International Journal of Thermophysics, Vol. 27, No. 5, September 2006 (© 2006)
DOI: 10.1007/s10765-006-0095-5

Densities and Refractive Indices of Binary Mixtures of Benzene with Triethylamine and Tributylamine at Different Temperatures

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Received May 20, 2005

Experimental densities, ρ, and refractive indices, n_D for binary liquid mixtures of benzene with triethylamine (TEA) and tributylamine (TBA) have been measured as a function of composition in the temperature range from 278.15 to 318.15 K. The excess molar volume, V^{E}, and its temperature dependence, dV^{E}/dT for the binary mixtures were calculated using the experimental data. The values of V^{E} for the mixtures were also estimated by using the Flory statistical theory and refractive index.

KEY WORDS: benzene; binary mixtures; densities; Flory's theory; refractive indices; tributylamine; triethylamine.

1. INTRODUCTION

The concentration and temperature dependences of the physicochemical properties of selected multicomponent liquid mixtures are often required for use in technological, biological, and industrial applications since they help to provide a wide choice of solvents with desired compositions and properties [1–3]. Binary mixtures of benzene with tert-alkylamines are expected to reveal many interesting properties because of the possible existence of specific interactions between the mixing components [4].

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Trialkylamines are industrially important liquids, which are used as intermediates in the production of polymers such as phenolic resins, corrosion inhibitors, agrochemicals, lubricating additives, pharmaceuticals, textile dyes, paints, and polyurethane foams [5]. A literature survey indicates that studies on molecular interactions in binary mixtures of amines with benzene [4], 2-methoxyethanol [6], alkanes [7], alcohols [8], linear and branched alkanes [9], and with chloroform [10] have been reported using volumetric and calorimetric methods at single, or at the most, three different temperatures. To the best of our knowledge, there are no literature data on binary mixtures of benzene with TEA and TBA for volumetric and refractive index measurements over a relatively wide range of temperature (278.15–318.15 K). In a continuation of our earlier work [11–15] on molecular interactions in nonaqueous binary liquid mixtures, we report here densities, and refractive indices of binary mixtures of benzene with TEA and TBA at 278.15, 283.15, 288.15, 293.15, 298.15, 303.15, 308.15, 313.15, and 318.15 K over the entire composition range expressed by the mole fraction, x_1, of benzene.

These experimental data have been used to calculate the excess molar volume, V^{E}, and its variation with temperature, dV^{E}/dT of the binary liquid mixtures. The excess molar volume, V^{E} has also been predicted by using Flory's theory [16, 17] and the refractive index. The variation of the excess volume with composition offers a convenient, model free approach, for the qualitative study of thermodynamic properties of the liquids and their mixtures [18]. Furthermore, the effect of nonpolar parts (alkyl groups), their size and the presence of the polar part N: in TEA and TBA molecules on intermolecular interactions between benzene and amine molecules has been examined.

2. EXPERIMENTAL

All the chemicals used for the present study were obtained from s.d. finechem Ltd., India, with purities greater than 99%. They were purified by methods described in the literature [6, 19, 20]. The water content from the pure components, if any, was removed by storing them over 0.4 nm molecular sieve. The binary mixtures were prepared by mass at room temperature and kept in special airtight bottles. The weighings were done on a "Precisa XB-120" (Swiss make) electronic balance with a precision of ±0.1 mg.

The densities of the pure liquids and of their binary solutions were measured at different temperatures by using a single-capillary pycnometer (made of Borosil glass) having a bulb capacity of $8 \times 10^{-6}\,\mathrm{m}^3$. The capillary, with graduated marks, had a uniform bore and can be closed by a well-fitting glass cap. The marks on the capillary were calibrated by using triple-distilled water.

The refractive indices of pure liquids and their binary mixtures were measured by using a thermostated Abbe refractometer (Metrex, India). The temperature of the test liquids and their binary mixtures was maintained to an uncertainty of ±0.02 K using a JULABO (Germany) electronically controlled thermostatted water bath.

The overall uncertainties in the measurements of ρ and n_D were estimated to be ±0.0001 g·cm^{-3} and ±0.0001, respectively. The experimental values of density and refractive index of the pure liquids at different temperatures were compared with literature values, as shown in Table I.

