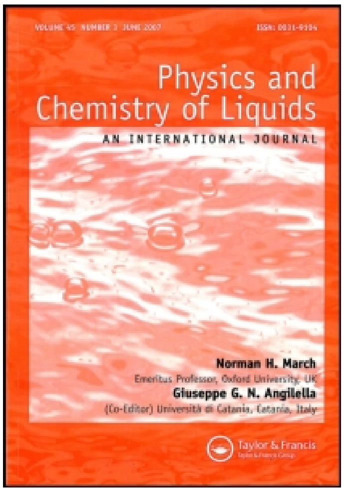

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Densities and excess molar volumes of tert-butanol with *n*-butylamine, di-*n*-butylamine and tri-*n*-butylamine

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Densities of the systems of tert-butanol (TB) + *n*-butylamine (NBA), + di-*n*-butylamine (DBA) and + tri-*n*-butylamine (TBA) were determined between 303.15 and 323.15 K at different compositions in the range $0 \leq x_2 \leq 1$, where x_2 is the mole fraction of butylamines BA, NBA, DBA and TBA. Excess molar volumes have been calculated from the density data. In the whole range of composition, systems of TB + NBA and TB + DBA show negative V_m^E values, whereas the values are positive for TB + TBA. While the negative values are accounted for by the effects of specific interaction and interstitial accommodation, positive values are due to dispersive forces.

Keywords: densities; excess molar volumes; dispersive force; tert-butanol; butylamines

1. Introduction

This is a part of our continuous research on molecular interactions through the measurement of volumetric and viscometric properties of pure liquids and their binary mixtures, consisting mainly of various alcohols and amines. Recently, a brief literature survey showed that quite a large number of investigations have been conducted on volumetric and viscometric properties of binary liquid systems, comprising of different types of alcohols and/or amines with water or non-aqueous solvents. Among these, a unique piece of work is reported by Kipkemboi and Easteal [1] on volumetric and viscometric properties of aqueous solutions of tert-butanol (TB) and tert-butylamine. Kim and Marsh [2] reported on volumetric properties of aqueous solutions of TB. As a part of the systems of water + amines, some other works [3,4] were also carried out, and in each case strong intermolecular interactions between the components was suggested. With relevance to the above studies, we have already reported on the volumetric and viscometric properties of *n*-, sec- and tert-butylamines [5], alcohols [6–8], ethylenediamine, trimethylenediamine and *N*,*N*-dimethyltrimethylenediamine [9], and very recently of diamines [10], all in the aqueous medium.

On the contrary, Reimann and Heintz [11], Papaioannou *et al.* [12], Pikkarainen [13] and Panayiotou [14] carried out different measurements on volumetric, viscometric, and

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enthalpic, as well as thermodynamic properties of the systems of alcohols + amines. Recently, Oswal and Desai [15,16] have investigated the systems of 1-alkanols with di-*n*-propylamine, di-*n*-butylamine (DBA) and tri-*n*-butylamine (TBA). Furthermore, other works [17–20] also studied amine–alkanol mixtures. Singh *et al.* [21] reported physico-chemical properties, including the volumetric properties of NBA + 1-butanol and NBA + TB systems. All of them unambiguously suggested there were strong interactions between amines and alcohols through H-bonding.

As far we know, there is no report yet on the systems involving TB with alkylamines of the above type. Therefore, in order to provide this new data, we made a systematic attempt to investigate the systems of TB with some alkylamines. In this investigation, densities (ρ) are measured at temperatures between 303.15 and 323.15 K, from which excess molar volumes (V_m^E) are estimated for binary solutions of TB with some substituted butylamines (BA), namely, *n*-butylamine (NBA), DBA and TBA. This would give us the necessary information about the nature of interaction and also provide an opportunity to examine the effect of the number of butyl groups attached to the amino group.

2. Experimental

All the chemicals used for the present investigation were procured from Aldrich Chemical Company, with quoted purities: TB (99.5%), NBA (99%), DBA (99%) and TBA (99%). These were used without further purification, except that each of the amines was kept over a molecular sieve (4A) for at least three weeks prior to use. As a measure of the purity check, measured densities of the pure liquids were compared with the available literature values [2,5,15–19,22–28], and all showed satisfactory agreement, as seen in Table 1.

