Excess Molar Volumes and Viscosity Deviations of Binary Liquid Mixtures of 1,3-Dioxolane and 1,4-Dioxane with Butyl Acetate, Butyric Acid, Butylamine, and 2-Butanone at 298.15 K

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Densities and viscosities were measured for the binary mixtures of 1,4-dioxane and 1,3-dioxolane with butyl acetate, butyric acid, butylamine, and 2-butanone at 298.15 K over the entire composition range. From density and viscosity data, the values of excess molar volume (V^E) and viscosity deviation ($\Delta\eta$) have been determined. These results were fitted to Redlich−Kister type polynomial equations. The density and viscosity data have been analyzed in terms of some semiempirical viscosity models.

Introduction

Studies on thermodynamic and transport properties are important in understanding the nature of molecular interactions in binary liquid mixtures. Properties of mixtures are useful for designing many types of transport and process equipment in the chemical industry.[1−5] 1,4-Dioxane, 1,3-dioxolane, and their binary liquid mixtures are important solvents widely used in various industries. 1,4-Dioxane and 1,3-dioxolane are cyclic diethers differing in one methylene group, thus they differ in quadrupolar and dipolar order.[6] In this work we report density and viscosity for {1,4-dioxane or 1,3-dioxolane + butyl acetate, butyric acid, butylamine, or 2-butanone} mixtures at 298.15 K.

Expermental Section

Materials. 1,4-Dioxane (Merck, India) was kept several days over potassium hydroxide (KOH), refluxed for 24 h, and distilled over lithium aluminum hydride ($LiAlH_4$). Details have been described earlier.[1] 1,3-Dioxolane (LR) was purified by standard methods.[7] It was refluxed with PbO_2 and fractionally distilled after the addition of xylene. Butyl acetate, butyric acid, butylamine, and 2-butanone (S.D. Fine Chemicals, Analytical Reagent, purity > 99 %) were used without further purification. The purity of the solvents was ascertained by GLC and also by comparing experimental values of densities and viscosities with those reported in the literature[8−13] as listed in Table 1.

Apparatus and Procedure. The densities were measured with an Ostwald-Sprengel type pycnometer having a bulb volume of 25 cm^3 and an internal diameter of the capillary of about 0.1 cm. The pycnometer was calibrated at 298.15 K with double-distilled water and benzene. The pycnometer with the test solution was equilibrated in a water bath maintained at ± 0.01 K of the desired temperature by means of a mercury in glass thermoregulator, and the absolute temperature was determined by a calibrated platinum resistant thermometer and Muller bridge. The pycnometer was then removed from the thermostatic bath, properly dried, and weighed. The evaporation losses remained insignificant during the time of actual measurements. An average of triplicate measurements was taken into account.

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Table 1. Comparison of Density ρ and Viscosity η of the Pure Liquids with Literature Data at 298.15 K

	ρ/(g·cm^{-3})		η/(mPa·s)	
pure liquid	expt.	lit.	expt.	lit.
1,4-dioxane	1.0265	1.0278[8]	1.196	1.196[8]
1,3-dioxolane	1.0571	1.0587[9]	0.531	
butyl acetate	0.8744	0.8761[10]	0.668	0.674[10]
butyric acid	0.9528		1.396	
butylamine	0.7319	0.7331[11]	0.493	0.496[12]
2-butanone	0.7981	0.7996[13]	0.373	0.378[13]

The mixtures were prepared by mixing known volume of pure liquids in airtight-stoppered bottles. The reproducibility in mole fraction was within ± 0.0002. The mass measurements accurate to ± 0.01 mg were made on a digital electronic analytical balance (Mettler, AG 285, Switzerland). The total uncertainty of density is ± 0.0001 g·cm^{-3} and that of the temperature is ± 0.01 K.

