



Transport Properties of Diethyl Malonate with Aniline and Benzaldehyde at 308.15 and 318.15 K

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Densities, viscosities and ultrasonic velocities of binary liquid mixtures of diethyl malonate with aniline and benzaldehyde have been measured at 308.15 and 318.15 K. From these data, excess volume (V^E), deviation in isentropic compressibility (ΔK_s), deviation in viscosity ($\Delta\eta$), deviation in intermolecular free length (ΔL_f), deviation in free volume (ΔV_f), deviation in internal pressure ($\Delta\pi_i$) and deviation in acoustic impedance (ΔZ) have been calculated. These results were correlated with the Redlich and Kister type polynomial equation to derive the coefficients and standard errors. Variations in the calculated excess quantities have been studied for mixtures of diethyl malonate with component liquids. From the value of excess properties, intermolecular interactions have been studied.

Keywords: Density, Viscosity, Ultrasonic velocity, Excess properties, Binary mixture, Molecular interaction.

INTRODUCTION

Study of transport properties is the most important way which provides information about molecular interactions between two liquids. Intermolecular interaction is the physical forces that attract or oppose the molecules each other. Every individual pure liquid is considered as an ideal solution, but when mix with other liquid it loses its ideal behaviour. The magnitude of deviation from ideal nature can give logical reason for intermolecular forces between liquid mixtures. This work is to determine the molecular interaction of binary mixtures through investigating thermodynamic properties. Diethyl malonate (DEM) is one of the useful ketoester molecule. Ketoesters are extremely polar and are self-associated by dipole-dipole interactions. The reactivity of its methylene group offer the series of reactions of alkylation, hydrolysis of the esters and decarboxylation resulting in substituted ketones. The methylene group in the center of the malonic part of the diethyl malonate molecule is neighbored by two carbonyl groups. The hydrogen atoms on a carbon nearby to two carbonyl groups are more acidic than hydrogen atom on the methyl group situated on the two ends. Diethyl malonate is an important starting material in pharmaceutical and agricultural chemical industries. From diethyl malonate several organic compounds can synthesize such as, vitamin B₁ and vitamin B₆, barbiturates, artificial flavouring agents. Volumetric, viscometric and optical study of molecular interactions in binary liquid mixtures of diethyl malonate with ketones have already been reported by Rathnam *et al.* [1]. Thermodynamic properties of diethyl

malonate with toluene and chlorobenzene have been studied by Revathy *et al.* [2]. Molecular interactions and geometric effects of diethyl malonate are investigated with the help of thermodynamic and acoustical data by Baluja *et al.* [3]. Theoretical evaluation of alkanols with diethyl malonate was studied by the Udayalakshmi *et al.* [4]. Intermolecular interaction of diethyl malonate with alkoxy alcohol have been studied by same team [5] with the help of theoretical ultrasonic velocity and viscosity values. Present work is to investigate the molecular interaction of diethyl malonate with aniline and benzaldehyde.

EXPERIMENTAL

Aniline (Merck Chemicals, Mumbai, Purity > 99 %) was dried over caustic potash for about 2 days and distilled twice. Benzaldehyde (Merck chemicals, Mumbai, Purity > 99 %) was purified by distillation and diethyl malonate (SRL Chemicals, Mumbai, purity > 99 %) was used without further purification. The purity of the solvents is established by comparing experimental values of densities, viscosities and ultrasonic velocities with reported literature values. Our experimental values of densities, viscosities and ultrasonic velocities match very well with those reported in the literature and are presented in Table-1.

