Optical, Volumetric And Viscometric Studies Of Binary Liquid Mixtures

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Abstract: Refractive indices, densities and viscosities of binary mixtures of dimethyl- sulfoxide (DMSO) with formamide and N,N-dimethylformamide have been measured over the entire composition range at 293, 303 and 313K. Refractive index, viscosity and density data have been used to evaluate the molar refraction deviation (ΔR_m), excess molar volume (V_m^E) and deviation in viscosity ($\Delta \eta$). The results were fitted to the Redlich –Kister polynomial equation of third degree and the adjustable parameters were obtained using least square method. Precise estimation and reliability of various mixing rules for refractive indices and densities have been scrutinized over the entire composition range for both the mixtures. Further, the experimental results were used to study the nature and extent of interaction present in the mixture.

Keywords: Refractive index, viscosity, excess molar volume, molecular interaction

I. INTRODUCTION

Excess and bulk properties of mixture provide insight into the molecular interactions between various components and can be used for the development of molecular models describing the thermodynamic behavior of mixtures. Experimental determination of all these properties is time consuming and expensive especially if data have to be known at various state of condition and compositions. Therefore, the properties of multi-component mixtures are often estimated from the corresponding data of the constituting binary mixtures but the reliability of such an estimation is always questionable and has to be tested ¹

Excess properties such as excess molar volume and deviation in refractive index and its variation with temperature and composition of binary mixtures are useful for design engineering process, in chemical and biological industries ²⁻³⁾ and to test theories of solution ⁴⁾. Measurements of refractive index, viscosity with molar volume of liquid and liquid mixtures have gained much importance during recent past ⁵⁻⁸⁾, since these data provide reliable information regarding the specific interactions between components.

Thermodynamic and transport property data are of great interest in process design and separation. Viscosity and density data are required in many chemical engineering calculations involving fluid flow, heat and mass transfer. Moreover, the knowledge of the dependence of refractive index and density on composition of mixture is of considerable interest in understanding the intermolecular interaction. The present work is a continuation of systematic experimental studies on thermodynamic and transport properties of binary mixtures of dimethyl sulfoxide (DMSO) with formamide (FA) and N, N- dimethylformamide (DMF), as these solvents and their mixtures have important applications in chemical industry and modern technology⁹.

In order to investigate the molecular interactions in the DMSO Nmixture of with formamide and N. dimethylformamide through optical, volumetric and viscometric properties, we report here the refractive indices (n_m) , densities (ρ_m) and viscosity (η_m) of these mixture over the entire composition range at temperatures 293, 303 and 313K. These experimental data have been used to evaluate

molar refraction deviation (ΔR_m) , excess volume (V_m^E) and deviation in viscosity ($\Delta \eta$). The results have been used to discuss the nature of interaction between unlike molecules in terms of dipole-dipole interaction and dispersive forces. Since refractive index is closely related with orientation, ordering and strength of the constituent atomic oscillators of given medium, it is expected that variation of refractive index of mixture with temperature and composition can give valuable insight into the molecular rearrangement due to mixing. Therefore, six mixing rule for refractive index proposed by Lorentz-Lorenz (L-L), Gladestone Dale (G-D), Wiener (W), Heller (H), Newton (Nn) and Eykman (Eyk)¹⁰⁾ and Hankinson - Brobst - Thomson (HBT) model ¹¹⁾ for estimation of density have also been applied to these mixtures. The relative merits of these relations were discussed and results have been presented in terms of average percentage deviation (APD) and root mean square deviation.

II. EXPERIMENTAL DETAILS

Refractive indices (n_m) were measured with Abbe's refractometer provided by Optics Technologies, Delhi. Calibration of refractometer was performed periodically under atmospheric pressure, in accordance with specifications, using triply distilled water and benzene at 293K. The uncertainty of refractive index measurement was estimated to be less than \pm 0.001 units. The sample mixtures were directly injected into the prism assembly of the instrument by means of an air tight hypodermic syringe. An average of four to five measurements was taken for each sample mixtures. Densities and viscosities of liquids and liquid mixtures have been measured using a single capillary pyknometer and Ostwald's viscometer as discussed earlier⁵). All measurements have been carried out at three temperatures viz 293, 303 and 313K using a microprocessor based temperature controller (Julabo F 25 HD) $(accuracy \pm 0.01^{\circ}C).$

Mixtures were prepared by weighing the liquids in specially designed ground glass stoppered weighing bottles, taking extreme precautions, in order to prevent evaporation losses during the experiment. A sartorius (BP 121S) single pan balance having a stated precision of 0.1 mg was used through out. The maximum possible error in the mole fraction is estimated to be 0.0001.