The viscosity was measured by means of a suspended Ubbelohde type viscometer, which was calibrated at 298.15 K with triple-distilled water and purified methanol using density and viscosity values from the literature. The flow times were accurate to ± 0.1 s, and the uncertainty in the viscosity measurements, based on our work on several pure liquids, was within ± 0.03 % of the reported value. Details of the methods and techniques of density and viscosity measurements have been described earlier.[4,14]

Results and Discussion

The experimental viscosities, densities, excess volumes (V^E), and viscosity deviations ($\Delta\eta$) for the binary mixtures studied at 298.15 K are listed in Table 2.

Excess Molar Volume. The excess molar volumes, V^E, were calculated using the equation

$$V^E = \sum_{i=1}^{j} x_i M_i \left(\frac{1}{\rho} - \frac{1}{\rho_i}\right) \quad (1)$$

where ρ is the density of the mixture; M_i, x_i, and ρ_i are the molecular weight, mole fraction, and density of ith component in the mixture, respectively. The values of excess molar volume,

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Table 2. Values of Density ρ, Viscosity η, Excess Molar Volume V^E, Viscosity Deviation $\Delta\eta$, and Grunberg−Nissan Interaction Parameter d_{12} for the Binary Mixtures of 1,4-Dioxane or 1,3-Dioxolane at 298.15 K

