009.6	1009.3	1009.9	1009.00	1009.8
AAD/%[a]		0.036	0.023	0.062	0.010

Diethanolamine (DEA)

T/K	this work	Wang et al.[24]	DiGuilio et al.[25]	Maham et al.[27]
313.15	1084.7	1084.3	1084.6	1084.01
323.15	1077.4	1077.8	1078.1	1077.32
333.15	1070.3	1071.4	1071.6	1070.74
AAD/%[a]		0.059	0.065	0.037

2-Amino-2-methyl-1-propanol (AMP)

T/K	this work	Xu et al.[28]	Li and Lie[7]
313.15	917.2	921.1	917.9
323.15	909.2	913.4	909.6
333.15	900.7	905.5	901.1
AAD/%[a]		0.473	0.055

[a] AAD/% = average absolute deviation in percent.

Table 3. Comparison of Density (ρ/kg·m^{-3}) Values of Aqueous Solutions of *N*-Methyldiethanolamine (MDEA) and Diethanolamine (DEA) at Different Temperatures

MDEA 30 Mass %

T/K	this work	Al-Ghawas et al.[29]	Li and Shen[6]	Mandal et al.[14]
313.15	1017.1	1018.0	1017.1	1018.2
323.15	1011.5	1013.0	1011.6	1011.2
333.15	1004.1	1006.9	1005.7	
AAD/%[a]		0.171	0.156	0.074

DEA 30 Mass %

T/K	this work	Hsu and Li[12]	Rinker et al.[9]	Mandal et al.[14]
313.15	1026.0	1026.4	1024.7	1026.4
323.15	1021.3	1022.5	1019.3	
333.15	1014.4	1017.2	1017.0	
AAD/%[a]		0.144	0.187	0.118

[a] AAD/% = average absolute deviation in percent.

Table 4. Comparison of Dynamic Viscosity (η/mPa·s) Values of an Aqueous Solution of MDEA (50 mass %) at Different Temperatures

T/K	this work	Al-Ghawas et al.[29]	Welsh and Davis[11]
303.15	7.39	7.436	7.320
313.15	5.08	5.105	5.200
323.15	3.62	3.642	3.478
333.15	2.72	2.700	2.596
AAD/%[a]		0.061	3.03

[a] AAD/% = average absolute deviation in percent.

We have obtained in this work new experimental results for the density and viscosity of aqueous solutions of 32.5 mass % MDEA + 12.5 mass % DEA with AMP in concentrations of 2, 4, 6, 8, and 10 mass %. The studied temperatures are 303.15, 308.15, 313.15, 323.15, 333.15, and 343.15 K for density and 303.15, 313.15, 323.15, and 333.15 K for viscosity. The results

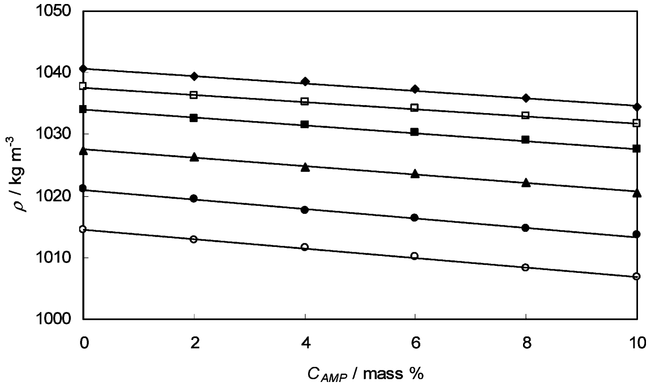

Figure 3. Experimental density results for the aqueous solution of 32.5 mass % MDEA + 12.5 mass % DEA as a function of AMP concentration (C_{AMP}/mass %), at different temperatures: ◆, 303.15 K; □, 308.15 K; ■, 313.15 K; ▲, 323.15 K; ●, 333.15 K; ○, 343.15 K. The full lines represent calculated values with eq 3.