The chemicals used were obtained from Ranbaxy Fine Chemical Limited. All the chemical used were purified by standard procedure, discussed by Perrin and Amarego¹²⁾.

III. THEORY

The molar refraction deviation ΔR_m was determined using the following equation ⁵⁾:

$$\Delta R_m = R_m^{\exp t} - R_m^{idl} \tag{1},$$

where

$$\Delta R_m^{\exp t} = \left(\frac{n_m^2 - 1}{n_m^2 + 2}\right) \frac{\left(x_1 M_1 + x_2 M_2\right)}{\rho_m}$$
(1.1),

and

$$R_m^{idl} = \left[\left(\frac{n_1^2 - 1}{n_1^2 + 2} \right) \frac{M_1}{\rho_m} \phi_1 + \left(\frac{n_2^2 - 1}{n_2^2 + 2} \right) \frac{M_2}{\rho_m} \phi_2 \right]$$
(1.2),

The experimentally measured densities were used to predict the excess molar volume using equation $^{13)}$

$$V_m^E = \frac{M_1 x_1 + M_2 x_2}{\rho} - \frac{M_1 x_1}{\rho_1} - \frac{M_2 x_2}{\rho_2}$$
(2),

and deviation in viscosity ($\Delta \eta$) were calculated from the experimental result by following equations:

$$\Delta \eta = \eta_m - (x_1 \eta_1 + x_2 \eta_2) \tag{3},$$

where symbols have their usual meaning. A Redlich-Kister equation was used to correlate the derived properties of the binary mixtures by least square method¹⁴.

$$Y^{E} = x_{1}x_{2}\sum_{i=1}^{3}a_{i}(x_{2} - x_{1})^{i-1}$$
(4),

where Y^{E} refers to ΔR_{m} , V_{m}^{E} or $\Delta \eta$ and a_{i} is the fitting parameter. The root mean square deviation is evaluated using the relation:

$$\sigma\left(Y^{E}\right) = \left[\frac{\sum (Y^{E}_{exp} - Y^{E}_{cal})^{2}}{n - m}\right]^{\frac{1}{2}}$$
(4.1),

where n is number of data point and m is the number of coefficients.

Six mixing rules for refractive indices, as described earlier ¹⁰⁾ have been used to scrutinize the relative merits of these mixing rules. Hankinson- Brobst – Thomson (HBT) model for liquid density ¹¹⁾ could be described as:

$$\rho = \frac{M}{V^* V_R^0 \left\{ 1 - \omega_{SRK} V_R^{(\delta)} \right\}}$$
(5),

where

$$V_{R}^{0} = 1 + a(1 - T_{r})^{\frac{1}{3}} + b(1 - T_{r})^{\frac{2}{3}} + c(1 - T_{r}) + d(1 - T_{r})^{\frac{4}{3}}$$
(5.1),
0.25 < T_{r} < 0.95

and
$$V_R^{(\delta)} = \frac{(e + fT_r + gT_r^2 + hT_r^3)}{(T_r - 1.00001)}$$
 (5.2),

$$0.25 < T_r < 1.0$$

and coefficients:	
<i>a</i> = -1.52816	e = -0.296123
<i>b</i> = 1.43907	f = 0.386914
c = 0.81446	g = -0.0427258
d = 0.190454	h = -0.0480645

IV. RESULTS

Refractive indices (n_m) , densities (ρ_m) , viscosity (η_m) , deviation in molar refraction (ΔRm) , excess molar volume

 (V_m^E) and deviation in viscosity $(\Delta \eta)$ are reported in Tables I and II for DMSO + formamide and DMSO + N, Ndimethylformamide mixtures respectively at 293, 303 and 313K. The fitting parameters (a_i) along with root mean square deviation $\sigma(Y^E)$ are gathered in Table III. Theoretically estimated values of refractive indices in terms of average percentage deviation (APD) are reported in Table IV.

