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Excess Molar Volumes and Deviations in Viscosity of Binary Mixtures of *N*,*N*-Dimethylformamide with Aniline and Benzonitrile at (298.15, 303.15, 308.15, and 313.15) K

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Densities and viscosities of binary mixtures of *N*,*N*-dimethylformamide (DMF) with aniline and benzonitrile have been measured at (298.15, 303.15, 308.15, and 313.15) K. From these data, excess molar volumes (V^E) and deviations in viscosity ($\Delta\eta$) have been calculated. Negative excess molar volumes and negative deviations in viscosity for DMF + aniline systems are attributed to the interstitial accommodation of DMF molecules into clusters of aniline. Negative excess molar volumes and positive deviations in viscosity for DMF + benzonitrile systems are due to the strong specific interactions. The excess molar volumes and deviations in viscosity are fitted to the Redlich−Kister polynomial equation.

Introduction

Thermodynamic and transport properties of binary liquid mixtures containing protic, aprotic, and associated liquids have been studied previously.[1−5] The calculated excess quantities from such data have been interpreted in terms of differences in size of the molecules and strength of the specific and nonspecific interactions taking place between the components of the mixtures. In the present study, interactions of DMF with aniline and benzonitrile have been reported. DMF is aprotic and unassociated[6] in its pure liquid state. It belongs to the so-called supersolvents, owing to its miscibility with almost all common polar and nonpolar solvents,[7] probably due to its high polarity with large dipole moment ($\mu = 3.8$ D) and moderately high dielectric constant ($\epsilon = 36.76$) at 298.15 K.[8] Aniline exists in associated form whereas benzonitrile has a nonassociated structure in the liquid state. When DMF is mixed with aniline and benzonitrile, mixing properties with varing intermolecular interactions may be generated. The transport and thermodynamic studies on the binary mixtures containing aniline and benzonitrile have been reported previously by us.[9−13] Gill et al.[14] reported densities and viscosities of benzonitrile + DMF mixtures at 298.15 K only. There are few reports[15,16]on the density and viscosity data for binary mixtures of DMF with aniline. Therefore, in the present paper we report density and viscosity data for binary systems formed by DMF with aniline and with benzonitrile over the entire range of composition and at (298.15, 303.15, 308.15, and 313.15) K.

Experimental Section

Aniline (S.D. Fine Chemicals, purity > 99%) was dried over caustic potash for about 2 days and distilled twice. Benzonitrile (Lancaster Estgute, Morecamble, England, purity > 99%) was dried over anhydrous calcium chloride for 2 days and then distilled at atmospheric pressure. DMF (S.D. Fine Chemicals, analytical reagent, purity > 99.5%) was directly used. The purity of solvents was ascertained by GLC and also by comparing experimental values of densities and viscosities with those reported in the literature (Table 1). Our experimental values of densities and viscosities match very well with those reported in the literature. Binary mixtures were prepared by mixing a known mass of each liquid in an airtight, stoppered glass bottle. The masses were recorded on a Adairdutt balance to an accuracy of ±1 × 10^{-4} g. The estimated error in mole fraction was <1 × 10^{-4}. Care was taken to avoid contamination during mixing.

Table 1. Comparison of Experimental Densities, ρ, and Viscosities, η, of Pure Liquids with Literature Values at (298.15, 303.15, 308.15, and 313.15) K

		ρ/g·cm^{-3}		η/mPa·s	
liquid	T/K	expt	lit.	expt	lit.
DMF	298.15	0.9445	0.9445[22]	0.803	0.802[21]
	303.15	0.9398	0.9397[23]	0.756	0.752[25]
	308.15	0.9351	0.9356[23]	0.710	0.707[25]
	313.15	0.9302	0.9298[24]	0.673	0.664[25]
aniline	298.15	1.0172	1.0173[26]	3.690	3.770[21]
	303.15	1.0129	1.0131[26]	3.190	3.170[26]
	308.15	1.0086	1.0088[26]	2.800	2.770[27]
	313.15	1.0042	1.0045[26]	2.420	2.400[26]
benzonitrile	298.15	1.0009	1.0006[26]	1.171	1.237[28]
	303.15	0.9964	0.9963[26]	1.126	1.111[26]
	308.15	0.9919	0.9918[28]	1.068	1.063[12]
	313.15	0.9872	0.9862[29]	1.001	0.993[12]