The density was measured by a 25 mL specific gravity bottle that was previously calibrated by doubly distilled water. The specific gravity bottle containing the solution was immersed in a constant temperature bath controlled within ± 0.05 K by using a circulator temperature controller (HAAKE DC 10). The weighing was performed by an analytical balance (Mettler Toledo B-S) with an accuracy of ± 0.0001 g. Each reported density data was an average of at least three measurements. The uncertainty in the measured density was estimated to be $\pm 2 \times 10^{-5}\,\mathrm{g\,cm^{-3}}$.

The excess molar volume V_m^E was calculated by the following equation:

$$V_\mathrm{m}^\mathrm{E} = \frac{X_1 M_1 + X_2 M_2}{\rho_\mathrm{mix}} - \left(\frac{X_1 M_1}{\rho_1} + \frac{X_2 M_2}{\rho_2}\right), \tag{1}$$

where ρ_mix is the measured density of the mixture, M_1, V_1 and x_1 are the molar mass, molar volume and mole fraction of component 1 and M_2, V_2 and x_2 are the corresponding quantities of component 2 in the mixture.

3. Results and discussions

Densities (ρ) of pure amines NBA, DBA, TBA and TB are shown in Table 1 at different temperatures between 303.15 and 323.15 K, together with literature values for comparison. The ρ and the estimated V_m^E of all the systems, TB + NBA, TB + DBA and TB + TBA, over the composition range, $0 \le x_2 \le 1$, where x_2 is the mole fraction of the respective BA, are listed in Table 2.

Table 1. Experimental and literature densities of pure TB, NBA, DBA and TBA at different temperatures.

	TB	NBA	DBA	TBA
T (K)	ρ	ρ	ρ	ρ
303.15	0.7752	0.7277	0.7523	0.7705
	0.77521[a]	0.7273[b]	0.75424[c]	0.77037[d]
	0.7754[f]	0.72865[e]	0.75248[g]	0.7706[k]
	0.7755[i]	0.7276[h]	0.75194[n]	0.77046[n]
	0.77524[j]			
308.15	0.7698	0.7229	0.7481	0.7667
	0.76997[a]	0.7226[b]	0.74791[n]	0.76695[n]
		0.7228[h]		
313.15	0.7644	0.7180	0.7440	0.7626
	0.76469[a]	0.7176[b]	0.74284[c]	0.76337[n]
		0.71917[e]	0.74393[n]	
		0.7192[l]		
		0.7182[m]		
318.15	0.7590	0.7131	0.7399	0.7591
	0.75937[a]	0.7127[b]	0.7399[n]	0.75974[n]
323.15	0.7536	0.7082	0.7358	0.7563
	0.75401[a]	0.7078[b]	0.73591[n]	0.75604[n]

Notes: [a]Kim and Marsh [2], [b]Saleh *et al.* [5], [c]Oswal and Desai [15], [d]Oswal and Desai [16], [e]Oswal and Desai [17], [f]Nikam *et al.* [18], [g]Oswal and Desai [19], [h]Acevedo and Katz [22], [i]Aminabhavi and Gopalkrishna [23], [j]Brown and Smith [24], [k]Oswal and Rao [25], [l]de Schaefer *et al.* [26], [m]Dominguez *et al.* [27], [n]Oswal *et al.* [28].

The values of ρ of the measured systems are fitted by a polynomial equation of the general form:

$$\rho(\,\mathrm{g\,cm^{-3}}) = \sum_{i=0}^{n} a_i x_2^i, \tag{2}$$

where x_2 is the mole fraction of BA, a_i ($\mathrm{g\,cm^{-3}}$) is the regression coefficient and n is the degree of polynomial. The values of ρ fit to Equation (2) well for $n = 6$. The coefficients a_i of Equation (2) and relevant values of r^2 are listed in Table 3.

The variation of ρ with respect to x_2 are plotted in Figures 1–3 for TB + NBA, TB + BDA and TB + TBA, respectively, and Figure 4 shows their comparison at 303.15 K. As Figures 1–4 show, densities of the amines vary in the order TBA > DBA > NBA. The curves of TB + NBA at different temperatures are convex towards x_2 and they are of a similar pattern. For TB + DBA, to a rough approximation, the curves bend down to about 0.10 mole fraction of DBA and then follow almost linearly. Unlike the other two systems, curves for TB + TBA are concave in nature, but the characteristics at extremely high concentrations of TBA are similar to those of TB + DBA.