x_1	ρ / g·cm^{-3}	η / mPa·s	$V^E \times 10^6$ / m^3·mol^{-1}	$\Delta\eta$ / mPa·s	d_{12}	T_{12}	H_{12}	x_1	ρ / g·cm^{-3}	η / mPa·s	$V^E \times 10^6$ / m^3·mol^{-1}	$\Delta\eta$ / mPa·s	d_{12}	T_{12}	H_{12}
1,3-Dioxolane (1) + 2-Butanone (2)															
0	0.7981	0.373	0	0				0.5935	0.9381	0.456	−0.207	−0.010	−0.0311	0.4447	0.4306
0.0976	0.8189	0.384	−0.084	−0.004	−0.0479	0.4405	0.4293	0.6943	0.9655	0.473	−0.173	−0.009	−0.0330	0.4444	0.4297
0.1957	0.8405	0.397	−0.133	−0.006	−0.0368	0.4430	0.4311	0.7956	0.9942	0.491	−0.113	−0.007	−0.0352	0.4440	0.4298
0.2944	0.8631	0.411	−0.164	−0.008	−0.0276	0.4451	0.4325	0.8975	1.0245	0.510	−0.045	−0.004	−0.0366	0.4438	0.4279
0.3935	0.8868	0.426	−0.185	−0.009	−0.0260	0.4455	0.4325	1	1.0571	0.531	0	0			
0.4933	0.9118	0.441	−0.201	−0.010	−0.0273	0.4454	0.4319								
1,3-Dioxolane (1) + Butyric acid (2)															
0	0.9528	1.396	0	0				0.6408	1.0169	0.854	−0.317	0.012	0.5576	0.9389	0.9906
0.1167	0.9631	1.297	−0.067	0.001	0.3771	0.9201	0.9703	0.7351	1.0272	0.770	−0.270	0.010	0.5965	0.9373	0.9897
0.2292	0.9737	1.202	−0.150	0.004	0.4054	0.9243	0.9743	0.8263	1.0374	0.688	−0.206	0.007	0.6393	0.9353	0.9882
0.3376	0.9843	1.111	−0.209	0.007	0.4372	0.9283	0.9784	0.9146	1.0473	0.608	−0.113	0.003	0.6751	0.9297	0.9827
0.4422	0.9953	1.023	−0.276	0.010	0.4735	0.9325	0.9829	1	1.0571	0.531	0	0			
0.5432	1.0061	0.938	−0.302	0.012	0.5140	0.9363	0.9873								
1,3-Dioxolane (1) + Butyl acetate (2)															
0	0.8744	0.668	0	0				0.7017	0.9761	0.571	−0.050	−0.001	0.0224	0.6068	0.5981
0.1484	0.8907	0.621	−0.128	−0.027	−0.3078	0.4728	0.4939	0.7853	0.9952	0.565	−0.034	0.005	0.0783	0.6204	0.6138
0.2816	0.9069	0.599	−0.149	−0.031	−0.2239	0.5167	0.5230	0.8625	1.0150	0.556	−0.023	0.006	0.1211	0.6291	0.6253
0.4019	0.9233	0.588	−0.129	−0.025	−0.1506	0.5490	0.5465	0.9338	1.0356	0.544	−0.012	0.004	0.1568	0.6349	0.6344
0.5111	0.9403	0.583	−0.103	−0.015	−0.0771	0.5761	0.5690	1	1.0571	0.531	0	0			
0.6106	0.9578	0.577	−0.072	−0.007	−0.0230	0.5939	0.5851								
1,3-Dioxolane (1) + Butylamine (2)															
0	0.7319	0.493	0	0				0.6002	0.8968	0.464	0.077	−0.052	−0.4384	0.4185	0.4041
0.1001	0.7551	0.477	0.013	−0.020	−0.4626	0.3922	0.3988	0.7002	0.9321	0.478	0.058	−0.041	−0.3899	0.4319	0.4142
0.2001	0.7796	0.463	0.038	−0.038	−0.0489	0.3907	0.3937	0.8001	0.9703	0.496	0.039	−0.027	−0.3285	0.4478	0.4277
0.3002	0.8058	0.458	0.065	−0.047	−0.4628	0.4022	0.4001	0.9001	1.0118	0.513	0.021	−0.013	−0.2860	0.4590	0.4370
0.4002	0.8338	0.455	0.082	−0.054	−0.4630	0.4062	0.4002	1	1.0571	0.531	0	0			
0.5002	0.8641	0.456	0.085	−0.056	−0.4606	0.4103	0.4003								
1,4-Dioxane (1) + 2-Butanone (2)															
0	0.7981	0.373	0	0				0.5511	0.9233	0.576	−0.205	−0.250	−0.8356	0.2909	0.2789
0.0833	0.8170	0.391	−0.077	−0.050	−0.632	0.4613	0.4576	0.6563	0.9472	0.650	−0.167	−0.263	−0.9285	0.2162	0.2009
0.1698	0.8365	0.411	−0.124	−0.102	−0.7139	0.4283	0.4237	0.7660	0.9722	0.751	−0.111	−0.252	−1.0713	0.1022	0.0821
0.2597	0.8568	0.436	−0.160	−0.150	−0.7555	0.3997	0.3938	0.8805	0.9985	0.901	−0.044	−0.197	−1.3692	−0.1215	−0.1502
0.3530	0.8779	0.464	−0.186	−0.200	−0.8467	0.3545	0.3472	1	1.0265	1.196	0	0			
0.4501	0.9001	0.512	−0.202	−0.231	−0.8346	0.3275	0.3180								
1,4-Dioxane (1) + Butyric Acid (2)															
0	0.9528	1.396	0	0				0.6000	1.0002	1.443	−0.395	0.167	0.5245	1.6385	1.6437
0.1000	0.9617	1.504	−0.183	0.128	0.9998	2.0259	2.0071	0.7000	1.0068	1.362	−0.307	0.106	0.3978	1.5416	1.5481
0.2000	0.9703	1.570	−0.332	0.214	0.9268	1.9767	1.9642	0.8000	1.0131	1.293	−0.184	0.057	0.2938	1.4670	1.4737
0.3000	0.9784	1.587	−0.420	0.251	0.8320	1.9004	1.8939	0.9000	1.0194	1.241	−0.065	0.026	0.2459	1.4322	1.4392
0.4000	0.9861	1.572	−0.467	0.256	0.7531	1.8312	1.8297	1	1.0265	1.196	0	0			
0.5000	0.9934	1.507	−0.456	0.211	0.6163	1.7161	1.7186								
1,4-Dioxane (1) + Butyl Acetate (2)															
0	0.8744	0.668	0	0				0.6641	0.9594	0.822	0.036	−0.196	−0.8028	0.5936	0.4920
0.1278	0.8875	0.682	0.005	−0.054	−0.4852	0.7261	0.6911	0.7547	0.9753	0.866	0.032	−0.201	−0.9734	0.5184	0.3903
0.2479	0.9010	0.692	0.014	−0.107	−0.5890	0.6856	0.6441	0.8406	0.9918	0.939	0.022	−0.172	−1.1077	0.4510	0.2898
0.3610	0.9149	0.708	0.023	−0.151	−0.6628	0.6570	0.6045	0.9223	1.0089	1.035	0.011	−0.119	−1.3746	0.3162	0.1013
0.4678	0.9292	0.739	0.032	−0.176	−0.6871	0.6462	0.5794	1	1.0265	1.196	0	0			
0.5687	0.9441	0.779	0.036	−0.189	−0.7249	0.6287	0.5459								
1,4-Dioxane (1) + Butylamine (2)															
0	0.7319	0.493	0	0				0.5579	0.8828	0.637	0.147	−0.248	−0.9672	0.3762	0.3411
0.0855	0.7534	0.509	0.020	−0.045	−0.5766	0.5699	0.5586	0.6625	0.9147	0.699	0.127	−0.259	−1.0620	0.3105	0.2648
0.1738	0.7761	0.524	0.052	−0.091	−0.6484	0.5408	0.5271	0.7709	0.9492	0.792	0.080	−0.243	−1.1845	0.2176	0.1570
0.2650	0.8001	0.541	0.089	−0.139	−0.7327	0.5055	0.4886	0.8834	0.9864	0.941	0.039	−0.172	−1.3201	0.0898	0.0074
0.3594	0.8258	0.560	0.117	−0.186	−0.8304	0.4626	0.4414	1	1.0265	1.196	0	0			
0.4569	0.8533	0.590	0.141	−0.224	−0.9088	0.4199	0.3927								

