

Density and Excess Properties of Aqueous *N*-Methyldiethanolamine Solutions from (298.15 to 338.15) K

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Densities (ρ) of pure and aqueous *N*-methyldiethanolamine (MDEA) solutions ($w_{\text{MDEA}} = 0.3228$ or $4 \text{ mol}\cdot\text{kg}^{-1}$ and $w_{\text{MDEA}} = 0.4880$ or $8 \text{ mol}\cdot\text{kg}^{-1}$) at (298.15 to 338.15) K were measured at atmospheric pressure and also correlated. The coefficients of thermal expansion (α_p) values were calculated from the experimental density data for the investigated compositions, using an empirical correlation. The excess molar volumes of aqueous MDEA solutions for mole fractions (0.0128 to 0.7531) were calculated from experimental densities at temperatures (298.15 to 338.15) K and correlated with the Redlich–Kister equation. The partial molar volumes of MDEA in water at infinite dilution and the change in volume were also determined using Redlich–Kister coefficients.

Introduction

Aqueous solutions of alkanolamine are of great interest as chemical absorbents for the removal of carbon dioxide (CO_2) and hydrogen sulfide (H_2S) from sour natural gas. A wide variety of alkanolamines such as monoethanolamine (MEA), diethanolamine (DEA), 2-amino-2-methyl-1 propanol (AMP), *N*-methyldiethanolamine (MDEA), and their blends have been used in gas purifications, oil refineries, and petroleum industries. Each of the alkanolamines offer distinct advantages for treating specific problems. However, the use of MDEA is of utmost importance due to its high loading capacity ($\approx 44 \text{ g}$ of CO_2 per mol of the amine) in the absence of hydrogen sulfide. In connection with the industrial treatment of acid gases, the derived volumetric properties such as excess molar volume and partial molar volume at infinite dilution from density data provide a useful basis for understanding the molecular interactions of solute and solvent in mixtures.^{1,2} An extensive literature review for the determination of densities and excess properties of pure and aqueous MDEA solutions has been summarized in Table 1.

In this work, new experimental densities of aqueous MDEA solutions have been determined over a wide range of compositions at temperatures (298.15 to 338.15) K. Excess molar volumes of (MDEA + water) for mole fractions (0.0128 to 0.7531) are calculated from the measured density data and correlated with the Redlich–Kister-type equation. Partial molar volumes of MDEA in water at infinite dilution and the change in volume are also determined with Redlich–Kister coefficients. Thermal expansion coefficients of all the considered systems are calculated from their experimental densities, using an empirical correlation.

Experimental Section

A digital vibrating glass U-tube densimeter (DMA5000, Anton-Paar) was used to measure the densities of water, MDEA, and (MDEA + water). The densimeter was calibrated before and after all the density measurements using Millipore quality

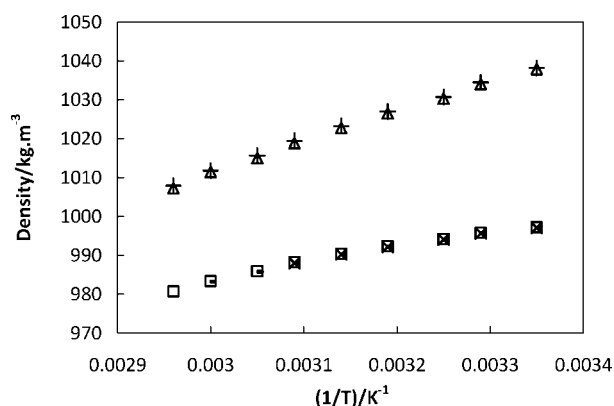


Figure 1. Comparison of densities for water: □, this work; ×, Alvarez et al.¹⁶ (2006); –, Al-Ghawaz et al.³ (1989). For MDEA: +, this work; Δ, Bernal-Garcá et al.¹³ (2003).

water. The accuracy of all density measurements was found to be better than $\pm 0.003 \text{ kg}\cdot\text{m}^{-3}$. A built-in platinum resistance thermometer was used to determine temperature with an accuracy of $\pm 0.01 \text{ K}$. The density of all samples were measured after achieving thermal equilibrium at slow mode with successive increments of 5 K at temperatures (298.15 to 338.15) K.