The excess molar volumes, V_m^E, as a function of mole fraction of BA at different temperatures are shown in Figures 5–7, and their comparative curves at 303.15 K are as shown in Figure 8. Each set of results of V_m^E covering the whole range of composition at a

Table 2. Experimental densities ρ (g cm^{-3}) and excess molar volumes V_m^E(cm^3 mol^{-1}) of the systems TB (x_1) + NBA (x_2), + DBA (x_2) and + TBA (x_2) for different molar ratios at different temperatures.

T (K)	303.15		308.15		313.15		318.15		323.15	
x_2	ρ	V_m^E	ρ	V_m^E	ρ	V_m^E	ρ	V_m^E	ρ	V_m^E
					TB (x_1) + NBA (x_2)					
0.0000	0.7752	0.000	0.7698	0.000	0.7644	0.000	0.7590	0.000	0.7536	0.000
0.0500	0.7743	−0.197	0.7692	−0.221	0.7640	−0.258	0.7588	−0.284	0.7535	−0.298
0.0996	0.7729	−0.329	0.7678	−0.363	0.7628	−0.413	0.7577	−0.450	0.7527	−0.502
0.2000	0.7696	−0.538	0.7647	−0.592	0.7598	−0.651	0.7548	−0.699	0.7498	−0.747
0.3000	0.7657	−0.666	0.7609	−0.728	0.7561	−0.795	0.7512	−0.851	0.7464	−0.922
0.4000	0.7614	−0.752	0.7567	−0.808	0.7519	−0.870	0.7471	−0.934	0.7422	−0.986
0.5004	0.7566	−0.747	0.7518	−0.799	0.7470	−0.850	0.7422	−0.908	0.7373	−0.953
0.6000	0.7513	−0.678	0.7466	−0.730	0.7418	−0.779	0.7370	−0.830	0.7321	−0.868
0.7001	0.7458	−0.577	0.7410	−0.608	0.7362	−0.649	0.7314	−0.692	0.7265	−0.721
0.7996	0.7400	−0.424	0.7352	−0.445	0.7303	−0.464	0.7255	−0.498	0.7207	−0.532
0.9000	0.7342	−0.267	0.7294	−0.278	0.7245	−0.289	0.7197	−0.313	0.7148	−0.324
1.0000	0.7277	0.000	0.7229	0.000	0.7180	0.000	0.7131	0.000	0.7082	0.000
					TB (x_1) + DBA (x_2)					
0.0500	0.7747	−0.191	0.7692	−0.205	0.7640	−0.219	0.7589	−0.233	0.7535	−0.248
0.1000	0.7738	−0.324	0.7687	−0.339	0.7636	−0.355	0.7586	−0.370	0.7534	−0.387
0.1999	0.7716	−0.506	0.7666	−0.511	0.7616	−0.517	0.7566	−0.523	0.7516	−0.528
0.3000	0.7690	−0.587	0.7642	−0.598	0.7594	−0.610	0.7546	−0.622	0.7498	−0.635
0.3999	0.7665	−0.631	0.7618	−0.636	0.7571	−0.641	0.7524	−0.647	0.7477	−0.652
0.5001	0.7639	−0.609	0.7593	−0.609	0.7547	−0.609	0.7501	−0.609	0.7455	−0.609
0.6000	0.7614	−0.553	0.7569	−0.549	0.7524	−0.545	0.7479	−0.541	0.7434	−0.538
0.7000	0.7590	−0.449	0.7545	−0.440	0.7501	−0.434	0.7457	−0.429	0.7413	−0.423
0.8000	0.7567	−0.334	0.7523	−0.324	0.7480	−0.318	0.7437	−0.312	0.7394	−0.306
0.9000	0.7544	−0.187	0.7502	−0.184	0.7460	−0.180	0.7418	−0.176	0.7376	−0.172
1.0000	0.7523	0.000	0.7481	0.000	0.7440	0.000	0.7399	0.000	0.7358	0.000
					TB (x_1) + TBA (x_2)					
0.0497	0.7745	0.020	0.7694	0.005	0.7643	–	0.7592	–	0.7541	–
0.0994	0.7360	0.082	0.7686	0.076	0.7636	0.069	0.7586	0.062	0.7536	0.050
0.2000	0.7723	0.175	0.7675	0.180	0.7627	0.186	0.7579	0.191	0.7531	0.196
0.3000	0.7715	0.228	0.7669	0.236	0.7623	0.244	0.7577	0.253	0.7531	0.262
0.4001	0.7710	0.250	0.7666	0.254	0.7621	0.278	0.7577	0.281	0.7532	0.305
0.4999	0.7707	0.248	0.7664	0.261	0.7621	0.274	0.7578	0.287	0.7535	0.300
0.6000	0.7706	0.210	0.7664	0.228	0.7622	0.246	0.7580	0.264	0.7538	0.283
0.7000	0.7705	0.175	0.7664	0.194	0.7623	0.214	0.7582	0.234	0.7541	0.254
0.8000	0.7705	0.117	0.7665	0.133	0.7625	0.150	0.7585	0.167	0.7545	0.185
0.9002	0.7705	0.058	0.7666	0.069	0.7627	0.079	0.7588	0.089	0.7549	0.100
1.0000	0.7705	0.000	0.7667	0.000	0.7629	0.000	0.7591	0.000	0.7553	0.000