V^E, have been presented in Table 2. The estimated uncertainty for excess molar volumes, V^E, is ± 0.005 cm^3·mol^{-1}.

The excess molar volumes, V^E, for all the 1,3-dioxolane systems, except for the system involving butylamine, are negative over the entire range of composition. The negative values of excess volumes for the three systems are in the order:

1,3-dioxolane + butyric acid > 1, 3-dioxolane + 2-butanone > 1,3-dioxolane + butyl acetate

The excess molar volumes for the two 1,4-dioxane systems involving butylamine and butyl acetate are positive, while the other two 1,4-dioxane systems involving 2-butanone and butyric acid are negative. The order of V^E values for the 1,4-dioxane systems irrespective of their sign is as follows:

1,4-dioxane + butylamine > 1,4-dioxane + butyl acetate > 1,4-dioxane + 2-butabnone > 1,4-dioxane + butyric acid

The negative values of excess molar volume suggest specific interactions[15] between the mixing components in the mixtures while its positive values suggest dominance of dispersion

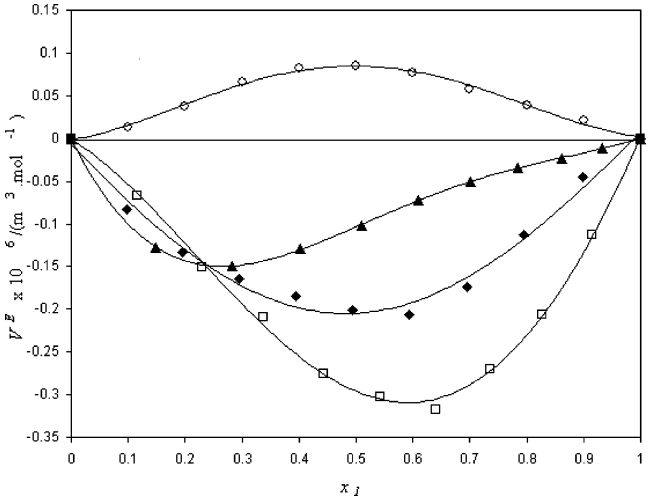

Figure 1. Excess molar volumes V^E at 298.15 K for binary mixtures of 1,3-dioxolane (1) with ◆, 2-butanone; □, butyric acid; ▲, butyl acetate; and ○, butylamine.