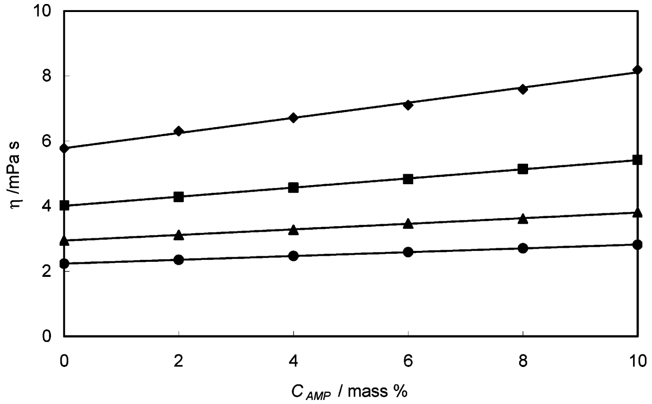

Figure 4. Experimental viscosity results for the aqueous solution of 32.5 mass % MDEA + 12.5 mass % DEA as a function of AMP concentration (C_{AMP}/mass %), at different temperatures: ◆, 303.15 K; ■, 313.15 K; ▲, 323.15 K; ●, 333.15 K The full lines represent calculated values with eq 4.

Table 5. Experimental Density (ρ) Results of This Work for the Aqueous Solution of 32.5 Mass % MDEA + 12.5 Mass % DEA with Varying Concentration of AMP (C_{AMP}) as a Function of Temperature

C_{AMP}	ρ/kg·m^{-3}					
mass %	303.15 K	308.15 K	313.15 K	323.15 K	333.15 K	343.15 K
0	1040.6	1037.7	1034.1	1027.6	1021.1	1014.5
2	1039.4	1036.4	1032.6	1026.3	1019.4	1012.8
4	1038.5	1035.3	1031.6	1024.6	1017.7	1011.6
6	1037.3	1034.2	1030.3	1023.6	1016.4	1010.1
8	1035.9	1033.0	1029.0	1022.1	1014.7	1008.3
10	1034.5	1031.8	1027.5	1020.6	1013.6	1006.9

Table 6. Absolute Viscosity Results of This Work for the Aqueous Solution of 32.5 Mass % MDEA + 12.5 Mass % DEA with Varying Concentration of AMP (C_{AMP}) as a Function of Temperature

C_{AMP}	η/mPa·s			
mass %	303.15 K	313.15 K	323.15 K	333.15 K
0	5.78	4.02	2.95	2.24
2	6.31	4.29	3.12	2.35
4	6.72	4.57	3.28	2.47
6	7.11	4.83	3.47	2.59
8	7.59	5.15	3.62	2.71
10	8.19	5.43	3.82	2.82

of density are given in Table 5, and the results of absolute viscosity are presented in Table 6.

Figures 3 and 4 show the experimental results of density and viscosity, respectively. It is possible to observe from both figures that the density and dynamic viscosity results of the aqueous alkanolamine solutions studied show a linear dependence with AMP concentration. Since the density of pure AMP is always lower than that corresponding to either DEA or MDEA in the range of temperature considered, then the density values of the studied solutions decrease as the AMP concentration increases. Figure 3 also shows the effect of temperature on the density values: for a given known concentration of the aqueous solutions their density decreases as the temperature increases.

It is not possible to carry out a direct comparison of our density results for the system MDEA + DEA + H_2O with those reported by Teng et al.[10] since their range of alkanolamines concentration does not include the concentration considered in this work.

Figure 4 shows that the addition of AMP to the aqueous solution of 32.5 mass % MDEA + 12.5 mass % DEA generates an increase on the viscosity of the resulting quaternary systems at given constant temperature. It can also be observed that, as expected, the viscosity for the different aqueous solutions of known concentration decreases as the temperature increases.

Using the experimental results obtained in this work, correlations were developed to allow the calculation of the density and viscosity of aqueous solutions of MDEA and DEA as a function of both AMP concentration and temperature. The equation used to correlate the 36 experimental density points is

$$\rho = A + BT \tag{3}$$

where ρ is the density in kg·m^{-3}, T is the temperature in K, and the parameters A and B are adjustable.