Table 3. Fitting coefficients a_i (g cm^{-3}) of polynomial Equation (2) and the value of r^2 for the systems TB (x_1) + NBA (x_2), + DBA (x_2) and + TBA (x_2) at different temperatures.

T (K)	a_0	a_1	a_2	a_3	a_4	a_5	a_6	r^2
				TB (x_1) + NBA (x_2)				
303.15	−0.1962	0.5794	−0.6493	0.3547	−0.1226	−0.0135	0.7752	1.0000
308.15	−0.2317	0.6895	−0.7798	0.4305	−0.1471	−0.0083	0.7698	1.0000
313.15	−0.2364	0.7164	−0.8310	0.4763	−0.1689	−0.0028	0.7644	1.0000
318.15	−0.2863	0.8652	−1.0004	0.5693	−0.1958	0.0020	0.7590	1.0000
323.15	−0.2499	0.7567	−0.8863	0.5236	−0.1950	0.0055	0.7536	1.0000
				TB (x_1) + DBA (x_2)				
303.15	−0.0416	0.1623	−0.2545	0.2067	−0.0890	−0.0068	0.7752	1.0000
308.15	−0.0506	0.1717	−0.2413	0.1868	−0.0832	−0.0050	0.7698	0.9999
313.15	−0.0808	0.2668	−0.3563	0.2543	−0.1036	−0.0007	0.7644	1.0000
318.15	−0.1557	0.5063	−0.6497	0.4261	−0.1525	0.0065	0.7590	0.9999
323.15	−0.1147	0.3737	−0.4871	0.3340	−0.1305	0.0068	0.7536	0.9999
				TB (x_1) + TBA (x_2)				
303.15	−0.0765	0.2452	−0.2934	0.1495	−0.0137	−0.0157	0.7752	0.9995
308.15	−0.1287	0.4080	−0.4865	0.2592	−0.0456	−0.0094	0.7698	0.9983
313.15	−0.1689	0.5406	−0.6554	0.3631	−0.0782	−0.0029	0.7645	0.9933
318.15	−0.2211	0.7034	−0.8485	0.4728	−0.1100	0.0034	0.7591	0.9785
323.15	−0.2612	0.8361	−1.0173	0.5768	−0.1427	0.0099	0.7537	0.9817

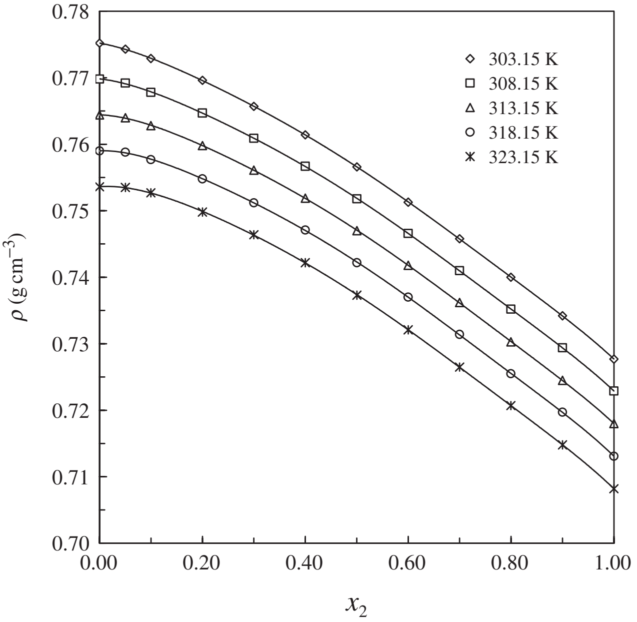

Figure 1. Densities for the solution of TB + NBA as a function of mole fraction of BA (x_2) at different temperatures. The full lines represent fitting values with Equation (2).