The densities of degassed pure liquids and binary mixtures were measured using a 15 cm^3 double-arm pycnometer, as reported earlier,[17] in a transparent glass-walled water bath having a thermal stability of ±0.01 K. The pycnometer was calibrated using conductivity water[18] having a conductivity of <1 × 10^{-6} Ω^{-1} cm^{-1}. Uncertainties in density measurement were within ±1 × 10^{-4} g·cm^{-3}.

Dynamic viscosities were measured using an Ubbelohde suspended-level viscometer,[17] calibrated with conductivity water.[18] An electronic digital stopwatch with accuracy to ±0.01 s was used for flow time measurements. At least three repetitions of each data point obtained were reproducible to ±0.05 s, and results were averaged. Since all flow times were >300 s, kinetic energy corrections were

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Table 2. Density, ρ, Viscosity, η, Excess Molar Volume, V^E, and Deviations in Viscosity, $\Delta\eta$, for DMF (1) + Aniline (2) and DMF (1) + Benzonitrile (2) at (298.15, 303.15, 308.15, and 313.15) K

x_1	$10^3\rho$/kg·m^{-3}	η/mPa·s	V^E/cm^3·mol^{-1}	$\Delta\eta$/mPa·s	x_1	$10^3\rho$/kg·m^{-3}	η/mPa·s	V^E/cm^3·mol^{-1}	$\Delta\eta$/mPa·s
DMF (1) + Aniline (2); 298.15 K									
0.0000	1.0172	3.690	0.000	0.000	0.6614	0.9788	1.731	−0.577	−0.050
0.1293	1.0103	3.296	−0.107	−0.021	0.7558	0.9692	1.466	−0.383	−0.042
0.2582	1.0058	2.908	−0.448	−0.037	0.8302	0.9614	1.257	−0.227	−0.036
0.3548	1.0005	2.621	−0.551	−0.045	0.9123	0.9523	1.038	−0.031	−0.018
0.4642	0.9939	2.300	−0.635	−0.050	1.0000	0.9445	0.803	0.000	0.000
0.5537	0.9876	2.040	−0.645	−0.051					
DMF (1) + Aniline (2); 303.15 K									
0.0000	1.0129	3.190	0.000	0.000	0.6614	0.9743	1.543	−0.588	−0.037
0.1293	1.0060	2.864	−0.113	−0.011	0.7558	0.9648	1.319	−0.404	−0.031
0.2582	1.0015	2.540	−0.461	−0.022	0.8302	0.9570	1.145	−0.249	−0.024
0.3548	0.9962	2.298	−0.568	−0.028	0.9123	0.9477	0.955	−0.037	−0.014
0.4642	0.9895	2.025	−0.648	−0.035	1.0000	0.9398	0.756	0.000	0.000
0.5537	0.9832	1.803	−0.662	−0.039					
DMF (1) + Aniline (2); 308.15 K									
0.0000	1.0086	2.800	0.000	0.000	0.6614	0.9698	1.387	−0.599	−0.031
0.1293	1.0017	2.521	−0.118	−0.009	0.7558	0.9604	1.194	−0.425	−0.026
0.2582	0.9972	2.242	−0.474	−0.018	0.8302	0.9526	1.044	−0.270	−0.021
0.3548	0.9919	2.034	−0.585	−0.024	0.9123	0.9431	0.880	−0.042	−0.013
0.4642	0.9851	1.800	−0.661	−0.030	1.0000	0.9351	0.710	0.000	0.000
0.5537	0.9789	1.611	−0.686	−0.032					
DMF (1) + Aniline (2); 313.15 K									
0.0000	1.0042	2.420	0.000	0.000	0.6614	0.9653	1.237	−0.624	−0.028
