Densities and Viscosities of Binary Mixtures of *N*,*N*-Dimethylformamide with Benzyl Alcohol and Acetophenone 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 benzyl alcohol and acetophenone have been measured at (298.15, 303.15, 308.15, and 313.15) K. Using the experimental data, excess molar volumes (V^E) and deviations in viscosity ($\Delta\eta$) have been calculated. The excess molar volumes and deviations in viscosity have been fitted to the Redlich−Kister polynomial equation. McAllister's three-body interaction model has been used to correlate the kinematic viscosities of binary liquid mixtures with mole fractions.

Introduction

The knowledge of densities and viscosities of pure liquids and liquid mixtures is needed for optimal design of many types of equipment in chemical technology.

DMF is a versatile solvent, used in the synthesis of pharmaceuticals, in agricultural chemistry, and as a solvent for polymers. A fundamental understanding of the mixture behavior of DMF with polar, nonpolar, associated, and nonassociated solvents is therefore important from the technical and engineering point of view. The density and viscosity data of binary mixtures of DMF with polar, nonpolar, associated, and nonassociated solvents have been reported previously.[1−4] Benzyl alcohol exists in associated form whereas DMF and acetophenone have nonassociated structures in the liquid state. To the best of our knowledge, there is single report[5] on density and viscosity data of binary mixtures of DMF with benzyl alcohol (at 298.15 K only). However, there are no reports on density and viscosity data of binary mixtures of DMF with acetophenone. Therefore, in the present paper we report densities and viscosities of binary mixtures of DMF with benzyl alcohol and acetophenone over the entire range of composition and at (298.15, 303.15, 308.15, and 313.15) K.

Experimental Section

Acetophenone (E. Merck, Extra pure, purity > 99%) was further purified[6] by fractional distillation and fusion until constancy of the boiling point and density for successive fractions. DMF (S. D. Fine Chemicals, analytical reagent, purity > 99.5%) and benzyl alcohol (S. D. Fine Chemicals, analytical reagent, purity > 99.5%) were directly used. 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 (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 $\pm 1 \times 10^{-4}$ g. The estimated error in mole fraction was $< 1 \times 10^{-4}$. 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[13]	0.803	0.802[12]
	303.15	0.9398	0.9397[14]	0.756	0.752[16]
	308.15	0.9351	0.9356[14]	0.710	0.707[16]
	313.15	0.9302	0.9298[15]	0.673	0.664[16]
benzyl alcohol	298.15	1.0413	1.0415[6]	5.160	
	303.15	1.0375	1.0376[6]	4.670	4.65[6]
	308.15	1.0337	1.0339[6]	4.004	
	313.15	1.0294	1.0306[6]	3.520	3.530[17]
acetophenone	298.15	1.0241	1.0238[6]	1.680	1.617[18]
	303.15	1.0199	1.0194[6]	1.540	1.511[6]
	308.15	1.0157	1.0150[6]	1.410	
	313.15	1.0112	1.0106[6]	1.310	

The densities of degassed pure liquids and binary mixtures were measured using a 15 cm^3 double-arm pycnometer, as reported earlier,[7] in a transparent glass-walled water bath having a thermal stability of ±0.01 K. The pycnometer was calibrated using conductivity water[8] having a conductivity of $< 1 \times 10^{-6}\ \Omega^{-1}\ \text{cm}^{-1}$. Uncertainties in density measurement were within $\pm 1 \times 10^{-4}$ g·cm^{-3}.

Dynamic viscosities were measured using an Ubbelohde suspended-level viscometer,[7] calibrated with conductivity water.[8] 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 not applied. To evaluate the viscometer constant, the length of the capillary of the viscometer (*l*) term is to be corrected as $l' = l + 0.5r$ with 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[7] using