In order to predict density using HBT model, critical values required for each compound along with root mean square deviation are given in Table V. The temperature and composition dependence of molar refraction deviation (ΔR_m) , excess molar volume (V_m^E) and deviation in viscosity $(\Delta \eta)$ are shown in Figs. 1-3 respectively for both the mixtures.

x_1	$ ho_{\scriptscriptstyle m}$	n_m	ΔR_m	$V_m^E(\times 10^{-3})$	$\Delta \eta$
	(kg/m^3)			(m ³ /mol)	(Pa-s)
		T=293K		· · · · · · · · · · · · · · · · · · ·	
0.0000	1137.0	1.4440	0.0000	0.0000	0.0000
0.0604	1133.9	1.4490	-0.3748	-0.0150	0.2241
0.1261	1130.5	1.4530	-0.7047	-0.0193	0.4351
0.1982	1127.2	1.4560	-0.9910	-0.0257	0.6275
0.2776	1123.8	1.4590	-1.2005	-0.0283	0.7919
0.3654	1120.4	1.4630	-1.2972	-0.0303	0.9157
0.4643	1117.0	1.4660	-1.3190	-0.0323	0.9822
0.5739	1113.5	1.4690	-1.2212	-0.0320	0.9657
0.6979	1110.0	1.4730	-0.9519	-0.0287	0.8327
0.8391	1106.6	1.4760	-0.5421	-0.0204	0.5332
1.0000	1103.0	1.4770	0.0000	0.0000	0.0000
		T=303K			
0.0000	1129.0	1.4400	0.0000	0.0000	0.0000
0.0604	1125.6	1.4430	-0.4093	-0.0135	0.1784
0.1261	1122.1	1.4460	-0.7556	-0.0179	0.3464
0.1982	1118.5	1.4500	-1.0362	-0.0239	0.4995
0.2776	1114.9	1.4530	-1.2398	-0.0262	0.6303
0.3654	1111.3	1.4560	-1.3566	-0.0279	0.7289
0.4643	1107.7	1.4590	-1.3727	-0.0297	0.7818
0.5739	1104.1	1.4620	-1.2701	-0.0292	0.7682
0.6979	1100.5	1.4650	-1.0275	-0.0271	0.6628
0.8391	1096.8	1.4680	-0.6147	-0.0185	0.4244
1.0000	1093.0	1.4710	0.0000	0.0000	0.0000
		T=313K			
0.0000	1121.0	1.4370	0.0000	0.0000	0.0000
0.0604	1117.3	1.4410	-0.3975	-0.0111	0.1535
0.1261	1113.3	1.4410	-0.8205	-0.0150	0.2980
0.1982	1109.5	1.4450	-1.0871	-0.0209	0.4298
0.2776	1105.6	1.4480	-1.2983	-0.0230	0.5423
0.3654	1101.7	1.4520	-1.3945	-0.0246	0.6272
0.4643	1097.8	1.4550	-1.4147	-0.0262	0.6727
0.5739	1093.9	1.4580	-1.3136	-0.0260	0.6614
0.6979	1090.0	1.4610	-1.0707	-0.0236	0.5703
0.8391	1086.0	1.4640	-0.6552	-0.0139	0.3652
1.0000	1082.0	1.4680	0.0000	0.0000	0.0000

Table	I: Experimen	ntally mea	sured val	ues of densities	s ($ ho_m$),	
refra	ctive index ()	n _m), mole	ar refracti	ion deviation (ΔR_m),	
excess	excess molar volume (V_m^{E}) and deviation in viscosity ($\Delta \eta$)					
	with mole fraction x_1 for DMSO + FA mixture					
x_1	$ ho_m$	n_	ΔR_m	$V_m^E(\times 10^{-3})$	$\Delta\eta$	
	(kg/m^3)	m		(m ³ /mol)	(Pa-s)	
		T=293K				