Chemicals. *N*-Methyldiethanolamine (MDEA) (Catalogue no. 805851, CAS no. 105-59-9, Batch no. S4605451) was supplied by Merck with a stated purity of 99.5 % (GC, area %) and used without further purification. The binary mixtures of MDEA were prepared gravimetrically with a precision of $\pm 0.0001 \text{ g}$, using an analytical balance (Mettler Toledo, model AS120S). The possible error in the mole fraction calculations was estimated to be around ± 0.0001 . All the measurements for each sample were performed in duplicate, and the values are reported as an average.

Results and Discussion

To establish the accuracy of densimeter calibration, the density of bidistilled water was determined and compared with the reported values of Al-Ghawaz et al.³ and Alvarez et al.,¹⁶ which are found to be within the average absolute percentage

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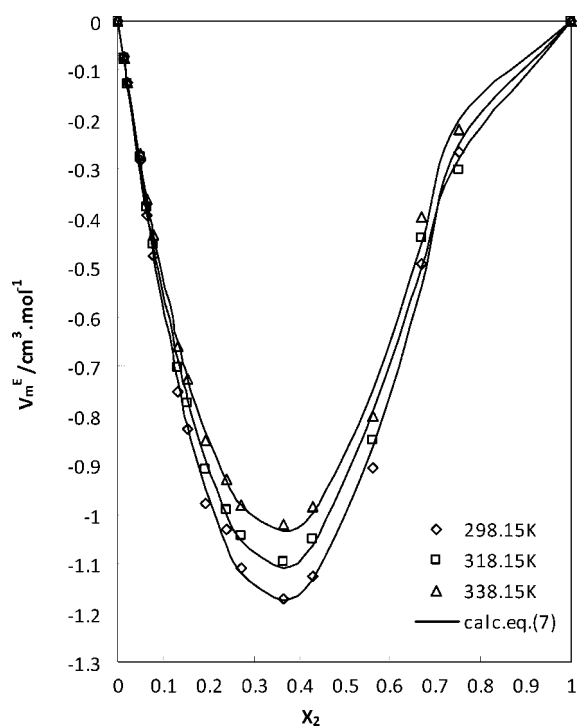
Table 1. Literature Review for the Determination of Physical Properties of Pure and Aqueous MDEA Solutions at Atmospheric Pressure

year	composition	T/K	ref	apparatus
Density				
1989	w = (0.1, 0.2, 0.3, 0.4, 0.5)	(288.15 to 333.15)	3	Gay-Lussac Pycnometer
1992	Pure	(303.1 to 353.15)	4	Gay-Lussac pycnometer
1992	Pure	(292.85 to 361.35)	5	Pycnometer
1992	Pure	(296.3 to 470.9)	6	High pressure pycnometer
1994	w = (0.2, 0.3)	(303.15 to 333.15)	7	Gay-Lussac pycnometer
1994	w = (0.1, 0.2, 0.3, 0.4, 0.5)	(293.15 to 373.15)	8	Gay-Lussac pycnometers
1995	w = 0.5	(283.15 to 333.15)	9	Gay-Lussac pycnometer
2000	Pure	(298.15 to 343.15)	10	DMA 45 density meter (Anton Paar)
2001	Pure	(303.15 to 333.15)	11	DMA 45 density meter (Anton Paar)
2003	w = 0.3	(293.15 to 323.15)	12	Gay-Lussac pycnometer
2006	Pure & w = (0.1, 0.2, 0.3)	(288, 313, 333)	14	Gay-Lussac pycnometer
2006	Pure & w = (0.3, 0.5)	(313.15 to 333.15)	15	Sodev 03D vibrating tube density meter
Density and Volumetric Properties				
1995	Pure & x ₂ = (0.0079 to 0.9475)	(298 to 353)	17	DMA 45 density meter (Anton Paar)
2000	Pure & x ₂ = (0.0050 to 0.9800)	(298, 308, 318)	1	DMA 45 density meter (Anton Paar)
2003	Pure & x ₂ = (0.0168 to 0.7430)	(283.15 to 348.15)	13	DMA 5000 vibrating U-tube densimeter (Anton Paar)

Table 2. Experimental Densities of Water, MDEA, and Binary Mixtures of MDEA + Water