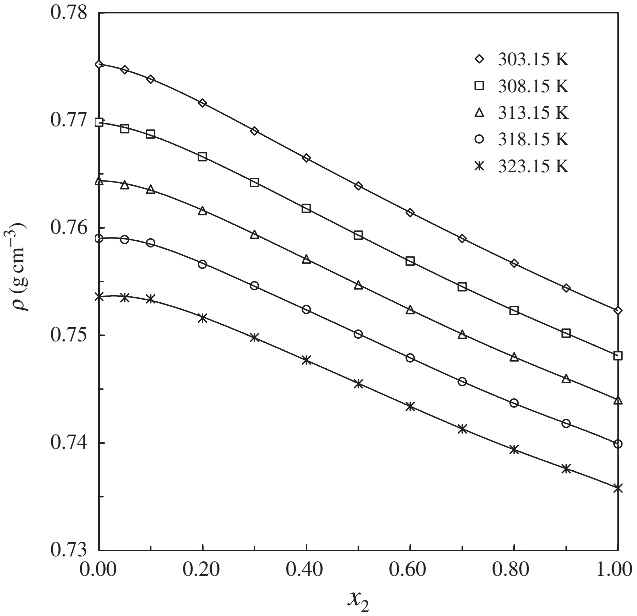

Figure 2. Densities for the solution of TB + DBA as a function of mole fraction of BA (x_2) at different temperatures. The full lines represent fitting values with Equation (2).

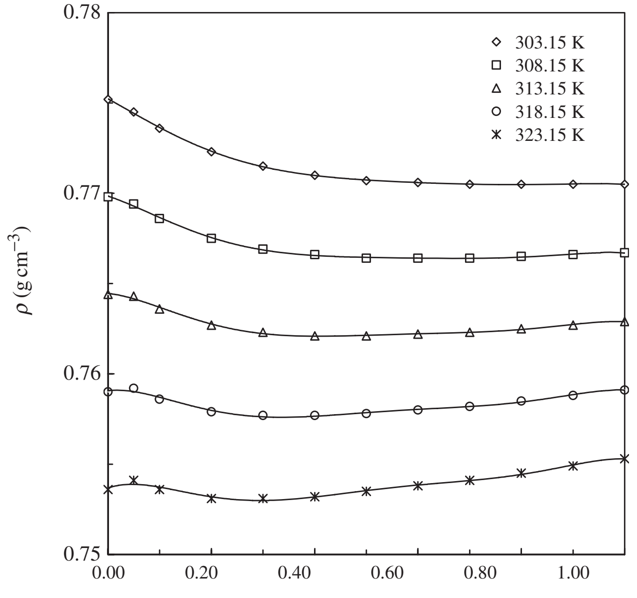

Figure 3. Densities for the solution of TB + TBA as a function of mole fraction of BA (x_2) at different temperatures. The full lines represent fitting values with Equation (2).

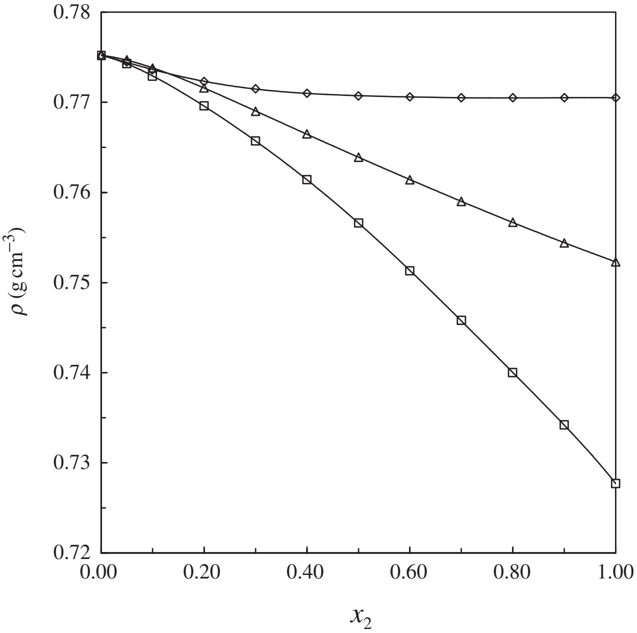

Figure 4. Comparison of densities for the solutions of TB + NBA (□), TB + DBA (△) and TB + TBA (◇) as a function of mole fraction of BA (x_2) at the temperature 303.15 K. The full lines represent fitting values with Equation (2).