3. RESULTS AND DISCUSSION

The experimental values of ρ, and n_D for the mixtures of benzene with triethylamine (TEA) and tributylamine (TBA) over the entire composition range expressed by the mole fraction, x_1, of benzene at vari-

Table I. Comparisons of Experimental Density, ρ, and Refractive Index, n_D of Pure Liquids with Literature Values at Different Temperatures

		ρ (g·cm^{-3})		n_D	
Liquid	T(K)	Expt.	Lit.	Expt.	Lit.
Benzene	298.15	0.8736	0.8736 [21,22] 0.8737 [23,24]	1.4979	1.4969 [21] 1.4979 [22] 1.4982 [24]
	303.15	0.8708	0.8686 [25]	1.4962	–
	308.15	0.8657	0.8629 [26]	1.4944	–
	313.15	0.8610	0.8595 [27]	1.4929	–
	318.15	0.8563	–	1.4911	–
TEA	298.15	0.7234	0.7234 [28] 0.7236 [29]	1.4011	1.4015 [30]
	303.15	0.7179	0.7188 [30] 0.7186 [5]	1.3983	1.3987 [30]
	308.15	0.7129	0.7137 [5]	1.3950	1.3957 [30]
	313.15	0.7083	0.7086 [31]	1.3928	1.3931 [30]
	318.15	0.7016	–	1.3898	1.3902 [30]
TBA	298.15	0.7743	0.7740 [22] 0.7739 [29] 0.7742 [32]	1.4274	1.4268 [22] 1.4267 [29]
	303.15	0.7708	0.7709 [22]	1.4259	1.4255 [5] 1.4256 [30]
	308.15	0.7672	0.7657 [5]	1.4231	1.4228 [5]
	313.15	0.7636	0.7631 [22] 0.7634 [33]	1.4210	–
	318.15	0.7598	0.7597 [30]	1.4188	–

ous temperatures are listed in Table IIa,b. Using these data, the derived properties such as excess molar volume, V^{E}, and its temperature dependence, dV^{E}/dT were calculated by using the following relation,

$$V^{\mathrm{E}} = x_1 M_1 (1/\rho - 1/\rho_1) + x_2 M_2 (1/\rho - 1/\rho_2) \tag{1}$$

where x and M are the mole fraction and molar mass, respectively; subscripts 1 and 2 represent the pure components benzene and amine, respectively.

The excess functions, a measure of deviations from ideal behavior, are found to be highly sensitive towards intermolecular interactions between the component molecules of the mixture. The variations of V^{E} with mole fraction, x_1, of benzene are shown in Fig. 1. The values of V^{E} (Fig. 1) are found to be positive for mixtures of benzene with TEA and TBA at all nine temperatures and over the entire composition range, and show a decreasing trend as the temperature increases from 278.15 to 318.15 K. For the sake of clarity, the plots of V^{E} vs. x_1 in Fig. 1 are shown only at 278.15 and 318.15 K.

Table II(a). Values of Densities, $\rho(\mathrm{g \cdot cm^{-3}})$, of Benzene with TEA and TBA as a Function of Mole Fraction, x_1, of Benzene at Different Temperatures

	T (K)								
x_1	278.15	283.15	288.15	293.15	298.15	303.15	308.15	313.15	318.15
Benzene + Triethylamine (TEA)									
0.1145	0.7513	0.7474	0.7433	0.7384	0.7334	0.7282	0.7234	0.7189	0.7126
0.2043	0.7594	0.7555	0.7515	0.7468	0.7418	0.7370	0.7323	0.7279	0.7217
0.3005	0.7692	0.7654	0.7614	0.7567	0.7517	0.7471	0.7424	0.7381	0.7321
0.4005	0.7806	0.7769	0.7729	0.7682	0.7631	0.7587	0.7540	0.7497	0.7439
0.5023	0.7936	0.7900	0.7860	0.7814	0.7761	0.7720	0.7673	0.7629	0.7572
0.6037	0.8081	0.8046	0.8007	0.7961	0.7907	0.7868	0.7821	0.7778	0.7723
0.7035	0.8242	0.8210	0.8172	0.8127	0.8072	0.8036	0.7990	0.7947	0.7895
0.8079	0.8439	0.8410	0.8374	0.8331	0.8274	0.8242	0.8196	0.8154	0.8105
0.9069	0.8662	0.8635	0.8599	0.8556	0.8496	0.8468	0.8421	0.8378	0.8331
Benzene + Tributylamine (TBA)									
0.0702	0.7901	0.7867	0.7832	0.7799	0.7756	0.7725	0.7691	0.7655	0.7618
0.1162	0.7911	0.7878	0.7844	0.7811	0.7769	0.7738	0.7704	0.7668	0.7631
0.1687	0.7925	0.7893	0.7860	0.7827	0.7785	0.7754	0.7719	0.7684	0.7648
0.2383	0.7949	0.7918	0.7885	0.7853	0.7810	0.7779	0.7744	0.7709	0.7672
0.3091	0.7977	0.7946	0.7913	0.7881	0.7838	0.7808	0.7773	0.7738	0.7701
0.4032	0.8022	0.7992	0.7959	0.7927	0.7883	0.7854	0.7819	0.7784	0.7747
0.5043	0.8085	0.8056	0.8023	0.7990	0.7945	0.7916	0.7881	0.7845	0.7808
0.6492	0.8213	0.8185	0.8152	0.8118	0.8070	0.8043	0.8006	0.7970	0.7933
0.8251	0.8468	0.8441	0.8408	0.8371	0.8316	0.8290	0.8249	0.8211	0.8172