$$\eta/\eta_0 = (\rho t)/(\rho_0 t_0) \quad (1)$$

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Table 2. Density, ρ, Viscosity, η, Excess Molar Volume, V^E, and Deviations in Viscosity, $\Delta\eta$, for DMF (1) + Benzyl Alcohol (2) and DMF (1) + Acetophenone (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) + Benzyl Alcohol (2)					
				298.15 K					
0.0000	1.0413	5.160	0.000	0.000	0.5865	0.9961	1.850	−0.403	−0.755
0.1088	1.0361	4.473	−0.281	−0.213	0.7170	0.9807	1.440	−0.232	−0.596
0.1712	1.0325	4.011	−0.396	−0.403	0.7962	0.9703	1.230	−0.090	−0.461
0.3055	1.0227	3.140	−0.496	−0.689	0.8972	0.9576	1.001	−0.017	−0.250
0.4003	1.0148	2.590	−0.520	−0.826	1.0000	0.9445	0.803	0.000	0.000
0.5393	1.0013	2.020	−0.456	−0.790					
				303.15 K					
0.0000	1.0375	4.670	0.000	0.000	0.5865	0.9920	1.710	−0.423	−0.664
0.1088	1.0323	4.060	−0.290	−0.184	0.7170	0.9764	1.335	−0.244	−0.529
0.1712	1.0287	3.648	−0.412	−0.352	0.7962	0.9659	1.146	−0.099	−0.408
0.3055	1.0188	2.870	−0.514	−0.604	0.8972	0.9531	0.938	−0.025	−0.220
0.4003	1.0108	2.378	−0.536	−0.725	1.0000	0.9398	0.756	0.000	0.000
0.5393	0.9972	1.860	−0.474	−0.699					
				308.15 K					
0.0000	1.0337	4.004	0.000	0.000	0.5865	0.9879	1.541	−0.445	−0.531
0.1088	1.0285	3.510	−0.310	−0.136	0.7170	0.9721	1.218	−0.257	−0.424
0.1712	1.0249	3.170	−0.428	−0.270	0.7962	0.9615	1.054	−0.108	−0.327
0.3055	1.0149	2.517	−0.532	−0.481	0.8972	0.9487	0.872	−0.042	−0.177
0.4003	1.0068	2.100	−0.552	−0.585	1.0000	0.9351	0.710	0.000	0.000
0.5393	0.9931	1.670	−0.492	−0.558					
				313.15 K					
0.0000	1.0294	3.520	0.000	0.000	0.5865	0.9834	1.411	−0.461	−0.429
0.1088	1.0243	3.106	−0.311	−0.104	0.7170	0.9676	1.127	−0.279	−0.352
0.1712	1.0206	2.817	−0.441	−0.216	0.7962	0.9569	0.982	−0.124	−0.271
0.3055	1.0106	2.260	−0.555	−0.390	0.8972	0.9440	0.819	−0.054	−0.147
0.4003	1.0024	1.900	−0.569	−0.480	1.0000	0.9302	0.673	0.000	0.000
0.5393	0.9887	1.520	−0.515	−0.465					
				DMF (1) + Acetophenone (2)					
				298.15 K					
0.0000	1.0241	1.680	0.000	0.000	0.6256	0.9858	1.156	−0.322	0.025
0.1123	1.0180	1.584	−0.004	0.002	0.7165	0.9772	1.072	−0.259	0.020
0.1641	1.0156	1.544	−0.068	0.008	0.7943	0.9690	0.999	−0.182	0.016
0.3080	1.0088	1.430	−0.288	0.020	0.9378	0.9525	0.863	−0.061	0.005
0.4328	1.0010	1.328	−0.355	0.028	1.0000	0.9445	0.803	0.000	0.000
0.5286	0.9942	1.246	−0.382	0.030					
				303.15 K					
0.0000	1.0199	1.540	0.000	0.000	0.6256	0.9814	1.069	−0.332	0.019
0.1123	1.0139	1.453	−0.019	0.001	0.7165	0.9728	0.995	−0.273	0.017
0.1641	1.0114	1.418	−0.076	0.007	0.7943	0.9645	0.930	−0.190	0.013
0.3080	1.0046	1.314	−0.304	0.015	0.9378	0.9479	0.809	−0.067	0.004
0.4328	0.9968	1.221	−0.376	0.020	1.0000	0.9398	0.756	0.000	0.000
0.5286	0.9899	1.148	−0.398	0.022					
				308.15 K					
0.0000	1.0157	1.410	0.000	0.000	0.6256	0.9771	0.984	−0.352	0.012
0.1123	1.0097	1.330	−0.025	0.001	0.7165	0.9684	0.918	−0.288	0.010
0.1641	1.0072	1.299	−0.084	0.004	0.7943	0.9600	0.861	−0.199	0.007
0.3080	1.0004	1.205	−0.319	0.011	0.9378	0.9433	0.755	−0.072	0.001
0.4328	0.9925	1.122	−0.388	0.015	1.0000	0.9351	0.710	0.000	0.000
0.5286	0.9856	1.056	−0.414	0.016					
				313.15 K					
0.0000	1.0112	1.310	0.000	0.000	0.6256	0.9725	0.922	−0.368	0.011
0.1123	1.0052	1.238	−0.029	0.000	0.7165	0.9638	0.862	−0.306	0.008
0.1641	1.0027	1.206	−0.090	0.001	0.7943	0.9553	0.810	−0.210	0.006
0.3080	0.9959	1.123	−0.333	0.009	0.9378	0.9385	0.714	−0.079	0.001
0.4328	0.9880	1.047	−0.407	0.013	1.0000	0.9302	0.673	0.000	0.000
0.5286	0.9811	0.987	−0.436	0.014					

where ρ, ρ_0, t, t_0, and η, η_0 refer to the 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 benzyl alcohol and acetophenone 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:

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

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

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

$$\Delta\eta/\text{mPa·s} = \eta_{\text{mix}} - x_1\eta_1 - x_2\eta_2 \quad (3)$$

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 fractions of DMF (x_1) for DMF + benzyl alcohol systems at (298.15, 303.15, 308.15, and 313.15) K are represented in Figures 1 and 2, respectively. The variation of V^E with mole fractions of DMF (x_1) for DMF + acetophenone systems at (298.15, 303.15, 308.15, and 313.15) K is represented in Figure 3. Since $\Delta\eta$ values for DMF + acetophenone systems are very small, they are not represented graphically. 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 benzyl alcohol. For binary mixtures of DMF with acetophenone, 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.[9] 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 the other 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 benzyl alcohol at (298.15, 303.15, 308.15, and 313.15) K are (118.266, 118.699, 119.135, and 119.633 $cm^3·mol^{-1}$), respectively, and those of DMF are (77.385, 77.772, 78.163, and 78.575 $cm^3·mol^{-1}$) at (298.15, 303.15, 308.15, and 313.15) K, respectively. These molar volume values of DMF and benzyl alcohol differ considerably; hence, nonassociated DMF molecules are interstitially accommodated into aggregates of benzyl alcohol, yielding a negative contribution to observed V^E values. This implies that the complex forming interactions are almost absent in DMF + benzyl alcohol systems, and therefore observed $\Delta\eta$ values are negative. Alternatively, it can be said that the two components, DMF + benzy alcohol, strongly interact to form heteroaggregated adducts and complex species, with the mixing process accompanied by prevailing "breaking structure effects" in bulking pure benzyl alcohol, while the complexes formed are quite unable to give an effective network sustaining the liquid structure. The V^E values for DMF + acetophenone systems are negative, over the entire range of composition and at all temperatures. The negative V^E values for DMF + acetophenone systems indicate strong specific interactions through dipolar association between DMF and acetophenone molecules. The deviations in viscosity ($\Delta\eta$) values for DMF + acetophenone systems are too small to make any conclusions about the nature of interactions between these two components; hence, no definite comments on $\Delta\eta$ could be made.

The results of V^E and $\Delta\eta$ are fitted in the Redlich−Kister equation:[10]

$$Y = x_1x_2\sum a_i(x_1 - x_2)^i \quad (4)$$

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).

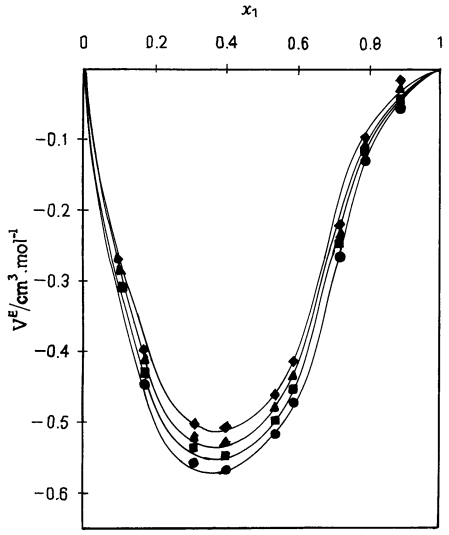

Figure 1. Excess molar volumes (eq 4), V^E, for (x_1)DMF + (1 − x_1)benzyl alcohol at various temperatures: ◆, 298.15; ▲, 303.15; ■, 308.15; ●, 313.15 K.

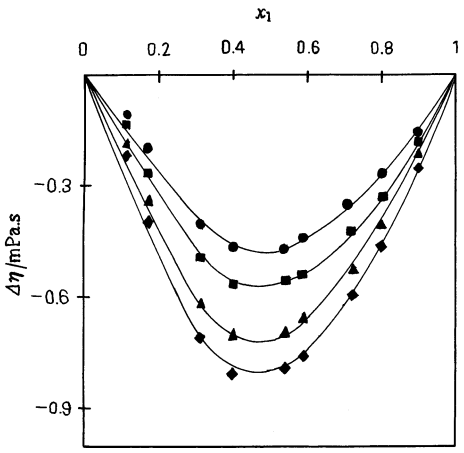

Figure 2. Deviations in viscosity (eq 4), $\Delta\eta$, for (x_1)DMF + (1 − x_1)benzyl alcohol at various temperatures: ◆, 298.15; ▲, 303.15; ■, 308.15; ●, 313.15 K.