0.0000

0.0006

0.0000

-0.0124

0.0000

-0.0362

1.4290

1.4330

0.1895	979.9	1.4380	0.0018	-0.0205	-0.0653		
0.2862	993.9	1.4420	0.0028	-0.0264	-0.0869		
0.3845	1008.3	1.4470	0.0034	-0.0301	-0.1007		
0.4832	1022.9	1.4510	0.0036	-0.0316	-0.1062		
0.5838	1038.1	1.4560	0.0035	-0.0307	-0.1034		
0.6862	1053.7	1.4610	0.0030	-0.0273	-0.0916		
0.7889	1069.6	1.4660	0.0020	-0.0215	-0.0709		
0.8943	1086.2	1.4720	0.0006	-0.0130	-0.0402		
1.0000	1103.0	1.4770	0.0000	0.0000	0.0000		
		T=303K					
0.0000	943.0	1.4240	0.0000	0.0000	0.0000		
0.0940	956.2	1.4280	0.0013	-0.0099	-0.0289		
0.1895	969.8	1.4320	0.0024	-0.0178	-0.0520		
0.2862	983.8	1.4370	0.0033	-0.0236	-0.0691		
0.3845	998.2	1.4410	0.0038	-0.0273	-0.0800		
0.4832	1012.8	1.4460	0.0040	-0.0288	-0.0843		
0.5838	1028.0	1.4510	0.0040	-0.0280	-0.0819		
0.6862	1043.6	1.4550	0.0035	-0.0243	-0.0725		
0.7889	1059.5	1.4600	0.0027	-0.0191	-0.0560		
0.8943	1076.1	1.4660	0.0016	-0.0108	-0.0318		
1.0000	1093.0	1.4710	0.0000	0.0000	0.0000		
		T=313K					
0.0000	934.0	1.4190	0.0000	0.0000	0.0000		
0.0940	947.0	1.4230	0.0032	-0.0027	-0.0103		
0.1895	960.6	1.4280	0.0056	-0.0116	-0.0185		
0.2862	974.4	1.4320	0.0072	-0.0183	-0.0246		
0.3845	988.7	1.4370	0.0082	-0.0225	-0.0284		
0.4832	1003.3	1.4420	0.0085	-0.0243	-0.0299		
0.5838	1018.3	1.4470	0.0082	-0.0236	-0.0290		
0.6862	1033.9	1.4520	0.0075	-0.0202	-0.0256		
0.7889	1049.7	1.4570	0.0059	-0.0141	-0.0198		
0.8943	1066.1	1.4620	0.0035	-0.0051	-0.0112		
1.0000	1083.0	1.4680	0.000	0.0000	0.0000		
Table II:	Experime	ntally mea	sured val	ues of densiti	es ($ ho_m$),		
refractive index ($n_{_m}$), molar refraction deviation ($\Delta \! R_{_m}$),							

excess molar volume (V_m^E) and deviation in viscosity ($\Delta \eta$) with mole fraction x_1 for DMSO + DMF mixture

jraciii	$m x_1 j 0 r$	DMSU	T	DW
	a_1	<i>a</i> 2		

 a_3

 $\sigma(Y^E)$

Parameters

		DMSO + FA		
		T=293K		
$V^{E}(\times 10^{-3}m^{3}/mol)$	-	-0.0510	-	0.0006
V_m (×10 m / mor)	0.1408		0.0975	
$\Lambda n(P_{a,s})$	3.9513	0.0000	-	0.0010
$\Delta \eta$ (Fu-s)			0.0047	
٨D	-	-1.6728	0.0835	0.0047
$\Delta \mathbf{n}_m$	5.1883			
		T=303K		
$V^{E}(\times 10^{-3} m^{3} / mol)$	-	-0.0018	-	0.0005
V_m (×10 m / mot)	0.1244		0.0300	
$\Lambda n(P_{\alpha})$	3.1440	-0.0041	-	0.0004
$\Delta \eta$ (Fu-s)			0.0074	
٨D	-	-1.5681	-	0.0039
$\Delta \mathbf{n}_m$	5.4373		0.3882	
		T=313K		
$V^{E}(\times 10^{-3} m^{3} / mol)$	-	-0.0533	-	0.0005
v_m ($10 m / mol$)	0.1296		0.0207	
$\Lambda n(P_{q-s})$	2.7052	-0.0039	-	0.0004
$\Delta \eta (1 u^{-3})$			0.0067	
٨D	-	-1.7764	-	0.0142
$\Delta \mathbf{n}_m$	5.6581		0.8653	