Table II(b). (*Continued*)

	T (K)								
x_1	278.15	283.15	288.15	293.15	298.15	303.15	308.15	313.15	318.15
				Benzene + Triethylamine (TEA)					
0.1145	1.4149	1.4133	1.4113	1.4090	1.4062	1.4038	1.4009	1.3988	1.3961
0.2043	1.4197	1.4180	1.4160	1.4137	1.4110	1.4088	1.4061	1.4043	1.4017
0.3005	1.4261	1.4243	1.4224	1.4201	1.4173	1.4151	1.4125	1.4107	1.4082
0.4005	1.4336	1.4318	1.4300	1.4278	1.4250	1.4228	1.4203	1.4185	1.4161
0.5023	1.4421	1.4404	1.4387	1.4366	1.4338	1.4317	1.4293	1.4275	1.4252
0.6037	1.4518	1.4501	1.4485	1.4465	1.4438	1.4418	1.4395	1.4378	1.4356
0.7035	1.4625	1.4609	1.4594	1.4576	1.4549	1.4531	1.4510	1.4495	1.4474
0.8079	1.4756	1.4740	1.4727	1.4710	1.4683	1.4666	1.4647	1.4632	1.4613
0.9069	1.4902	1.4886	1.4872	1.4856	1.4829	1.4812	1.4795	1.4779	1.4759
				Benzene + Tributylamine (TBA)					
0.0702	1.4425	1.4414	1.4406	1.4394	1.4385	1.4383	1.4374	1.4366	1.4358
0.1162	1.4495	1.4485	1.4480	1.4466	1.4456	1.4454	1.4443	1.4438	1.4431
0.1687	1.4566	1.4557	1.4551	1.4538	1.4527	1.4524	1.4514	1.4508	1.4500
0.2383	1.4636	1.4628	1.4621	1.4609	1.4602	1.4600	1.4589	1.4581	1.4575
0.3091	1.4707	1.4700	1.4692	1.4681	1.4671	1.4667	1.4658	1.4650	1.4643
0.4032	1.4779	1.4771	1.4763	1.4752	1.4742	1.4740	1.4733	1.4726	1.4718
0.5043	1.4850	1.4842	1.4834	1.4824	1.4816	1.4812	1.4803	1.4795	1.4788
0.6492	1.4922	1.4914	1.4905	1.4896	1.4881	1.4876	1.4868	1.4860	1.4853
0.8251	1.4994	1.4985	1.4976	1.4967	1.4951	1.4946	1.4936	1.4928	1.4920

The behavior of V^E with composition may be qualitatively examined by considering the nature of the component liquids in the pure state and in the mixture. The benzene molecule is reported [34] to have a large quadrupole moment, causing an order in the pure state. The molecules of TEA and TBA are associated through dipole–dipole interactions. On mixing with benzene, TEA and TBA (order destroyers [10]) (*i*) would induce a net decrease in order in benzene and (*ii*) benzene, on the other hand, would cause disruption [8] of dipole–dipole association in TEA and TBA molecules in the solution, resulting in an expansion in volume, and, hence, positive V^E values. Our view is substantiated by Barbosa et al. [6] who have explained the positive excess partial molal volumes of trialkylamines in benzene by considering the factor (*i*), and also by Patel and co-workers [8] who have interpreted the positive V^E values for TEA/TBA in cyclohexane by considering the factor (*ii*).