The σ was calculated using

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

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

The kinematic viscosities, ν, of the binary liquid mixtures are calculated from their dynamic viscosities and densities. McAllister's three-body interaction model[11] has been used to correlate the kinematic viscosities of binary liquid mixtures.

$$\ln \nu = x_1^3 \ln \nu_1 + x_2^3 \ln \nu_2 + 3x_1^2x_2 \ln \nu_{12} + 3x_2^2x_1 \ln \nu_{21} - \ln(x_1 + x_2M_2/M_1) + 3x_1^2x_2 \ln(^2/_3 + M_2/3M_1) + 3x_2^2x_1 \ln(^1/_3 + 2M_2/3M_1) + x_2^3 \ln(M_2/M_1) \quad (6)$$

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

property	T/K	a_0	a_1	a_2	a_3	a_4	a_5	σ
				DMF (1) + Benzyl Alcohol (2)				
V^E/cm^3·mol^{-1}	298.15	−1.934	1.071	1.095	3.190	−0.741	−3.441	0.007
	303.15	−2.006	1.032	1.067	3.675	−0.735	−4.110	0.006
	308.15	−2.092	1.017	1.310	3.894	−1.402	−4.375	0.006
	313.15	−2.164	0.987	1.069	4.230	−0.993	−5.044	0.007
$\Delta\eta$/mPa·s	298.15	−3.257	0.737	0.538	−1.729	1.224		0.009
	303.15	−2.867	0.611	0.443	−1.946	1.174		0.008
	308.15	−2.301	0.501	0.433	−1.356	0.977		0.008
	313.15	−1.889	0.392	0.349	−1.182	0.887		0.008
				DMF (1) + Acetophenone (2)				
V^E/cm^3·mol^{-1}	298.15	−1.526	0.224	1.631	−1.444			0.009
	303.15	−1.585	0.254	1.562	−1.459			0.009
	308.15	−1.649	0.247	1.556	−1.468			0.010
	313.15	−1.726	0.255	1.571	−1.552			0.010
				DMF (1) + Acetophenone (2)				
$\Delta\eta$/mPa·s	298.15	0.116	−0.004	−0.099	0.075			0.001
	303.15	0.087	0.006	−0.069	0.046			0.001
	308.15	0.062	−0.005	−0.072	0.023			0.001
	313.15	0.054	−0.001	−0.073	0.023			0.000

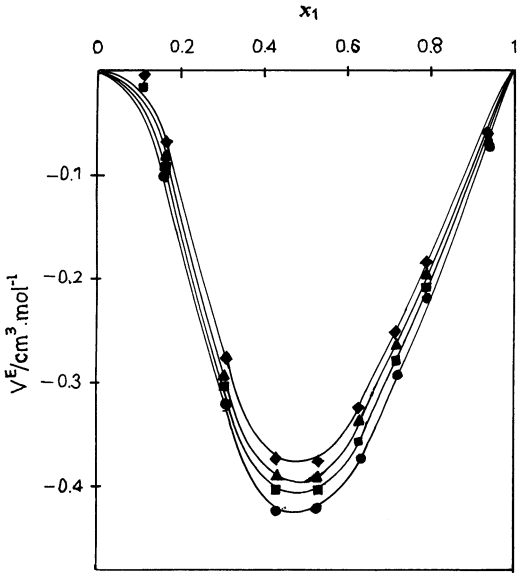

Figure 3. Excess molar volumes (eq 4), V^E, for (x_1)DMF + (1 − x_1)acetophenone at various temperatures: ◆, 298.15; ▲, 303.15; ■, 308.15; ●, 313.15 K.

Table 4. Parameters of McAllister's Model and Standard Percentage Deviation for Kinematic Viscosities at Various Temperatures

system	T/K	ν_{12}	ν_{21}	σ%
DMF (1) + benzyl alcohol (2)	298.15	3.372	1.508	2.27
	303.15	3.088	1.403	2.20
	308.15	2.724	1.275	2.31
	313.15	2.452	1.181	2.24
DMF (1) + acetophenone (2)	298.15	1.416	1.238	0.20
	303.15	1.306	1.143	0.14
	308.15	1.201	1.050	0.24
	313.15	1.125	0.983	0.17

where ν_{12} and ν_{21} are model parameters.

The percentage standard deviation was calculated by using the following equation.

$$\sigma(\%) = (\sum(100(\nu_{expt} - \nu_{cal})/\nu_{expt})^2/(n - m))^{1/2} \quad (7)$$

where n represents the number of experimental points and m the number of coefficients. Table 4 includes the different parameters and the percentage standard deviation. From Table 4 it is clear that McAllister's three-body interaction model is suitable to correlate the kinematic viscosities of the binary mixtures studied.

In conclusion, it is observed that DMF molecules are interstitially accommodated into aggregates of benzyl alcohol or form heteroaggregated adducts and complex species, resulting in negative excess molar volumes and negative deviations in viscosity for DMF + benzyl alcohol systems. However, negative excess molar volumes for DMF + acetophenone systems are attributed to strong specific interactions through dipolar association between DMF and acetophenone molecules.

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