Binary liquid mixtures of different compositions were prepared by mixing fixed quantity of pure liquids in air tight stoppered bottles of 50 mL capacity. Densities of pure liquids and liquid mixtures were measured by specific gravity method [10-12] with 10 mL relative density bottle and weighed with an exactness of $\pm 0.001 \text{ kg m}^{-3}$. Viscosities were determined

TABLE-1
COMPARISON OF EXPERIMENTAL DENSITY (ρ), VISCOSITY (η), ULTRASONIC
VELOCITY (U) OF PURE LIQUIDS WITH LITERATURE VALUES AT 308.15 AND 318.15 K

Liquids	T (K)	Density (ρ) (g cm ³)		Viscosity (η) (mPa.s)		Ultrasonic velocity (U) (m s ⁻¹)	
		Exp.	Lit.	Exp.	Lit.	Exp.	Lit.
Diethyl malonate	308	1.0418	1.0387 [Ref. 3]	1.6003	1.602 [Ref. 5]	1277.0	1267.0 [Ref. 3]
	318	1.0283	–	1.4235	1.425 [Ref. 5]	1235.5	1244.0 [Ref. 4]
Benzaldehyde	308	1.0381	1.0313 [Ref. 6]	1.2483	1.249 [Ref. 6]	1417.5	1421.2 [Ref. 7]
	318	1.0233	1.0229 [Ref. 7]	1.0508	–	1395.0	1384.7 [Ref. 7]
Aniline	308	1.0123	1.0129 [Ref. 8]	2.8692	2.800 [Ref. 9]	1614.0	1614.0 [Ref. 8]
	318	0.9990	1.0049 [Ref. 8]	2.4079	–	1582.5	1582.0 [Ref. 8]

by Oswald viscometer 10 mL capability with an accuracy of ± 0.001 cP [10–12]. From the measured values of density and flow time 't', viscosity ' η ' was calculated with the relation:

$$\eta = (A \times t - B/t) \times \rho \quad (1)$$

where A and B are viscometer constant. The values of constants were occurred by measuring the flow time with distilled water and pure nitrobenzene as standard liquids. The flow time were measured with electronic stop clock. Ultrasonic velocities of pure and liquid mixtures were measured by a single crystal variable path interferometer (Pico Enterprises Chennai, Model BL-02) [12] at a frequency of 2 MHz with an accuracy of ± 0.02 %. All the measurements were finished at both 308.15 and 318.15 K with the assist of a digital thermostat with a temperature precision of ± 0.01 K.

RESULTS AND DISCUSSION

Experimental values of density (ρ), viscosity (η) and ultrasonic velocity (u) were measured from which, calculated

values of excess volume (V^E), deviation in isentropic compressibility (ΔK_s), ΔL_f and ΔZ for the two binary mixtures are presented in Table-2.

The excess volume V^E was calculated by the relation [9,13,14]:

$$V^E = \left(\frac{[X_A M_A + X_B M_B]}{\rho_{AB}} \right) - \left(\frac{X_A M_A}{\rho_A} + \frac{X_B M_B}{\rho_B} \right) \quad (2)$$

where X_A, X_B are mole fraction of components A & B, M_A, M_B are the molecular mass of components A & B. ρ_A, ρ_B , are the density of component A, B respectively and ρ_{AB} is the density of mixture.

$$K_s = 1/U^2 \rho \quad (3)$$

$$L_f = k/U\rho^{1/2} \quad (4)$$

$$Z = U\rho \quad (5)$$

where U is ultrasonic velocity of pure liquid and liquid mixtures and ρ is the density of pure and mixture, k is Jacobson's constant [11,15] which is temperature dependant constant but not character

TABLE-2
PHYSICAL AND THERMODYNAMIC PARAMETERS FOR BINARY MIXTURES OF DIETHYL MALONATE +
ANILINE AND DIETHYL MALONATE + BENZALDEHYDE AT 308.15 AND 318.15 K