However, another equally important factor to be considered is the intermolecular interaction of the type n–π between TEA/TBA and benzene molecules. Such a type of interaction between tertiary amines and benzene molecules has also been reported by other researchers [6, 35]. This

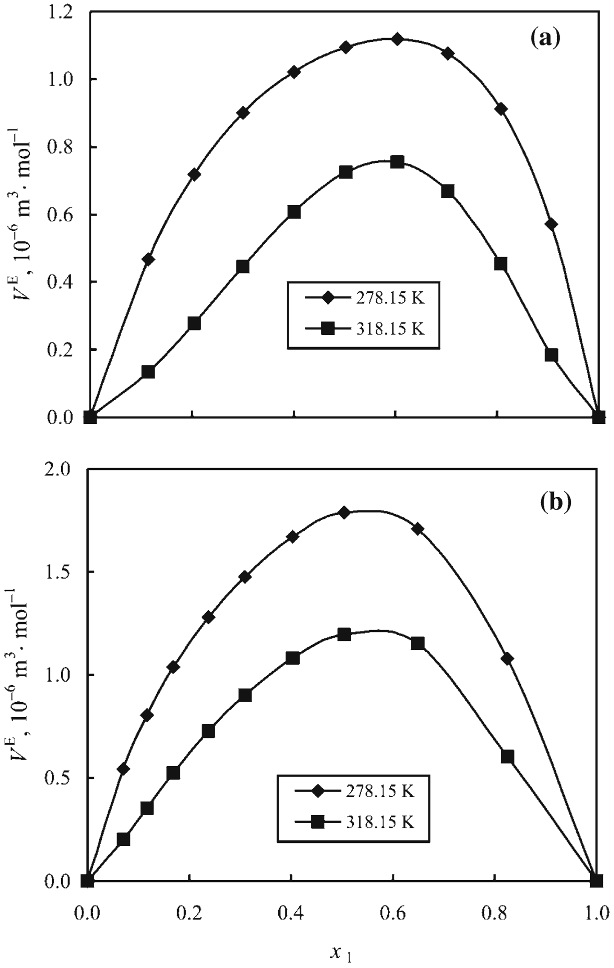

Fig. 1. Variation of excess molar volume, V^E, with mole fraction, x_1, of benzene for (a) benzene + TEA, and (b) benzene + TBA binary mixtures at 278.15 and 318.15 K.

interaction between component molecules is expected to decrease the volume of the system. But, the observed positive V^E values for both mixtures at all the investigated temperatures clearly suggest that the dissociative effects (*i*) and (*ii*) outweigh the effect due to intermolecular interaction. It is interesting to note that at each temperature V^E values for benzene + TBA mixtures are significantly larger than those for benzene + TEA mixtures. This may be due to the fact that the bigger butyl groups –C_4H_9 of TBA would cause much more destruction of order of benzene in solution than relatively smaller ethyl groups $-C_2H_5$ of TEA. As a result, there would be a larger expansion in volume for the benzene + TBA mixture than for benzene + TEA, thereby, yielding larger V^E values for the benzene + TBA mixture.

Furthermore, the presence of steric crowding by three $-C_2H_5$ groups around nitrogen in TEA restricts its closer approach to π-electrons of benzene ring, resulting in weak n–π interactions. This steric crowding (around nitrogen atom in TBA) is further enhanced as three $-C_2H_5$ groups of TEA are replaced by three $-C_4H_9$ groups in TBA, making n–π interactions between TBA and benzene molecules even more weaker. Thus, the steric hindrance, together with the factors (*i*) and (*ii*) cited above would be responsible for larger positive V^E values for benzene + TBA than for benzene + TEA mixtures. Scott et al. [36] also considered the steric hindrance of the alkyl groups of tertiary amines in forming the hydrogen-bonded complexes with proton donors.

As mentioned above and shown in Fig. 1 for both mixtures, V^E values become less positive as the temperature of the system increases from 278.15 to 318.15 K. This indicates that the mixtures tend towards ideal behavior with an increase in temperature. It is worth noting that the second-order thermodynamic mixing volume, dV^E/dT (Table III), with negative values at each mole fraction, clearly suggests the destruction of order in the solution when TEA and TBA are mixed with benzene. This is in accordance with the reported negative dV^E/dT values for the destruction of n-alkane order when mixed with cyclohexane [34]. Furthermore, the values of dV^E/dT for benzene + TBA are more negative than those for benzene + TEA over the whole composition range, suggesting greater destruction of benzene order by TBA molecules than by TEA molecules. Thus, the behaviors of V^E and dV^E/dT are consistent.