T/K	$\rho/\text{kg}\cdot\text{m}^{-3}$			
	water	MDEA	MDEA + water	
			w _{MDEA} = 0.3228	w _{MDEA} = 0.4880
298.15	997.210	1038.224	1027.273	1041.371
303.15	995.805	1034.493	1024.819	1038.162
308.15	994.183	1030.749	1022.162	1034.938
313.15	992.364	1026.993	1019.437	1031.741
318.15	990.359	1023.255	1016.541	1028.354
323.15	988.180	1019.469	1013.629	1024.827
328.15	985.837	1015.645	1010.471	1021.382
333.15	983.338	1011.796	1007.394	1017.754
338.15	980.692	1007.921	1003.915	1014.175
100AAD	5.35	0.11	3.31	5.64

deviation of 0.017 % and 0.016 %, respectively. Experimental density values of water, MDEA, and (MDEA + water) with compositions of ($w = 0.3228$ and $w = 0.4880$) at atmospheric pressure along with average absolute percentage deviations have

**Figure 2.** Excess molar volumes of (MDEA + water) mixtures at different mole fractions correlated with the Redlich–Kister equation.

been presented in Table 2. The comparison of measured densities for water and pure MDEA between this work and the available literature values are shown in Figure 1. The influence of temperature on the density of water, MDEA, and all the compositions of binary mixtures of MDEA + water was found to be linear and correlated using

$$\rho = A_0 + A_1 T \quad (1)$$

where A_0 and A_1 are the fitting parameters which were estimated using a method of least-squares and presented in Table 3, along with the values of standard deviations (SD). The coefficients of thermal expansion values for the systems studied in this work are calculated from their experimental density data using

$$\alpha_p = -1/\rho(\partial\rho/\partial T)_p = -A_1/(A_0 + A_1 T) \quad (2)$$

where α_p is the coefficient of thermal expansion; ρ is the density; T is the temperature; and A_0 and A_1 are correlation coefficients taken from eq 1 by fitting experimental density data. It can be observed from Table 4 that the change in coefficients of thermal expansion values is not significant and the variation of volume expansion of the systems studied in the present work could be considered as independent of temperature. However, the values of α_p varied from $(4.40 \text{ to } 7.32) \cdot 10^{-4} \text{ K}^{-1}$, with the increase in composition of aqueous MDEA solutions (0.0128 to 0.7531). Excess molar volume of solution is a thermodynamic property and is defined as the amount of volume which is in excess to that of an ideal solution at the same conditions of composition, temperature, and pressure.

$$V_m^E = V_m - V_m^{\text{ideal}} \quad (3)$$

where V_m and V_m^{ideal} are the molar volumes of solution and pure components, respectively. Excess molar volumes of aqueous MDEA solutions with mole fractions (0.0128 to 0.7531) were calculated using eq 4 which was obtained after rearranging eq 3 using mole fractions, molar masses, and densities to represent the volumes of solution and pure components.

$$V_m^E/\text{cm}^3 \cdot \text{mol}^{-1} = \left[x_2 M_2 \left(\frac{1}{\rho} + \frac{1}{\rho_2} \right) + (1 - x_2) M_1 \left(\frac{1}{\rho} - \frac{1}{\rho_2} \right) \right] \cdot 1000 \quad (4)$$

where V_m^E is the excess molar volume; M_1 and M_2 are molar masses of water and MDEA, respectively; x_2 is the mole fraction of MDEA; ρ is the density of aqueous MDEA solutions; and ρ_1 and ρ_2 are densities of bidistilled water and pure MDEA.

Table 3. Fitting Parameters of Equation 1 to Correlate the Density of Water, MDEA, and Binary Mixtures of MDEA + Water along with Standard Deviations (SD)

		$\rho/\text{kg}\cdot\text{m}^{-3}$									
		MDEA (2) + water (1)									
		w_{MDEA}		x_2							
T/K	water	MDEA	0.3228	0.4880	0.0128	0.0212	0.0498	0.0645	0.0799	0.1332	0.1557
A_0	1122	1264	1202	1245	1137	1147	1179	1197	1210	1248	1257
A_1	-0.414	-0.757	-0.583	-0.680	-0.442	-0.462	-0.529	-0.571	-0.600	-0.688	-0.707
SD	0.7281	0.0663	0.4871	0.6166	0.6022	0.4749	0.6825	0.4247	0.4188	0.4000	0.4673
		x_2									
		0.1944	0.2407	0.2725	0.366	0.4302	0.5642	0.6687	0.7531		
A_0		1272	1275	1280	1287	1285	1278	1271	1267		
A_1		-0.745	-0.749	-0.763	-0.786	-0.785	-0.774	-0.769	-0.761		
SD		0.3940	0.3618	0.2337	0.1748	0.2501	0.2579	0.1794	0.5161		