308.15 K						318.15 K					
x_1	ρ (g cm ⁻³)	η (mPa s ⁻¹)	U (m s ⁻¹)	V^E (cm ³ mol ⁻¹)	ΔK_s (Tpa ⁻¹)	x_1	ρ (g cm ⁻³)	η (mPa s ⁻¹)	U (m s ⁻¹)	V^E (cm ³ mol ⁻¹)	ΔK_s (Tpa ⁻¹)
Diethyl malonate + Aniline						Diethyl malonate + Aniline					
0.0000	1.0123	2.8692	1614.00	0.0000	0.0000	0.0000	0.9999	2.4079	1582.50	0.0000	0.0000
0.1133	1.0190	2.7648	1589.50	-0.1416	-27.6327	0.1133	1.0058	2.3216	1539.50	-0.0857	-21.7135
0.1933	1.0230	2.6925	1573.50	-0.2284	-44.2584	0.1933	1.0096	2.2632	1512.50	-0.1652	-34.3840
0.2898	1.0272	2.5988	1550.00	-0.3143	-58.9070	0.2898	1.0136	2.1916	1484.50	-0.2402	-48.1061
0.3888	1.0308	2.4942	1526.50	-0.3650	-70.7791	0.3888	1.0171	2.1118	1453.50	-0.2879	-56.4996
0.4891	1.0336	2.3816	1495.50	-0.3733	-75.4974	0.4891	1.0199	2.0225	1422.00	-0.3021	-60.7925
0.5897	1.0360	2.2447	1456.50	-0.3462	-72.0263	0.5897	1.0222	1.9159	1384.00	-0.2798	-56.4945
0.6888	1.0377	2.0935	1413.50	-0.2832	-61.7292	0.6888	1.0239	1.7962	1347.00	-0.2178	-48.2337
0.7918	1.0392	1.9347	1369.50	-0.1928	-47.0816	0.7918	1.0255	1.6709	1307.00	-0.1460	-33.9373
0.8797	1.0405	1.7955	1332.00	-0.1225	-31.0514	0.8797	1.0267	1.5659	1275.00	-0.0738	-19.9330
1.0000	1.0418	1.6003	1277.00	0.0000	0.0000	1.0000	1.0283	1.4235	1235.50	0.0000	0.0000
Diethyl malonate + Benzaldehyde						Diethyl malonate + Benzaldehyde					
0.0000	1.0381	1.2483	1417.50	0.0000	0.0000	0.0000	1.0233	1.0508	1395.00	0.0000	0.0000
0.1005	1.0389	1.3011	1411.00	-0.0238	-11.6329	0.1005	1.0242	1.0983	1379.50	-0.0147	-8.4637
0.2038	1.0396	1.3508	1401.50	-0.0438	-20.0162	0.2038	1.0250	1.1478	1363.50	-0.0293	-14.8384
0.2953	1.0401	1.3929	1392.50	-0.0561	-25.7686	0.2953	1.0256	1.1923	1350.50	-0.0406	-19.6709
0.3958	1.0405	1.4366	1381.50	-0.0654	-30.0451	0.3958	1.0262	1.2384	1337.00	-0.0509	-23.9482
0.5002	1.0409	1.4766	1368.00	-0.0689	-31.6593	0.5002	1.0268	1.2827	1322.00	-0.0563	-25.9018
0.5888	1.0412	1.5059	1354.50	-0.0654	-30.4725	0.5888	1.0271	1.3136	1307.50	-0.0519	-24.7571
0.6897	1.0414	1.5348	1337.00	-0.0568	-26.2769	0.6897	1.0275	1.3444	1290.00	-0.0413	-21.1095
0.7922	1.0416	1.5611	1319.00	-0.0415	-20.5399	0.7922	1.0278	1.3719	1270.50	-0.0275	-14.2379
0.8937	1.0417	1.5838	1299.50	-0.0225	-12.1391	0.8937	1.0280	1.3975	1252.50	-0.0143	-7.1124
1.0000	1.0418	1.6003	1277.00	0.0000	0.0000	1.0000	1.0283	1.4235	1235.50	0.0000	0.0000

where X_{exp} and X_{cal} represents experimental and theoretical values of excess properties V^E , ΔK_S , ΔL_f , $\Delta \eta$, ΔV_f , $\Delta \pi_i$ and ΔZ . N is the number of data points and n is the number of coefficients (Table-3).

$$\pi_i = bRT (k\eta/U)^{1/2} (\rho^{2/3}/M_{\text{eff}}^{7/6}) \quad (7)$$

$$\sigma = \left(\frac{\sum (X_{\text{exp}} - X_{\text{cal}})^2}{N - n} \right)^{1/2} \quad (11)$$