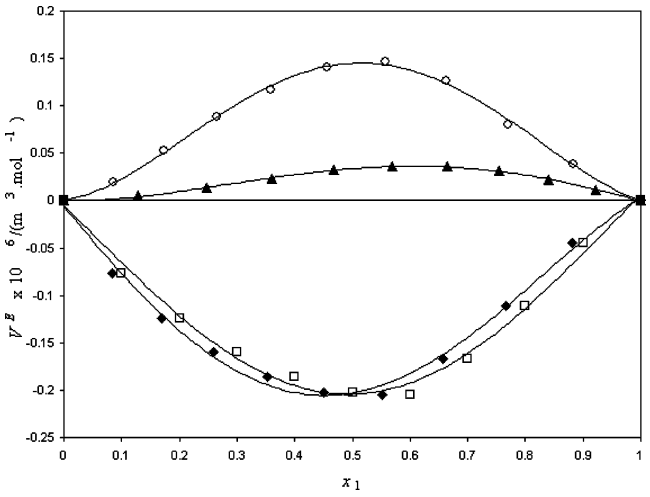

Figure 2. Excess molar volumes V^E at 298.15 K for binary mixtures of 1, 4-dioxane (1) with ◆, 2-butanone; □, butyric acid; ▲, butyl acetate; and ○, butylamine.

forces[16,13] between them. The negative V^E values indicate the specific interactions such as intermolecular hydrogen bonding between the mixing components and also the interstitial accommodation of the mixing components because of the difference in molar volume. The negative V^E values may also be due to the difference in the dielectric constants of the liquid components of the binary mixture.[15] The negative V^E values for all the systems studied may be attributed to dipole−induced dipole interactions between the components liquids of the mixtures resulting in the formation of electron donor−acceptor complexes.[17] The plots of excess molar volume, V^E, versus mole fraction, x_1. of 1,3-dioxolane or 1,4-dioxane are presented in Figures 1 and 2.

Viscosity Deviation. The deviation in viscosities, $\Delta\eta$, was computed using the relationship[18]

$$\Delta\eta = \eta - \sum_{i=1}^{j} (x_i\eta_i) \qquad (2)$$

where η is the dynamic viscosities of the mixture; x_i and η_i are the mole fraction and viscosity of *i*th component in the mixture, respectively.

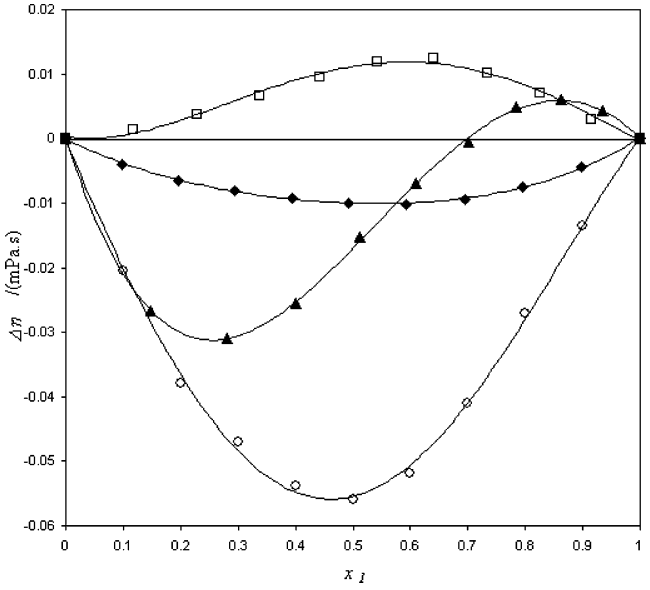

Figure 3. Viscosity deviations $\Delta\eta$ at 298.15 K for the binary mixtures of 1,3-dioxolane (1) with ◆, 2-butanone; □, butyric acid; ▲, butyl acetate; and ○, butylamine.