0.1293	0.9973	2.186	−0.134	−0.008	0.7558	0.9559	1.079	−0.452	−0.021
0.2582	0.9929	1.952	−0.498	−0.017	0.8302	0.9481	0.954	−0.300	−0.016
0.3548	0.9876	1.779	−0.615	−0.021	0.9123	0.9384	0.819	−0.056	−0.007
0.4642	0.9807	1.583	−0.688	−0.026	1.0000	0.9302	0.673	0.000	0.000
0.5537	0.9745	1.423	−0.716	−0.030					
DMF (1) + Benzonitrile (2); 298.15 K									
0.0000	1.0009	1.171	0.000	0.000	0.6061	0.9722	1.049	−0.138	0.101
0.1335	0.9954	1.170	−0.035	0.048	0.7176	0.9652	0.992	−0.115	0.085
0.1981	0.9927	1.169	−0.062	0.071	0.7997	0.9596	0.943	−0.086	0.066
0.3191	0.9874	1.152	−0.114	0.098	0.9209	0.9506	0.862	−0.026	0.030
0.3873	0.9843	1.134	−0.147	0.106	1.0000	0.9445	0.803	0.000	0.000
0.4907	0.9790	1.099	−0.164	0.109					
DMF (1) + Benzonitrile (2); 303.15 K									
0.0000	0.9964	1.126	0.000	0.000	0.6061	0.9677	0.992	−0.150	0.090
0.1335	0.9909	1.124	−0.038	0.047	0.7176	0.9606	0.936	−0.119	0.076
0.1981	0.9882	1.119	−0.066	0.066	0.7997	0.9550	0.889	−0.091	0.059
0.3191	0.9829	1.097	−0.120	0.089	0.9209	0.9460	0.811	−0.033	0.026
0.3873	0.9798	1.078	−0.155	0.095	1.0000	0.9398	0.756	0.000	0.000
0.4907	0.9745	1.042	−0.174	0.098					
DMF (1) + Benzonitrile (2); 308.15 K									
0.0000	0.9919	1.068	0.000	0.000	0.6061	0.9632	0.932	−0.162	0.081
0.1335	0.9864	1.064	−0.040	0.044	0.7176	0.9560	0.879	−0.124	0.068
0.1981	0.9837	1.056	−0.070	0.059	0.7997	0.9504	0.834	−0.097	0.052
0.3191	0.9784	1.033	−0.127	0.079	0.9209	0.9414	0.761	−0.041	0.023
0.3873	0.9753	1.015	−0.164	0.086	1.0000	0.9351	0.710	0.000	0.000
0.4907	0.9700	0.980	−0.185	0.088					
DMF (1) + Benzonitrile (2); 313.15 K									
0.0000	0.9872	1.001	0.000	0.000	0.6061	0.9585	0.875	−0.174	0.073
0.1335	0.9817	0.990	−0.043	0.033	0.7176	0.9512	0.825	−0.128	0.059
0.1981	0.9790	0.985	−0.074	0.049	0.7997	0.9456	0.781	−0.103	0.042
0.3191	0.9738	0.963	−0.144	0.067	0.9209	0.9365	0.715	−0.039	0.016
0.3873	0.9706	0.948	−0.172	0.074	1.0000	0.9302	0.673	0.000	0.000
0.4907	0.9653	0.918	−0.195	0.078					

not applied. To evaluate the viscometer constant, the length of the capillary of the viscometer (l) term is to be corrected as $l' = l + 0.5r$, r being the radius of the viscometer capillary. Since l is much larger (50 to 60 mm) as compared to r ($r = 0.5$ mm), $l = l'$ and hence end effects in viscometers are negligible. The dynamic viscosity (η) of the liquids was calculated[17] using

$$\eta/\eta_0 = (\rho t)/(\rho_0 t_0) \tag{1}$$

where ρ, ρ_0, t, t_0, and η, η_0 refer to density, flow time, and viscosity of liquids and water, respectively. The reproducibility in the measurement of viscosity was ±0.003 mPa·s.