According to Sridevi *et al.* [23], negative excess values are due to closely packed molecules which are the causes of the existence of strong molecular interactions, whereas positive excess values revealed the weak interactions between unlike molecules. The entire range of negative values [24,25] of V^E and ΔK_s (Figs. 1 and 2) for diethyl malonate with aniline specify the presence of molecular interaction between unlike molecules. It may be explained by the factor of chemical contribution. The reduction in the mixture of DEM with aniline is caused by an intensification of the intermolecular forces on mixing. There may be a strong attractive force between the

308.15 K					318.15 K				
$\Delta\eta$ (mPa.s)	$\Delta L_f \cdot 10^{-10}$ (m)	ΔV_f (m ³ mol ⁻¹)	ΔZ (Kg m ⁻² s ⁻¹)	$\Delta\pi_i$ (Pa)	$\Delta\eta$ (mPa.s)	$\Delta L_f \cdot 10^{-10}$ (m)	ΔV_f (m ³ mol ⁻¹)	ΔZ (Kg m ⁻² s ⁻¹)	$\Delta\pi_i$ (Pa)
Diethyl malonate + Aniline					Diethyl malonate + Aniline				
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0394	-0.4200	-1.0837×10 ⁻¹⁵	20.1673	-0.0021	0.0252	-0.1523	-1.3378×10 ⁻¹⁵	1.3741	-0.00173344
0.0685	-0.7149	-1.7932×10 ⁻¹⁵	34.4584	-0.0027	0.0455	-0.2904	-2.2179×10 ⁻¹⁵	4.9554	-0.00228073
0.0974	-0.9823	-2.5805×10 ⁻¹⁵	46.2592	-0.0030	0.0690	-0.5059	-3.1775×10 ⁻¹⁵	12.7391	-0.00252354
0.1184	-1.2376	-3.2619×10 ⁻¹⁵	57.5870	-0.0029	0.0867	-0.6482	-4.0344×10 ⁻¹⁵	17.1789	-0.0024778
0.1331	-1.3432	-3.8285×10 ⁻¹⁵	60.3582	-0.0026	0.0961	-0.7506	-4.7016×10 ⁻¹⁵	20.4597	-0.00225035
0.1239	-1.2710	-4.1606×10 ⁻¹⁵	53.9412	-0.0022	0.0885	-0.6793	-5.0902×10 ⁻¹⁵	16.2414	-0.00190909
0.0983	-1.0635	-4.1499×10 ⁻¹⁵	41.9703	-0.0018	0.0663	-0.5638	-4.9846×10 ⁻¹⁵	11.6502	-0.00150472
0.0702	-0.8021	-3.663×10 ⁻¹⁵	29.6344	-0.0012	0.0424	-0.3571	-4.3292×10 ⁻¹⁵	4.9084	-0.00103499
0.0425	-0.5343	-2.6605×10 ⁻¹⁵	18.9637	-0.0007	0.0239	-0.1842	-3.1333×10 ⁻¹⁵	0.9946	-0.00060867
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Diethyl malonate + Benzaldehyde					Diethyl malonate + Benzaldehyde				
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0175	-0.1855	-8.6487×10 ⁻¹⁷	8.5085	-0.0002	0.0100	-0.0582	-2.198×10 ⁻¹⁶	1.0967	-0.0002
0.0307	-0.3168	-1.5104×10 ⁻¹⁶	14.1582	-0.0004	0.0210	-0.1071	-5.0741×10 ⁻¹⁶	2.0157	-0.0003
0.0406	-0.4169	-2.0156×10 ⁻¹⁶	18.4108	-0.0004	0.0314	-0.1678	-7.6108×10 ⁻¹⁶	3.9168	-0.0003
0.0490	-0.4997	-2.5412×10 ⁻¹⁶	21.7976	-0.0004	0.0401	-0.2438	-9.1796×10 ⁻¹⁶	6.6848	-0.0003
0.0522	-0.5367	-2.6512×10 ⁻¹⁶	23.0107	-0.0004	0.0454	-0.2912	-9.9863×10 ⁻¹⁶	8.3993	-0.0003
0.0504	-0.5193	-2.4751×10 ⁻¹⁶	21.8194	-0.0003	0.0433	-0.2817	-9.1037×10 ⁻¹⁶	7.9072	-0.0003
0.0438	-0.4449	-2.1666×10 ⁻¹⁶	18.1459	-0.0003	0.0365	-0.2358	-7.3698×10 ⁻¹⁶	6.2077	-0.0002
0.0340	-0.3530	-1.5926×10 ⁻¹⁶	14.1038	-0.0002	0.0258	-0.1344	-5.4303×10 ⁻¹⁶	2.6582	-0.0002
0.0209	-0.2118	-1.1325×10 ⁻¹⁶	8.2885	-0.0001	0.0136	-0.0521	-3.0184×10 ⁻¹⁶	0.4456	-0.0001
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