Table 4. Coefficients of Thermal Expansion of Water, MDEA, and Binary Mixtures of MDEA + Water Using Equation 2

		$\alpha_p \cdot 10^{-4}/\text{K}^{-1}$									
		MDEA (2) + water (1)									
		w_{MDEA}		x_2							
T/K	water	MDEA	0.3228	0.4880	0.0128	0.0212	0.0498	0.0645	0.0799	0.1332	
298.15	4.15	7.29	5.67	6.52	4.40	4.58	5.18	5.56	5.82	6.60	
303.15	4.16	7.31	5.69	6.55	4.41	4.59	5.19	5.58	5.84	6.62	
308.15	4.17	7.34	5.70	6.57	4.42	4.60	5.21	5.59	5.85	6.64	
313.15	4.18	7.37	5.72	6.59	4.43	4.61	5.22	5.61	5.87	6.66	
318.15	4.18	7.39	5.74	6.61	4.44	4.62	5.23	5.62	5.89	6.69	
323.15	4.19	7.42	5.75	6.63	4.45	4.63	5.25	5.64	5.90	6.71	
328.15	4.20	7.45	5.77	6.65	4.46	4.64	5.26	5.66	5.92	6.73	
333.15	4.21	7.48	5.79	6.68	4.47	4.65	5.28	5.67	5.94	6.75	
338.15	4.22	7.50	5.80	6.70	4.48	4.66	5.29	5.69	5.96	6.78	
		x_2									
T/K	0.1557	0.1944	0.2407	0.2725	0.3660	0.4302	0.5642	0.6687	0.7531		
298.15	6.76	7.10	7.12	7.25	7.47	7.47	7.39	7.38	7.32		
303.15	6.78	7.12	7.15	7.28	7.49	7.50	7.42	7.41	7.34		
308.15	6.80	7.15	7.17	7.30	7.52	7.53	7.45	7.44	7.37		
313.15	6.83	7.17	7.20	7.33	7.55	7.55	7.47	7.46	7.40		
318.15	6.85	7.20	7.22	7.36	7.58	7.58	7.50	7.49	7.43		
323.15	6.87	7.22	7.25	7.38	7.61	7.61	7.53	7.52	7.45		
328.15	6.90	7.25	7.28	7.41	7.64	7.64	7.56	7.55	7.48		
333.15	6.92	7.28	7.30	7.44	7.67	7.67	7.59	7.58	7.51		
338.15	6.95	7.30	7.33	7.47	7.70	7.70	7.62	7.61	7.54		

Densities and excess molar volumes of aqueous MDEA solutions are presented in Table 5. The negative values of excess molar volume show the significant deviations from ideality at temperatures (298.15 to 338.15) K. The excess molar volumes of aqueous MDEA solutions as a function of mole fraction at temperatures (298.15, 318.15, and 338.15) K are also plotted in Figure 2. The large excess molar volumes of (MDEA + water) reflect the compactness of solution, which could be due to the strong hydrogen bonding interactions. Therefore, aqueous mixtures of amines acquire more efficient packing than the pure liquids, which is considered to be the major contributor for a high magnitude of negative V_m^E values. The excess molar volumes of (MDEA + water) were less negative with an increase in temperature. Figure 2 shows the minimum values of excess molar volumes of (MDEA + water), in the range of (0.3 to 0.4) mole fractions, which can be explained by the occupation of free volume or cavities by MDEA in the open solvent structure. The excess molar volumes were correlated with a Redlich–Kister-type equation by least-squares fittings.

$$V_m^E/\text{cm}^3 \cdot \text{mol}^{-1} = x_2(1-x_2) \sum_{i=0}^i A_i(1-2x_2)^i \quad (5)$$

where A_i are the adjusted coefficients, and their values are presented in Table 6 along with the values of standard

deviations (SD). Partial molar volumes of MDEA in water at infinite dilution ($x_2 = 0$) are presented in Table 7 and compared with available literature values of Hawrylak et al.¹ and Maham et al.¹⁷ The approach followed for the determination of the partial molar volumes of MDEA in water at infinite dilution is similar to that of Hawrylak et al.,¹ i.e., using fitting parameters of the Redlich–Kister equation with the following expression

$$\bar{V}_2^0 = V_2 + \sum_{i=0}^j A_i \quad (6)$$

where \bar{V}_2^0 is the partial molar volume of MDEA in water at infinite dilution; V_2 is the volume of pure MDEA; and A_i is adjusted coefficients. The partial molar volumes of MDEA in water at infinite dilution are lower than the molar volumes of pure MDEA, which may be due to the filling up of the (partially) icelike open structure of pure water with MDEA and forming a new structure with more compact arrangements. The partial molar volumes of MDEA in water at infinite dilution also increase with an increase in temperature. These observations are found to be in good agreement with the reported values of Hawrylak et al.¹ for MDEA. The change in volume of MDEA at infinite dilution at all