		DMSO+DMF		
		T=293K		
V_m^E (×10 ⁻³ m ³ / mol)	- 0.1238	-0.0034	0.0257	0.0002
$\Delta\eta$ (Pa-s)	0.4271	-0.0096	- 0.0096	0.0003

0.0000

0.0940

953.0

966.3

ΔR_m	0.0169	0.0014	- 0.0149	0.0002
		T=303K		
$V_m^E(\times 10^{-3}m^3 / mol)$	0.1178	-0.0009	0.0037	0.0001
$\Delta\eta$ (Pa-s)	- 0.3396	-0.0009	0.0045	0.0002
ΔR_m	0.0160	0.0000	0.0016	0.0001
		T=313K		
$V_m^E(\times 10^{-3}m^3 / mol)$	0.0841	0.1101	0.1882	0.0001
$\Delta\eta$ (Pa-s)	0.1212	-0.0011	0.0033	0.0001
ΔR_m	0.0334	0.0007	0.0069	0.0001

Table III: The values of co-efficient a_i from Redlich Kister equation for V_m^E , $\Delta \eta$ and ΔR_m and standard deviation

$\sigma(Y^{E})$	for binary	mixtures
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DMSO+FA						
Temp(K)	L-L	G D	W	Н	Ν	Eyk
293	-	0.0027	0.0028	0.0028	0.0027	-
	0.0786					0.0738
303	-	-	-0.0062	-	-	-
	0.1146	0.0061		0.0061	0.0062	0.1078
313	-	-	-0.0128	-	-	-
	0.1115	0.0128		0.0127	0.0128	0.1335

DMSO+D

			MF			
 293	0.0007	0.0080	0.0080	0.0082	0.0078	0.0078
303	0.0058	0.0058	0.0059	0.0060	0.0057	0.0057
313	0.0058	0.0059	0.0059	0.0061	0.0057	0.0057

Table IV: Average percentage deviation for the mixing rules of refractive index (n) for DMSO + FA and DMSO + DMF mixtures at varying temperatures

Critical values	DMSO	FA	DMF
T_c (K)	727.56	765.33	643.15
$\omega_{_{SRK}}$	0.2985	0.4061	0.3672
V^*	0.2288	0.1305	0.2399
	DMSO + FA		
Temperature	293K	303K	313K
APD	293K 0.0311	303K 0.0684	313K 0.0676
APD	293K 0.0311	303K 0.0684 DMSO + DMF	<u>313K</u> 0.0676

 Table V: Critical values and estimated root mean square deviation of density from HBT model

V. DISCUSSION

In general, negative values of ΔR_m and V_m^E and positive values of $\Delta \eta$ indicate strong interaction in liquid mixtures which include charge transfer, dipole-dipole, dipole-induceddipole interaction etc. where as positive values of $\Delta R_{\rm m}$ and V_m^E and negative values of $\Delta \eta$ are indicative of the weakening of interaction between component molecules. ΔR_m measures the strength of interactions in mixtures and is sensitive functions of wavelength, temperature and mixture composition $^{15)}$. As can be seen from Figs. 1(a) and 1(b) that ΔR_{m} values are negative for DMSO + FA mixture and positive for DMSO + DMF mixture. An increase in the negative ΔR_m values and decrease in positive ΔR_m values with temperature indicate that the molecular association becomes stronger with temperature. The negative and positive values of ΔR_m in DMSO + FA and DMSO + DMF mixtures respectively indicate that stronger interaction is taking place between DMSO and FA molecules than that between DMSO and DMF molecules. This may be due to the fact that the steric hindrance of the two methyl groups of DMF makes specific interaction weaker in DMSO + DMF mixture exhibiting positive values of ΔR_m which in turn indicates the predominance of long range dispersive forces.



Figure 1: Molar refraction deviation (ΔR_m) as a function of mole fraction of DMSO (x_1) for (a) DMSO + FA and (b) DMSO + DMF mixtures.