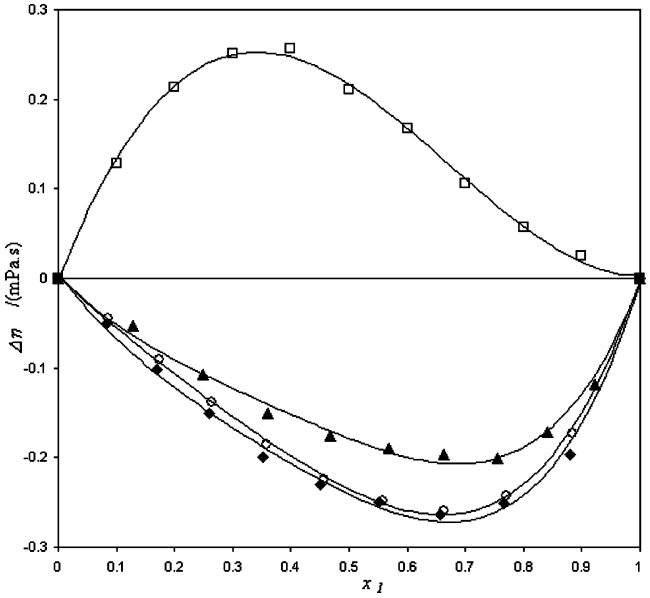

Figure 4. Viscosity deviations $\Delta\eta$ at 298.15 K for the binary mixtures of 1,4-dioxane (1) with ◆, 2-butanone; □, butyric acid; ▲, butyl acetate; and ○, butylamine.

A perusal of Table 2 shows that the values of viscosity deviation, $\Delta\eta$, are negative over the entire composition range for all the binary liquid mixtures studied except for the mixtures involving butyric acid. The estimated uncertainty for viscosity deviation, $\Delta\eta$, is ± 0.004 mPa·s. The negative values imply the presence of dispersion forces[18] between the mixing components in these mixtures, while positive values may be attributed to the presence of specific interactions[18] between them. The plots of viscosity deviation, $\Delta\eta$, versus mole fraction, x_1, for the different binary mixtures have been presented in Figures 3 and 4.

Viscosity Models and Interaction Parameters. Several semiempirical models have been proposed to estimate the dynamic viscosity (η) of the binary liquid mixtures in terms of pure-component data.[19,20] Some of them we examined are as follows:

Table 3. Values of Coefficients a_I of Equation 6 and Standard Deviations σ for Excess Properties of the Binary Mixtures of 1, 3-Dioxolane (1) or 1,4-Dioxane (1) Studied at 298.15 K

binary mixture	excess property	a_0	a_1	a_2	a_3	a_4	σ
1,3-dioxolane + 2-butanone	$V^E \times 10^6/(m^3 \cdot mol^{-1})$	−0.798	0.084				0.013
	$\Delta\eta$/(mPa·s)	−0.040	−0.009	−0.009	0.129		0.001
1,3-dioxolane + butyric acid	$V^E \times 10^6/(m^3 \cdot mol^{-1})$	−1.184	−0.640	0.215	0.253		0.006
	$\Delta\eta$/(mPa·s)	0.044	0.040	−0.027	−0.037		0.001
1,3-dioxolane + butyl acetate	$V^E \times 10^6/(m^3 \cdot mol^{-1})$	−0.414	0.580	−0.371			0.001
	$\Delta\eta$/(mPa·s)	−0.068	0.183	−0.029			0.001
1,3-dioxolane + butylamine	$V^E \times 10^6/(m^3 \cdot mol^{-1})$	0.342	−0.070	−0.321	0.200	0.123	0.001
	$\Delta\eta$/(mPa·s)	−0.222	0.031	0.056	0.040	−	0.001
1,4-dioxane + 2-butanone	$V^E \times 10^6/(m^3 \cdot mol^{-1})$	−0.824	0.063	0.442	0.486	−0.337	0.003
	$\Delta\eta$/(mPa·s)	−0.972	−0.422	−0.315	−0.623	−0.353	0.002
1,4-dioxane + butyric acid	$V^E \times 10^6/(m^3 \cdot mol^{-1})$	−1.816	0.673	0.454	0.234	0.343	0.004
	$\Delta\eta$/(mPa·s)	0.859	−0.819	−	−	−	0.006
1,4-dioxane + butyl acetate	$V^E \times 10^6/(m^3 \cdot mol^{-1})$	0.135	0.112	−0.055	−0.063	−	0.001
	$\Delta\eta$/(mPa·s)	−0.720	−0.319	−0.408	−0.582	−	0.003
1,4-dioxane + butylamine	$V^E \times 10^6/(m^3 \cdot mol^{-1})$	0.579	0.093	−0.443	−0.105	−	0.003
	$\Delta\eta$/(mPa·s)	−0.944	−0.527	−0.247	−0.469	−0.122	0.001