In the present work, we have calculated excess molar volumes, V^E, by three different methods. One is calculated using experimental data and the other two from Flory's statistical theory [16, 17], V_F^E, and by using refractive index measurements, $V_{n_D}^E$. Flory's theory, which bears some similarities to the corresponding states theory of Prigogine and co-workers [37], treats the properties of mixtures in terms of reduced properties of the pure components [38]. The theory has proved to give good results for excess thermodynamic properties of liquid mixtures of nonpolar molecules. Here, an attempt is made to apply the theory to mixtures of nonpolar–polar components. According to the Flory equation of state, V_F^E is given as

$$V_F^E = \left[x_1 V_1^* + x_2 V_2^*\right]\left[(\tilde{V}^\circ)^{7/3} / \left(4/3 - (\tilde{V}^\circ)^{1/3}\right)\right](\tilde{T} - \tilde{T}^\circ) \qquad (2)$$

where $\tilde{V}^\circ$ is the ideal reduced volume, calculated by

Table III. Experimental Values of Excess Molar Volume and Theoretical Values Calculated Using the Flory Theory and Refractive Index at 298.15 K and Temperature Dependence of V^{E} for the Binary Mixtures

x_1	$V^{\mathrm{E}}_{\mathrm{Expt}}$	$V^{\mathrm{E}}_{\mathrm{F}}$	$V^{\mathrm{E}}_{n_{\mathrm{D}}}$	dV^{E}/dT ($10^{-6}\,\mathrm{m^3{\cdot}mol^{-1}{\cdot}K^{-1}}$)
	($10^{-6}\,\mathrm{m^3{\cdot}mol^{-1}}$)			
Benzene + Triethylamine				
0.1145	0.2681	0.5232	0.5984	−0.0074
0.2043	0.4847	0.8101	0.9270	−0.0108
0.3005	0.6734	1.0076	1.0717	−0.0112
0.4005	0.8226	1.1037	1.0735	−0.0103
0.5023	0.9147	1.0993	1.0238	−0.0093
0.6037	0.9409	1.0045	0.8991	−0.0091
0.7035	0.8685	0.8350	0.7359	−0.0101
0.8079	0.6558	0.5897	0.4856	−0.0113
0.9069	0.3562	0.3045	0.1979	−0.0095
Benzene + Tributylamine				
0.0702	0.4200	0.1058	−4.1963	−0.0096
0.1162	0.5852	0.1614	−6.5051	−0.0118
0.1687	0.7690	0.2125	−8.4227	−0.0133
0.2383	0.9621	0.2617	−9.8501	−0.0136
0.3091	1.1618	0.2917	−10.706	−0.0148
0.4032	1.3638	0.3046	−10.756	−0.0157
0.5043	1.4723	0.2895	−10.336	−0.0150
0.6492	1.3878	0.2287	−8.1037	−0.0148
0.8251	0.8411	0.1179	−4.6287	−0.0114

$$\widetilde{V}^{\circ} = \phi_1 \widetilde{V}_1 + \phi_2 \widetilde{V}_2 \tag{3}$$

$V_i^* = (V_i/\widetilde{V}_i)$ is the characteristic volume, $\widetilde{V}_i$ is the reduced volume, and V_i is the molar volume of the ith component. $\widetilde{V}_i$ is calculated from the thermal expansion coefficient, $\alpha[= -(1/\rho)(\partial\rho/\partial T)_P]$ using the relation,

$$\widetilde{V}_i = \frac{[1 + (4/3)\alpha_i T]}{(1 + \alpha_i T)^3} \tag{4}$$

The reduced temperature, $\widetilde{T}$ and the ideal reduced temperature, $\widetilde{T}^{\circ}$in Eq. (2) are calculated using the following relations:

$$\widetilde{T}^{\circ} = \frac{(\widetilde{V}^{\circ})^{1/3} - 1}{(\widetilde{V}^{\circ})^{4/3}} \tag{5}$$

$$\widetilde{T}=\frac{\phi_1 P_1^*\widetilde{T}_1+\phi_2 P_2^*\widetilde{T}_2}{\phi_1 P_1^*+\phi_2 P_2^*} \tag{6}$$

The segment or hard-core volume fraction, ϕ_i, in the above equations, is calculated using the relation,

$$\phi_i=x_i V_i^*/\sum_{i=1}^{2} x_i V_i^* \quad \text{and } \phi_1=1-\phi_2 \tag{7}$$

The characteristic pressure, P_i^* is calculated as,

$$P_i^*=T\,\widetilde{V}_i^2\,\alpha_i/k_\mathrm{T} \tag{8}$$

where k_T is the isothermal compressibility, and is computed from the isentropic compressibility, k_s and heat capacity, C_p of liquids using the relation,

$$k_\mathrm{T}=k_\mathrm{s}+TV\alpha^2/C_\mathrm{p} \tag{9}$$

The values of α were calculated from the experimental densities at different temperatures.