The variation of V_m^E with mole fraction of DMSO for both the mixtures at three temperatures are presented in Figs. (2a) and (2b). The sign and magnitude of V_m^E play vital role in assessing for the molecular rearrangement as a result of molecular interactions between component molecules in the liquid mixtures ¹⁶⁾. The negative V_m^E values obtained for both the mixtures indicate the presence of strong intermolecular interaction between unlike molecules. Two effects mainly influence the negative values of V_m^E . The negative values of V_m^E over the entire mole fraction for both the mixture may be attributed mainly to the association through intermolecular hydrogen bonds between the oxygen atom of sulfoxide group and the hydrogen atom of amide group. Another effect, which would give negative contribution to the excess volumes, is the differences in molecular sizes between the two components in binary mixtures. The difference in free volumes between two components in binary mixtures would facilitate the penetration of one component into the other. The decrease in negative values of V_m^E for both the mixture with increase in temperature indicates the decrease in strength of molecular interaction with temperature.



Figure 2: Excess molar volume (V_m^E) as a function of mole fraction of DMSO (x_1) for (a) DMSO + FA and (b) DMSO + DMF mixtures.

Figure 3 illustrates that the deviation in viscosity $(\Delta \eta)$ are positive in DMSO + FA mixture and decreases with increase in temperature while $\Delta \eta$ is negative in DMSO + DMF mixture and decreases with increase in temperature. Positive contribution to $\Delta \eta$ values for DMSO + FA mixture suggest the presence of strong specific dipole-dipole

interaction between unlike molecules. The negative values of $\Delta \eta$ in case of DMSO + DMF mixture can be attributed to the fact that mixing of DMSO with DMF will induce the partial dissociation and loosening of dipolar interaction between DMSO molecules. Thus, the apparent intermolecular interaction in DMSO + DMF mixture may be arising from the weak dipole induced type forces resulting from the polarization of DMSO molecules by the dipoles of the surrounding DMF molecules. This effect can be superimposed upon dipole-dipole interaction and lead to a slight increase in attraction giving less negative $\Delta \eta$ values at higher temperatures. It can also be seen from Fig. 3 that specific interaction arising from induced dipole-dipole type force tends to increase with increasing temperature. Similar trends in ΔR_m , V_m^E and $\Delta \eta$ values have also been reported earlier 9,17,18)



Figure 3: Deviation in viscosity ($\Delta \eta$) as a function of mole fraction of DMSO (x_1) for (DMSO + FA) mixture (Mix 1) and (DMSO + DMF) mixture (Mix 2)

Further, theoretically estimated values of refractive index in terms of average percentage deviation (APD) using six mixing rules as described earlier ¹⁰⁾ are listed in Table IV for both the mixtures. All the mixing rules show good agreement between experimental and theoretically evaluated refractive index, Glad-Stone Dale (GD), Wiener's (W), Heller's (H), and Newton's (Nn) relations provide an excellent agreement between experimental and estimated refractive indices, with maximum APD of -0.0128 and minimum APD of -0.0027 in case of DMSO + FA mixture. However Lorentz-Lorenz (L-L) and Eykman's (Eyk) relations give maximum APD of -0.1115 and -0.1335 respectively for this mixture. Also the values of APD obtained from Wiener's and Heller's relations are very close for both the mixtures as expected since Heller's equation is simply a limiting form of the Wiener's equation and this trend is consistent with the observations reported earlier $^{19)}$. In the case of DMSO + DMF mixture, all the six relations exhibit an excellent agreement between the experimental and predicted refractive indices.

In order to predict density using HBT model, the critical values required for each compound along with root mean square deviation are given in Table V. The deviations obtained from HBT model for both the mixtures show that it is one of

the most accurate and most general model for prediction of mixing densities of the two binary mixtures.

VI. CONCLUSION

Results obtained in this paper seem to indicate that the stable inter molecular complexes are formed in case DMSO + FA mixture. However dipole-dipole interaction and dispersive forces seem to be responsible in DMSO + DMF mixture. Comparison of experimental and predicted values of refractive index and density in terms of APD / root mean square deviation for both the mixtures show the suitability of mixing rules and HBT model for representing the mixing refractive indices and densities respectively.

ACKNOWLEDGMENT

The authors are thankful to Prof. J.D. Pandey, Emeritus Fellow UGC, Former Head, Chemistry Department, Allahabad University for continuous support and valuable discussions.

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