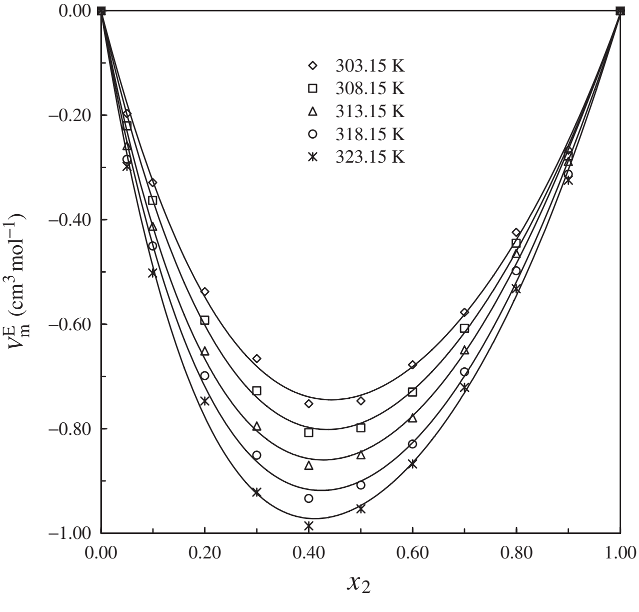

Figure 5. Excess molar volumes for the solution of TB + NBA as a function of mole fraction of BA (x_2) at different temperatures. The full lines represent fitting values with Equation (3).

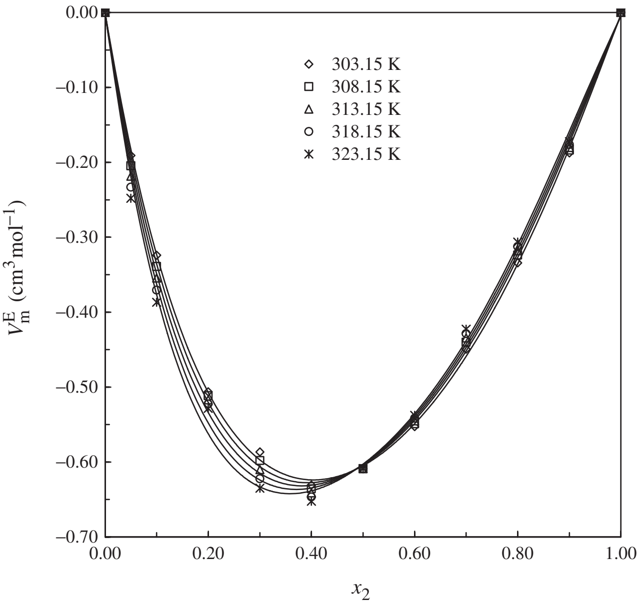

Figure 6. Excess molar volumes for the solution of TB + DBA as a function of mole fraction of BA (x_2) at different temperatures. The full lines represent fitting values with Equation (3).

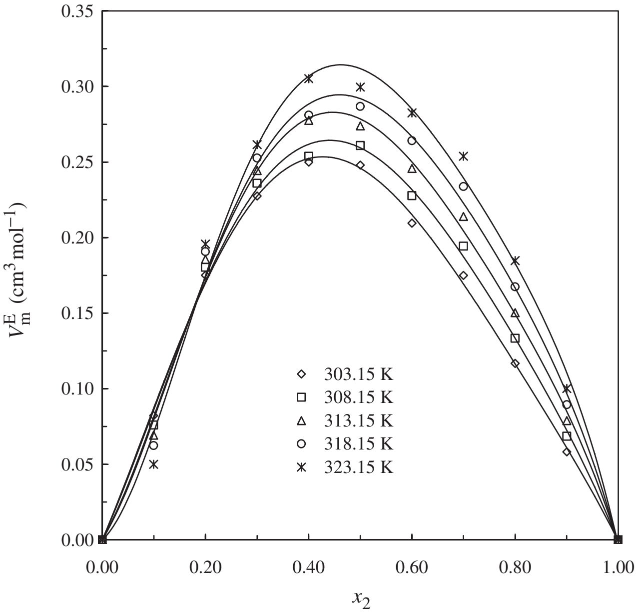

Figure 7. Excess molar volumes for the solution of TB + TBA as a function of mole fraction of BA (x_2) at different temperatures. The full lines represent fitting values with Equation (3).