Results and Discussion

Table 2 lists experimental values of densities (ρ) and viscosities (η) of binary mixtures of DMF with aniline and benzonitrile at (298.15, 303.15, 308.15, and 313.15) K. The ρ values have been used to calculate the excess molar volumes (V^E) using the following equation:

$$V^E/\text{cm}^3\cdot\text{mol}^{-1} = \frac{x_1M_1 + x_2M_2}{\rho_{\text{mix}}} - \frac{x_1M_1}{\rho_1} - \frac{x_2M_2}{\rho_2} \tag{2}$$

where x_1, x_2, M_1, M_2, ρ_1, and ρ_2 are mole fractions, molecular weights, and densities of pure components 1 and 2, respectively, and ρ_{mix} is the density of the binary mixture.

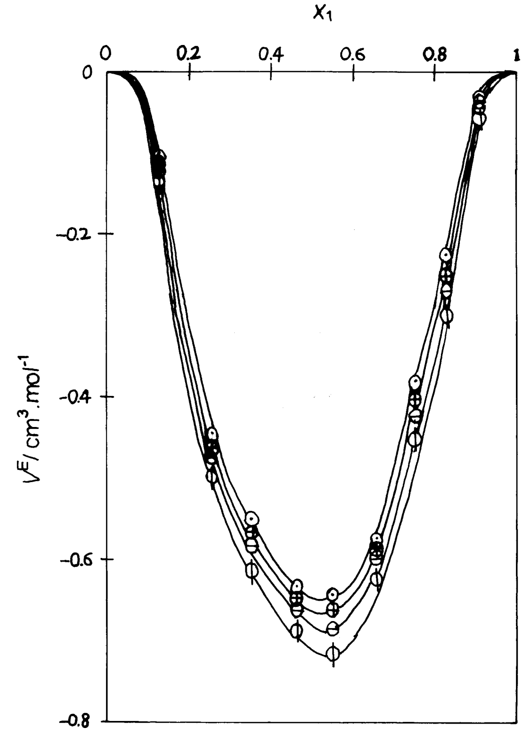

Figure 1. Excess molar volumes (eq 2), V^E, for (x_1)DMF + $(1 - x_1)$aniline at various temperatures: ⊙, 298.15; ⊕, 303.15; ⊖, 308.15; Φ, 313.15 K.

The viscosity deviations ($\Delta\eta$) were calculated by using the equation

$$(\Delta\eta) = \eta_{mix} - x_1\eta_1 - x_2\eta_2 \qquad (3)$$

where η_{mix} is the viscosity of the binary mixture and η_1, η_2 are the viscosities of components 1 and 2, respectively. The variations of V^E and $\Delta\eta$ with mole fraction of DMF (x_1) for the two systems studied at (298.15, 303.15, 308.15, and 313.15) K are represented in Figures 1−4, respectively. The V^E and $\Delta\eta$ values over the entire range of composition and at all temperatures are negative for binary mixtures of DMF with aniline. For binary mixtures of DMF with benzonitrile, the V^E and $\Delta\eta$ values are negative and positive, respectively, over the entire range of composition and at all temperatures.

Treszczanowicz et al.[19] suggested that V^E is the resultant contribution from several opposing effects. These may be divided arbitrarily into three types, namely chemical, physical, and structural. Physical contributions, that is, nonspecific interactions between the real species present in the mixture, contribute a positive term to V^E. The chemical or specific intermolecular interactions result in a volume decrease, and these include charge-transfer type forces and other complex-forming interactions. This effect contributes negative values to V^E. The structural contributions are mostly negative and arise from several effects, especially from interstitial accommodation and changes of free volume. In other words, structural contributions arising from geometrical fitting (interstitially accommodated) of one component into another due to the differences in the free volume and molar volume between components lead to a negative contribution to V^E.

The molar volumes of aniline at (298.15, 303.15, 308.15, and 313.15) K are (91.5552, 91.9439, 92.3359, and 92.7404 cm^3·mol^{-1}), respectively, and those of DMF are (77.3848, 77.7718, 78.1627, and 78.5745 cm^3·mol^{-1}) at (298.15, 303.15, 308.15, and 313.15) K, respectively. These molar volume values of DMF and aniline differ considerably; hence, nonassociated DMF molecules are interstitially accommodated into clusters of aniline, yielding a negative contribution to observed V^E values. This implies that the complex-forming interactions are almost absent in DMF + aniline systems and therefore observed $\Delta\eta$ values are negative. The V^E and $\Delta\eta$ values for DMF + benzonitrile