Table 5. Experimental Densities and Excess Molar Volumes of MDEA (2) + Water (1) Mixtures

x_2	$T = 298.15 \text{ K}$		$T = 303.15 \text{ K}$		$T = 308.15 \text{ K}$		$T = 313.15 \text{ K}$		$T = 318.15 \text{ K}$		
	ρ kg·m ⁻³	V_m^E cm ³ ·mol ⁻¹	ρ kg·m ⁻³	V_m^E cm ³ ·mol ⁻¹	ρ kg·m ⁻³	V_m^E cm ³ ·mol ⁻¹	ρ kg·m ⁻³	V_m^E cm ³ ·mol ⁻¹	ρ kg·m ⁻³	V_m^E cm ³ ·mol ⁻¹	
0.0128	1004.057	-0.0717	1002.479	-0.0718	1000.698	-0.0721	998.731	-0.0723	996.584	-0.0723	
0.0212	1008.406	-0.1244	1006.702	-0.1243	1004.804	-0.1242	1002.724	-0.1241	1000.480	-0.1240	
0.0498	1020.080	-0.2833	1017.933	-0.2805	1015.632	-0.2780	1013.185	-0.2758	1010.589	-0.2734	
0.0645	1026.392	-0.3953	1023.980	-0.3895	1021.433	-0.3844	1018.768	-0.3799	1015.978	-0.3754	
0.0799	1030.846	-0.4769	1028.249	-0.4695	1025.530	-0.4628	1022.694	-0.4565	1019.743	-0.4504	
0.1332	1042.798	-0.7518	1039.647	-0.7375	1036.417	-0.7244	1033.102	-0.7121	1029.709	-0.6999	
0.1557	1045.445	-0.8274	1042.174	-0.8120	1038.830	-0.7977	1035.413	-0.7843	1031.918	-0.7709	
0.1944	1049.907	-0.9790	1046.417	-0.9603	1042.857	-0.9425	1039.229	-0.9253	1035.539	-0.9081	
0.2407	1050.842	-1.0320	1048.106	-1.0457	1044.446	-1.0272	1040.729	-1.0094	1036.948	-0.9909	
0.2725	1052.408	-1.1110	1048.800	-1.0934	1045.139	-1.0766	1041.409	-1.0598	1037.630	-1.0427	
0.3660	1052.539	-1.1736	1048.778	-1.1537	1044.967	-1.1342	1041.104	-1.1146	1037.198	-1.0939	
0.4302	1050.974	-1.1257	1047.201	-1.1074	1043.379	-1.0890	1039.509	-1.0703	1035.603	-1.0503	
0.5642	1046.854	-0.9073	1043.076	-0.8922	1039.276	-0.8779	1035.454	-0.8642	1031.608	-0.8491	
0.6687	1041.445	-0.4917	1037.681	-0.4786	1033.894	-0.4658	1030.084	-0.4531	1026.252	-0.4382	
0.7531	1039.267	-0.2673	1035.872	-0.2883	1031.924	-0.2628	1028.732	-0.3062	1025.017	-0.3027	
$T = 323.15 \text{ K}$			$T = 328.15 \text{ K}$			$T = 333.15 \text{ K}$			$T = 338.15 \text{ K}$		
0.0128	994.273	-0.0724	991.808	-0.0725	989.191	-0.0726	986.433	-0.0726	984.433	-0.0726	
0.0212	998.073	-0.1238	995.518	-0.1237	992.820	-0.1236	989.984	-0.1234	988.984	-0.1234	
0.0498	1007.874	-0.2716	1005.020	-0.2697	1002.039	-0.2679	998.939	-0.2660	998.939	-0.2660	
0.0645	1013.062	-0.3713	1010.027	-0.3674	1006.729	-0.3637	1003.614	-0.3599	1003.614	-0.3599	
0.0799	1016.683	-0.4448	1013.511	-0.4395	1010.236	-0.4344	1006.853	-0.4293	1006.853	-0.4293	
0.1332	1026.226	-0.6885	1022.667	-0.6779	1019.024	-0.6675	1015.295	-0.6572	1015.295	-0.6572	
0.1557	1028.343	-0.7584	1024.692	-0.7465	1020.961	-0.7348	1017.154	-0.7232	1017.154	-0.7232	
0.1944	1031.783	-0.8920	1027.966	-0.8769	1024.082	-0.8620	1020.126	-0.8472	1020.126	-0.8472	
0.2407	1033.108	-0.9736	1029.246	-0.9585	1025.291	-0.9425	1021.295	-0.9273	1021.295	-0.9273	
0.2725	1033.742	-1.0245	1029.769	-1.0058	1025.947	-0.9963	1021.898	-0.9797	1021.898	-0.9797	
0.3660	1033.245	-1.0746	1029.243	-1.0561	1025.197	-1.0378	1021.103	-1.0196	1021.103	-1.0196	
0.4302	1031.659	-1.0321	1027.676	-1.0149	1023.664	-0.9987	1019.603	-0.9823	1019.603	-0.9823	
0.5642	1027.753	-0.8375	1023.830	-0.8248	1019.875	-0.8125	1015.875	-0.7996	1015.875	-0.7996	
0.6687	1022.397	-0.4260	1018.519	-0.4157	1014.618	-0.4062	1010.695	-0.3977	1010.695	-0.3977	
0.7531	1020.863	-0.2643	1016.835	-0.2410	1012.829	-0.2222	1008.962	-0.2190	1008.962	-0.2190	