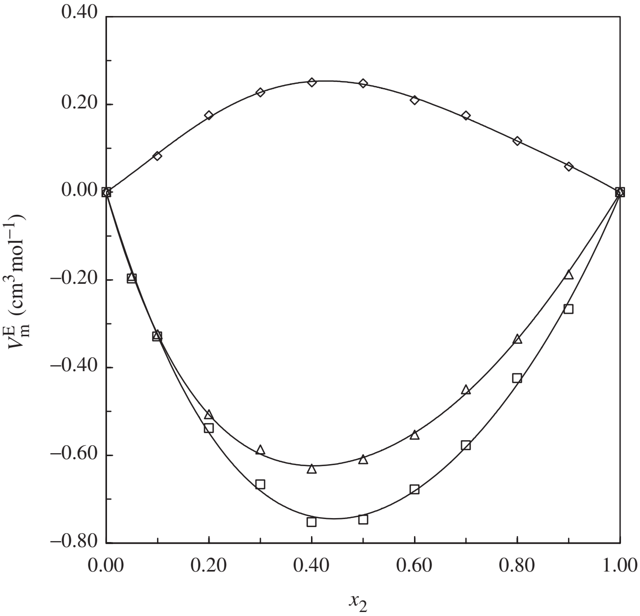

Figure 8. Comparison of excess molar volumes for the solutions of TB + NBA (□), TB + DBA (△) and TB + TBA (◇) as a function of mole fraction of BA (x_2) at the temperature 303.15 K. The full lines represent fitting values with Equation (3).

particular temperature between 303.15 and 323.15 K was fitted to the Redlich–Kister polynomial equation of the type:

$$V_m^E(\text{cm}^3\text{mol}^{-1}) = x_2(1-x_2)\sum_{i=1}^{n} A_i(1-2x_2)^{i-1}, \tag{3}$$

where A_i (cm^3 mol^{-1}) is the i-th fitting coefficient of the Redlich–Kister polynomial equation, and all the other terms have their usual significance. Using $n = 6$, six different A_i values and the relevant standard deviation, σ (cm^3 mol^{-1}) were obtained through the least-squares method. All the A_i coefficients of Equation (3) and their relevant σ values are listed in Table 4.

At a particular temperature, V_m^E values are found to be negative in the whole range of composition for TB + NBA and TB + DBA, showing minima at about $x_2 = 0.40$ and 0.45, respectively. But the values of V_m^E of TB + NBA are more negative than those of TB + DBA at least at the concentration of minimum V_m^E. Unlike these two systems, V_m^E values of TB + TBA are found to be positive in the whole range of composition at all temperatures, showing maxima at ~0.40 mole fraction of TBA.

Generally, it is believed that both TB and NBA are associated liquids in their pure state, TB being more associated than NBA. When NBA is added to highly associated TB, it is likely that a reasonable proportion of NBA, existing as multimeric, dissociates into monomers. As fast as the NBA monomers are formed, they are likely to interact with either monomeric TB or their multimers, resulting in a volume contraction. Other factors responsible for volume contraction may be the interstitial accommodation as well as the

Table 4. Fitting coefficients A_i (cm^3 mol^{-1}) of Redlich–Kister polynomial Equation (3) and the values of σ (cm^{3}mol^{-1}) for V_m^E (cm^3 mol^{-1}) for the systems TB(x_1) + NBA(x_2), + DBA(x_2) and + TBA(x_2) at different temperatures.

T (K)	A_1	A_2	A_3	A_4	A_5	A_6	σ
			TB(x_1) + NBA(x_2)				
303.15	−2.9975	−0.6778	0.5657	0.4591	−1.6922	−0.2453	0.00803
308.15	−3.2161	−0.7769	0.5677	0.1103	−1.8132	0.1000	0.00745
313.15	−3.4397	−0.9161	0.5066	0.0447	−1.9871	−0.1759	0.01137
318.15	−3.6778	−1.0474	0.5800	0.3793	−2.3459	−0.6087	0.01240
323.15	−3.8535	−1.2892	0.1718	0.8974	−1.9798	−1.2837	0.01022
			TB(x_1) + DBA(x_2)				
303.15	−2.4450	−0.7904	−0.1546	−0.2595	−0.7766	−0.0210	0.00550
308.15	−2.4524	−0.9265	−0.0049	0.0378	−1.1331	−0.4590	0.00405
313.15	−2.4571	−1.0646	0.0372	0.4512	−1.3240	−1.0973	0.00434
318.15	−2.4632	−1.2076	0.0726	0.9056	−1.4909	−1.7806	0.00620
323.15	−2.4700	−1.3477	0.1310	1.3501	−1.7101	−2.4761	0.00952
			TB(x_1) + TBA(x_2)				
303.15	0.9695	0.3320	0.0547	0.2568	−0.6126	−0.9106	0.00652
308.15	1.0134	0.1713	0.2383	0.9403	−0.9588	−1.9178	0.00807
313.15	1.0878	0.2466	0.2068	0.0559	−0.9415	−0.8233	0.00599
318.15	1.1306	0.0957	0.4174	0.6103	−1.3305	−1.6160	0.00525
323.15	1.2003	0.1073	0.5399	0.3327	−1.6983	−1.5844	0.00804