Now, for the calculation of V^E from the refractive index measurement of the mixtures, we have used the procedure adopted by Fucaloro [39] who has estimated the partial molar volumes of binary liquid mixtures by measuring the index of refraction of pure liquids and their mixtures. Assuming the molar refraction, R_m of a mixture to be a linear function of mole fraction, x_i, it can be calculated by using the relation,

$$R_\mathrm{m}=x_1R_1+x_2R_2=\frac{M_\mathrm{eff}}{\rho}\left[\frac{(n_\mathrm{D}^2-1)}{(n_\mathrm{D}^2+2)}\right] \tag{10}$$

where R_i is the molar refraction of pure component i. M_eff is the effective molar mass of the mixture, calculated as

$$M_\mathrm{eff}=x_1M_1+x_2M_2 \tag{11}$$

Thus, knowing the densities and refractive indices of the pure components, one can estimate the density of a mixture by measuring its refractive index using Eq. (10). Using this density, one can easily estimate the excess molar volume, $V_{n_\mathrm{D}}^\mathrm{E}$ of the mixtures with the help of Eq. (1).

The values of excess molar volumes determined experimentally, predicted by Flory's theory, and estimated by using refractive index measurements, are reported in Table III and are graphically shown in Fig. 2 at

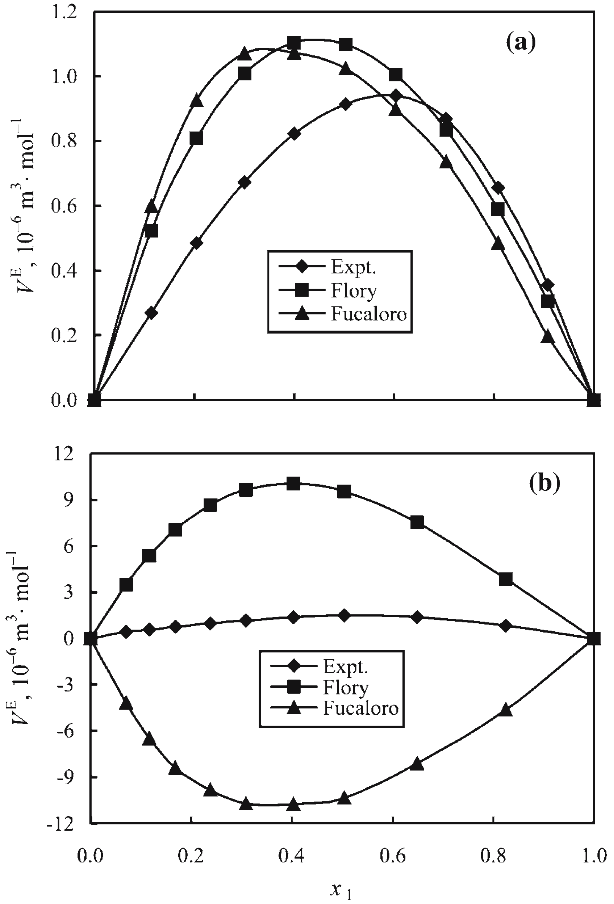

Fig. 2. Variation of experimental and theoretical excess molar volume, V^E, with mole fraction, x_1, of benzene for (a) benzene + TEA, and (b) benzene + TBA binary mixtures at 298.15 K.

298.15 K. It is clear from Fig. 2 that both V_F^E and $V_{n_D}^E$ qualitatively support the experimental values of V^E and, thus, predict the trends observed for benzene + TEA mixtures. For benzene + TBA mixtures, however, V_F^E is in reasonable agreement with the experimental V^E values while $V_{n_D}^E$ shows negative deviations from V^E values. Thus, we conclude that Flory's theory seems to be reasonable in predicting the excess molar volumes of the present binary mixtures.

ACKNOWLEDGMENTS

One of the authors AKN is thankful to the Department of Science and Technology, New Delhi for the award of a Fast Track Young Scientist Scheme.

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