The equation chosen to correlate the 24 experimental viscosity points is

$$\ln \eta = A + B/T \tag{4}$$

where η is the viscosity in mPa·s, T is the temperature in K, and the parameters A and B are adjustable.

The adjustable parameters in eqs 3 and 4 were given a functionality with AMP concentration (C_{AMP}/mass %) as follows: $A = a_1 + a_2$ (C_{AMP}/mass %) and $B = b_1 + b_2$ (C_{AMP}/mass %). Table 7 shows the values of the optimized parameters a_1, a_2, b_1, and b_2 that were obtained from the density and viscosity results for the aqueous solutions of 32.5 mass % MDEA + 12.5 mass % DEA with varying concentration of AMP (0 to 10 mass %) in the studied range of temperature. It can be observed that the standard deviation of each fit is much smaller than the estimated uncertainty of the experimental results for both density and viscosity.

Although the concentration range considered in this work is limited to those values that are of interest for the industrial application of the studied systems we carried out the calculation

Table 7. Parameters of Equations 3 and 4 for the Correlation of the Experimental Results of Density and Absolute Viscosity for the Aqueous Solution of 32.5 Mass % MDEA + 12.5 Mass % DEA with AMP

property	a_1	a_2	b_1	b_2	T/K	standard deviation
ρ/kg·m^{-3}	1240.35	0.8417	−0.6584	−0.00472	303.15−343.15	0.04 kg·m^{-3}
η/mPa·s	−9.065	−0.0895	3281.14	37.33	303.15−333.15	0.01 mPa·s

Table 8. Excess Molar Volume (V^{E}) of the Aqueous Solution of 32.5 Mass % MDEA + 12.5 Mass % DEA with Varying Concentrations of AMP as a Function of Temperature

C_{AMP}	$10^4 V^{E}$/m^3·kmol^{-1}					
mass %	303.15 K	308.15 K	313.15 K	323.15 K	333.15 K	343.15 K
0	−5.9	−5.9	−5.7	−5.4	−5.4	−5.3
2	−6.2	−6.2	−6.0	−5.8	−5.7	−5.5
4	−6.6	−6.5	−6.3	−6.0	−5.9	−5.9
6	−6.9	−6.8	−6.6	−6.4	−6.2	−6.2
8	−7.1	−7.1	−6.9	−6.6	−6.4	−6.3
10	−7.3	−7.3	−7.1	−6.8	−6.7	−6.6

of the molar excess volume (V^{E}) using experimental density results for the multicomponent mixtures according with the following equation:

$$V^{E} = x_1 PM_1\left(\frac{1}{\rho_M} - \frac{1}{\rho_1}\right) + x_2 PM_2\left(\frac{1}{\rho_M} - \frac{1}{\rho_2}\right) + x_3 PM_3\left(\frac{1}{\rho_M} - \frac{1}{\rho_3}\right) + x_4 PM_4\left(\frac{1}{\rho_M} - \frac{1}{\rho_4}\right) \quad (5)$$

where x_i is the mole fraction of component i, PM_i is the relative molar mass of component i, ρ_i is the density of pure compound i, and ρ_M is the density of the aqueous blend of alkanolamines of known concentration.

Table 8 contains the experimentally obtained values of the molar excess volume for each mixture studied as a function of temperature in the range (303.15 to 343.15) K. It is observed that the V^{E} values are all negative which are congruent with the known behavior for similar binary and ternary systems of water + alkanolamines.[7,12−14]

The total number of V^{E} values is not large enough as to use a correlating equation such as the well-known Redlich−Kister polynomial equation which would require for the quaternary mixtures here considered at least 36 fitting parameters in order to consider both the concentration and temperature functionalities.[13]

The results here presented are part of a large body of experimental information on physical chemical properties of aqueous solutions of blends of two and three alkanolamines that have been integrated over a period of several years for the design, operation, and optimization of gas sweetening processes to achieve higher efficiency. Therefore, previous and present experimental results from our laboratory will be useful to test physicochemical models since commercially available process simulators do not have the capability to deal with aqueous solutions of blends of three alkanolamines because they do not carry databases on the thermophysical and phase equilibria behavior for such systems.

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Note Added after ASAP Publication

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