differences in the size and shape of the component molecules. However, as the V_m^E curves virtually fall at low concentration of NBA and are skewed towards its higher concentration side, the interstitial accommodation of NBA monomers into TB networks causing considerable volume contraction seems to play an important role in the highly TB-rich region. Again, factors that are likely to cause volume expansion, such as the effect of dispersive forces, seem to be much less important. Thus, in this particular system, as the contractive factors are much stronger than the expansive factors, it is expected that the resultant volume of the mixture should be less than the volume calculated ideally. This is indeed the case, as shown by the negative excess molar volume in the whole range of concentration. From the work of Papaioannou *et al.* [12], relatively large negative excess molar volumes for the systems NBA + 1-alkanol (methanol, ethanol, 1-propanol, 1-butanol) were found, which were interpreted considering the specific interactions between the hydroxyl and amine groups in the mixture. The minimum values of V_m^E for the system NBA + tert-butanol is observed as about −0.70 cm^3 mol^{-1} at 303.15 K compared to −1.20 cm^3 mol^{-1} of NBA + 1-butanol at 298.15 K [12]. The less negative value of V_m^E of NBA + TB is attributed to the branching effect of TB.

A similar argument, for negative V_m^E for TB + NBA, can be applied to the negative V_m^E for TB + DBA. The effect, however, is reduced to a large extent because of larger steric hindrance due to the attachment of two butyl groups with the N-atom of DBA.

Of the three amines, TBA is the most extensively crowded, posing maximum steric hindrance. It is to be remembered that TB is also a sterically hindered molecule. The obvious consequence is that these two molecules can hardly come close enough to form a H-bond between them, nor can they incorporate into the interstitial spaces. Therefore, in TB + TBA the contraction of volume cannot be expected as in TB + NBA or TB + DBA. Moreover, because of maximum steric hindrance and perhaps due to the dissociation effect of TBA on TB, volume expansion is more likely to take place. This is indeed the case, as proved experimentally.

For the TB + NBA system, with the rise of temperature, the negative values have been found to increase, i.e. $\delta V_\mathrm{m}^\mathrm{E}/\delta \mathrm{T}$ is negative in the whole range of concentration. The reverse effect of temperature is found for the TB + DBA system above $x_2 = 0.50$, while $\delta V_\mathrm{m}^\mathrm{E}/\delta \mathrm{T}$ is positive for TB + TBA in the whole range of composition.

Oswal and Desai [15] have found very large negative excess molar volumes for the mixtures of di-*n*-propylamine and DBA with 1-alkanols, and they have explained those by considering the mixed association of alkanol with diamine through the OH ⋯ N bond. The magnitude of V_m^E for the DBA + *n*-butanol system is more than double than that of the DBA + TB system at the minima. This is also true for the systems when DBA is replaced by NBA. Similar results, i.e. negative excess molar volumes, were also found for the NBA + acetonitrile [26] and TBA + 1-alkanol [16] systems. Other reports, by Oswal and Desai [17,19] and very recently Weng and Chen [20], have observed negative excess molar volumes for the NBA + 1-alkanol, *n*-propylamine + 1-alkanol and NBA + hexanol systems. On the other hand, positive excess molar volumes are also found for the TB + dimethylsulfoxide [29], TBA + ethylacetate, + ethanol, + propan-1-ol, + propan-2-ol, + butan-1-ol, + 2-methylpropan-1-ol and + 2-methylpropan-2-ol systems [18]. Likewise, in our current investigation, we have also found small but positive excess molar volumes for the TB + TBA system in the whole range of composition within the studied range of temperature.

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