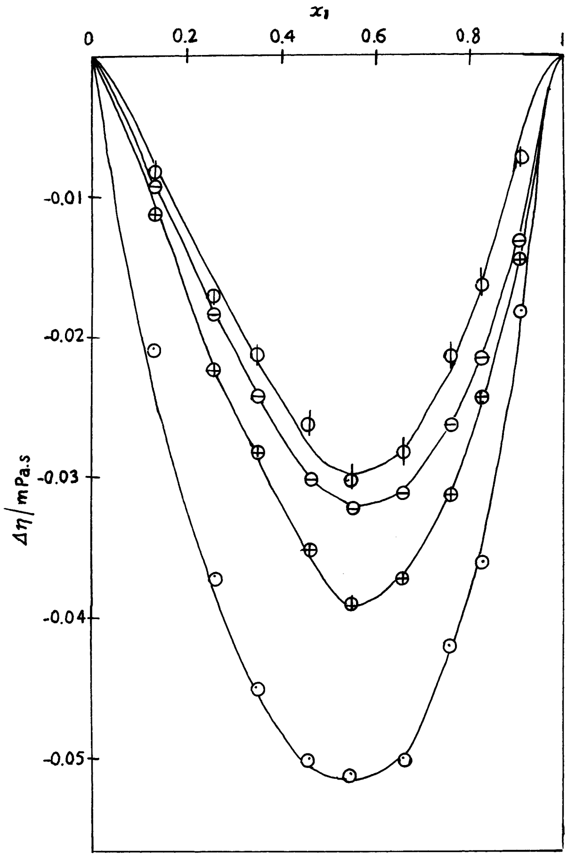

Figure 2. Deviations in viscosity (eq 3), $\Delta\eta$, for (x_1)DMF + $(1 - x_1)$aniline at various temperatures: ⊙, 298.15; ⊕, 303.15; ⊖, 308.15; Φ, 313.15 K.

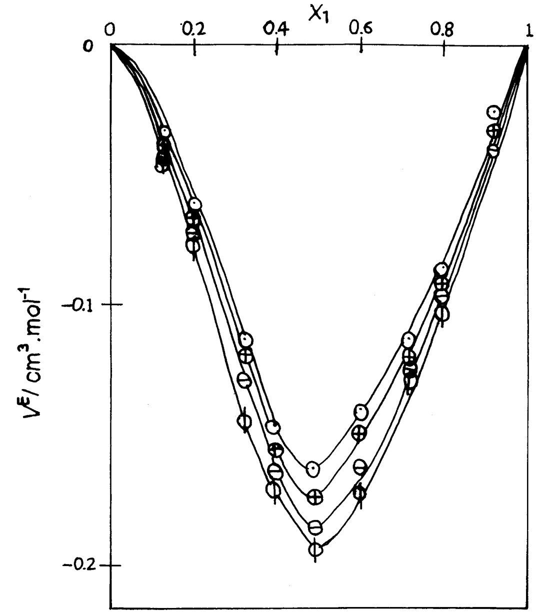

Figure 3. Excess molar volumes (eq 2), V^E, for (x_1)DMF + $(1 - x_1)$benzonitrile at various temperatures: ⊙, 298.15; ⊕, 303.15; ⊖, 308.15; Φ, 313.15 K.

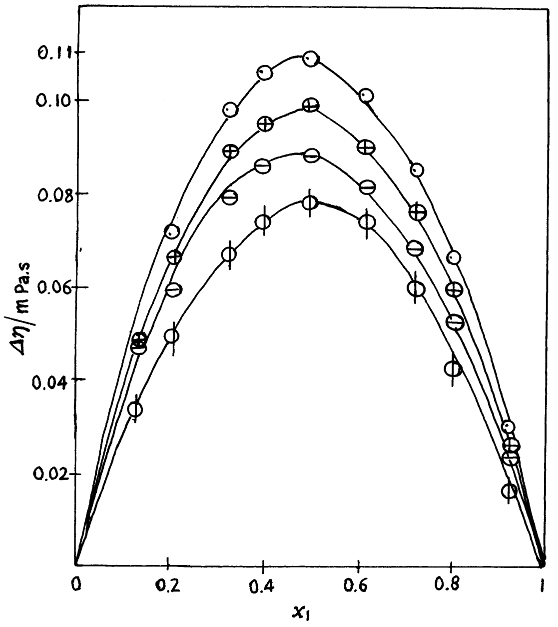

Figure 4. Deviations in viscosity (eq 3), $\Delta\eta$, for (x_1)DMF + (1 − x_1)benzonitrile at various temperatures: ⊙, 298.15; ⊕, 303.15; ⊖, 308.15; Φ, 313.15 K.