Grunberg and Nissan[21] have suggested the following logarithmic relation between the viscosity of the binary mixtures and the pure components:

$$\eta = \exp\left[\sum_{i=1}^{j} (x_i \ln \eta_i) + d_{12}\prod_{i=1}^{j} x_i\right] \quad (3)$$

where d_{12} is a constant proportional to the interchange energy. It may be regarded as an approximate measure of the strength of molecular interactions between the mixing components. The values of the interchange parameter d_{12} have been calculated as a function of the composition of the binary liquid mixtures of 1,3-dioxolane and 1,4-dioxane with butyl acetate, butyric acid, butylamine, and 2-butanone as listed in Table 2.

It has been found that the values of d_{12} are negative for all the binary systems studied, except the systems involving butyric acid. The negative values of d_{12} indicate the presence of dispersion forces[18] between the mixing components in the mixtures while its positive values indicate the presence of specific interactions[18] between them.

Tamura and Kurata[22] put forward the following equation for the viscosity of the binary liquid mixtures:

$$\eta = \sum_{i=1}^{j} x_i\phi_i\eta_i + 2T_{12}\prod_{i=1}^{j}[x_i\phi_i]^{1/2} \quad (4)$$

where T_{12} is the interaction parameter and ϕ_i is the volume fraction of ith pure component in the mixture.

Molecular interactions may also be interpreted by the following viscosity model of Hind et al.:[23]

$$\eta = \sum x_i^2\eta_i + 2H_{12}\prod_{i=1}^{j} x_i \quad (5)$$

where H_{12} is Hind interaction parameter, which may be attributed to unlike pair interaction.[24] In the present study, the values of interaction parameter T_{12} and H_{12} have been calculated from eqs 4 and 5, respectively, and are listed in Table 2. It has been observed that for a given binary mixture T_{12} and H_{12} do not differ appreciably from each other, and this is in agreement with the view put forward by Fort and Moore[18] in regard to the nature of parameters T_{12} and H_{12}. It is also significant to note that the values of T_{12} and H_{12} are larger for the binary mixtures of butyric acid with both the cyclic ethers, which involve specific molecular interactions[25] as revealed by the positive values of $\Delta\eta$ and d_{12}.

Redlich−Kister Polynomial Equation. The excess properties (V^E and $\Delta\eta$) were fitted to the Redlich−Kister polynomial equation:[26]

$$Y^E = x_1x_2\sum_{i=1}^{K} a_i(x_1 - x_2)^i \quad (6)$$

where Y^E refers to excess properties; x_1 and x_2 are the mole fraction 1,3-dioxolane or 1,4-dioxane and the other component, respectively. The coefficients a_i were obtained by fitting eq 6 to experimental results using a least-squares regression method. In each case, the optimal number of coefficients was ascertained from an approximation of the variation in the standard deviation (σ). The calculated values of a_i along with the tabulated standard deviations (σ) are listed in Table 3. The standard deviation (σ) was calculated using

$$\sigma = [(Y^E_{exp} - Y^E_{cal})^2/(n - m)]^{1/2} \quad (7)$$

where n is the number of data points and m is the number of coefficients. The σ values lies between 0.013 $m^3 \cdot mol^{-1}$ and 0.001 $m^3 \cdot mol^{-1}$ for V^E and between 0.006 mPa·s and 0.001 mPa·s for $\Delta\eta$, respectively. The largest σ values corresponds to the 1,3-dioxolane + 2-butanone system for V^E and the 1,4-dioxane + butyric acid for $\Delta\eta$, respectively. In the present study, V^E and $\Delta\eta$ are quite systematic and a function of the composition of the binary mixtures.

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