TABLE-4
COEFFICIENT VALUES OF REDLICH-KISTER TYPE POLYNOMIAL EQUATION (eqn. 10)
AND STANDARD DEVIATION (eqn. 11) AT DIFFERENT TEMPERATURES

T (K)	Diethyl malonate + Aniline				Diethyl malonate + Benzaldehyde			
	a	b	c	σ	a	b	c	σ
	V^E ($\text{cm}^3 \text{mol}^{-1}$)				V^E ($\text{cm}^3 \text{mol}^{-1}$)			
308	-1.48791	0.243523	0.413219	0.000064	-0.273882	0.0126909	0.0387452	4.6496×10^{-6}
318	-1.20673	0.167536	0.703787	0.000038	-0.217936	0.00851364	0.118165	3.35735×10^{-5}
	η (mPa.s)				η (mPa.s)			
308	8.81185	-4.18187	18.705	0.005431	5.43051	1.17509	12.7468	0.005077
318	7.48685	-3.27111	15.9585	0.009448	4.71602	1.25224	10.9898	0.004343
	ΔK_S (Tpa $^{-1}$)				ΔK_S (Tpa $^{-1}$)			
308	-277.438	-155.036	-26.9082	0.804768	-120.003	-49.738	-15.2657	0.194161
318	-225.92	-115.596	25.5992	0.457174	-97.5591	-41.8661	16.3227	0.094112
	$\Delta\eta$ (mPa s)				$\Delta\eta$ (mPa s)			
308	0.5158	-0.0118	-0.2305	4.89927×10^{-14}	2.06×10^{-1}	1.37×10^{-2}	-1.36×10^{-2}	1.88662×10^{-5}
318	0.3739	-0.0320	-0.2634	8.20779×10^{-14}	1.78×10^{-1}	2.21×10^{-2}	-9.20×10^{-2}	2.91517×10^{-5}
	ΔZ ($\text{Kg m}^{-2} \text{s}^{-1}$)				ΔZ ($\text{Kg m}^{-2} \text{s}^{-1}$)			
308	233.74	-34.0154	-92.2923	0.026706	90.1391	-3.19258	-7.0685	0.013223
318	76.7498	-8.91207	-119.546	0.009677	32.0433	4.30517	-45.5929	0.012929
	ΔL_f (10^{-10}m)				ΔL_f (10^{-10}m)			
308	-5.26×10^{-10}	-2.63×10^{-11}	1.42×10^{-10}	1.07911×10^{-9}	-2.11×10^{-10}	-1.29×10^{-11}	1.04×10^{-11}	1.15808×10^{-9}
318	6.52×10^{-12}	2.27×10^{-11}	-7.43×10^{-11}	0.003113	2.56×10^{-12}	8.61×10^{-12}	-2.92×10^{-11}	1.23601×10^{-9}
	ΔV_f ($10^{-15} \text{m}^3 \text{mol}^{-1}$)				ΔV_f ($10^{-15} \text{m}^3 \text{mol}^{-1}$)			
308	-1.40×10^{-14}	-9.14×10^{-15}	-1.43×10^{-14}	1.78786×10^{-17}	-9.70×10^{-16}	-4.94×10^{-17}	-3.39×10^{-16}	4.91537×10^{-15}
318	-1.78×10^{-14}	-1.03×10^{-14}	-1.22×10^{-14}	1.52288×10^{-15}	-3.84×10^{-15}	-3.41×10^{-17}	1.50×10^{-15}	6.87135×10^{-15}
	$\Delta\pi_i$ (Pa)				$\Delta\pi_i$ (Pa)			
308	-1.04×10^{-2}	8.10×10^{-3}	-5.16×10^{-3}	0.000500015	-1.56×10^{-3}	8.43×10^{-4}	-3.79×10^{-4}	0.00017746
318	-8.84×10^{-3}	6.80×10^{-3}	-4.28×10^{-3}	2.72477×10^{-7}	-1.29×10^{-3}	6.81×10^{-4}	-3.14×10^{-4}	0.000157442