Table 3. Parameters and Standard Deviations, σ, of Eqs 4 and 5 for DMF (1) + Aniline (2) and DMF (1) + Benzonitrile (2)

	T/K	a_0	a_1	a_2	a_3	σ
			DMF−Aniline			
V^E/cm^3·mol^{-1}	298.15	−2.7726	−0.2545	3.1238	0.5633	0.035
	303.15	−2.8408	−0.2929	3.1055	0.5919	0.037
	308.15	−2.8895	−0.5336	3.0853	0.9134	0.031
	313.15	−3.0349	−0.3866	3.0684	0.5758	0.043
$\Delta\eta$/mPa·s	298.15	−0.2042	−0.0530	−0.0212	0.0448	0.002
	303.15	−0.1479	−0.1262	−0.0237	0.0896	0.004
	308.15	−0.1219	−0.0397	0.0116	−0.0221	0.001
	313.15	−0.1111	−0.0497	0.0446	0.0567	0.001
			DMF−Benzonitrile			
V^E/cm^3·mol^{-1}	298.15	−0.6226	−0.0057	0.4179	−0.2705	0.006
	303.15	−0.6648	−0.0375	0.4797	−0.1651	0.005
	308.15	−0.7001	−0.0052	0.4658	−0.3057	0.006
	313.15	−0.7517	−0.0029	0.5392	−0.2846	0.005
$\Delta\eta$/mPa·s	298.15	0.4347	−0.0488	0.0095	0.0192	0.000
	303.15	0.3916	−0.0455	−0.0049	0.0145	0.001
	308.15	0.3507	−0.0396	−0.0022	−0.0006	0.001
	313.15	0.3120	−0.0181	−0.0871	−0.0245	0.003

systems are negative and positive, respectively, over the entire range of composition and at all temperatures. The negative V^E and positive $\Delta\eta$ values for DMF + benzonitrile systems indicate strong specific interactions through dipolar association between DMF and benzonitrile molecules.

From close observation of Table 2, it is seen that the negative V^E values are much higher than those of $\Delta\eta$ for binary systems of DMF + aniline. This clearly supports mare addition of DMF molecules into aggregates of aniline. The negative V^E values are comparable with positive $\Delta\eta$ values for DMF + benzonitrile systems, suggesting strong specific intermolecular interactions.

The results of V^E and $\Delta\eta$ are fitted in the Redlich−Kister eqation:[20]

$$Y = x_1x_2\sum a_i(x_1 - x_2)^i \tag{4}$$

where Y refers to V^E/cm^3·mol^{-1} or $\Delta\eta$/mPa·s and x_1 and x_2 are the mole fractions of components 1 and 2, respectively. The coefficients a_i were obtained by fitting eq 4 to experimental results using a least-squares regression method. In each case, the optimum number of coefficients is ascertained from the examination of the variation in standard deviation (σ). The third-order form of eq 4 gives the minimum standard deviation in V^E and $\Delta\eta$.

The σ was calculated using

$$\sigma(Y) = (\sum(Y_{\text{exp}} - Y_{\text{cal}})^2/(n - m))^{1/2} \tag{5}$$

where n is the number of data points and m is the number of coefficients. The calculated values of a_i along with standard deviations (σ) are given in Table 3.

In conclusion, it is observed that DMF molecules are interstitially accommodated into clusters of aniline, resulting in negative excess molar volumes and negative deviations in viscosity for DMF + aniline systems. However, negative excess molar volumes and positive deviations in viscosity for DMF + benzonitrile systems are attributed to strong specific interactions through dipolar association between DMF and benzonitrile molecules.

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