Table 6. Fitting Coefficients of the Redlich–Kister Equation along with the Standard Deviations (SD)

T/K	A_0	A_1	A_2	A_3	A_4	SD
298.15	-4.0771	-4.1091	1.7636	-1.1441	0.9365	0.0232
303.15	-3.9933	-4.1898	1.4857	-0.2041	0.3922	0.0226
308.15	-3.9359	-4.1073	1.6707	-0.6369	0.5737	0.0214
313.15	-3.8517	-4.2102	1.5006	0.9720	-0.9451	0.0245
318.15	-3.7789	-4.1928	1.5512	1.2088	-1.3133	0.0251
323.15	-3.7307	-4.0489	1.7933	0.2654	-0.7215	0.0236
328.15	-3.6772	-3.9445	1.9326	-0.2448	-0.4536	0.0226
333.15	-3.6252	-3.8700	2.0234	-0.5914	-0.2540	0.0216
338.15	-3.5660	-3.8176	2.0472	-0.5188	-0.4451	0.0215

Table 7. Partial Molar Volumes of MDEA in Water at Infinite Dilution \bar{V}_2^0 and Values of Change in Volume ΔV

T/K	\bar{V}_2^0		ΔV
	cm ³ ·mol ⁻¹		
298.15	108.14	108.9, ¹ 109.5 ¹⁷	-6.63
303.15	108.68	110.0 ¹⁷	-6.51
308.15	109.17	110.0 ¹	-6.44
313.15	109.49	110.7 ¹⁷	-6.53
318.15	109.93	110.7 ¹	-6.53
323.15	110.44	111.4 ¹⁷	-6.44
328.15	110.94		-6.39
333.15	111.45	112.5 ¹⁷	-6.32
338.15	111.92		-6.30

temperatures is presented in Table 7, which was calculated using

$$\Delta V = \bar{V}_2^0 - V_2 \quad (7)$$

The larger change in volume of sterically hindered amine (MDEA) may be explained by the interstitial contribution of the methyl group to occupy the empty spaces or cavities. Hence, it may be concluded that the change in volume (ΔV) of

ethanolamines reflects the magnitude of hydrophobic hydration and hydrogen bonding with water.

Conclusions

The density of pure and binary mixtures of (MDEA + water) have been measured at a wide temperature range, and a satisfactory agreement was obtained with the available literature values. Excess molar volumes were estimated from density data and correlated with a Redlich–Kister type equation, which showed negative deviations from ideality (i.e., complete miscibility of MDEA in water) at all the investigated temperatures and became less negative with the increase of temperature. The coefficients of thermal expansion values reveal that the volume expansions of the system do not depend on temperature.

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