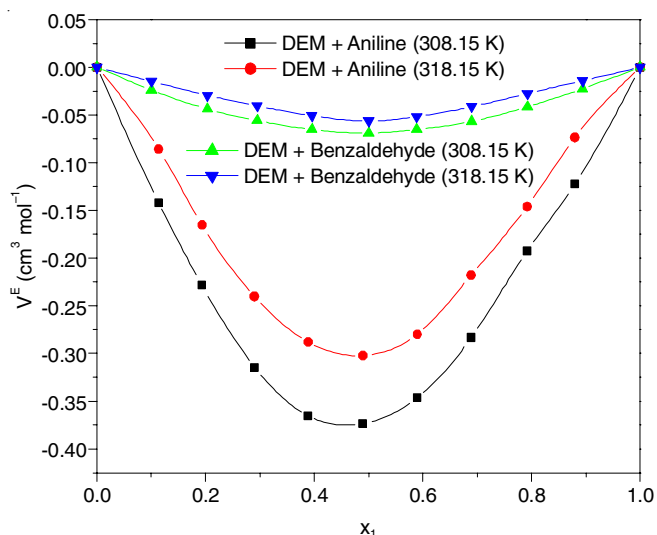


Fig. 1. V^E for diethyl malonate (DEM) with aniline and benzaldehyde at 308.15 and 318 K

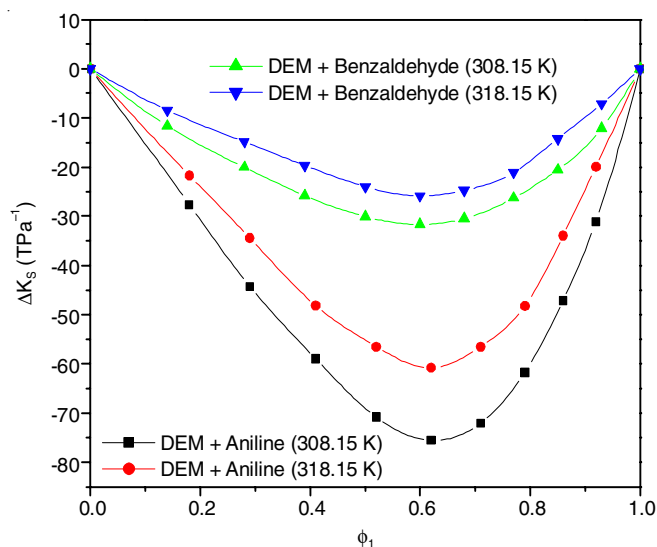


Fig. 2. ΔK_S for diethyl malonate (DEM) with aniline and benzaldehyde at 308.15 and 318.15 K

hydrogen atom of the aniline and the carbonyl oxygen atom of the DEM. The observed ΔL_f , ΔV_f , $\Delta\pi_i$ values (Figs. 3-5) also display negative values over the entire array of composition clearly representing the presence of strong hydrogen bonding interactions between unlike molecules.

Both excess volume V^E and deviation in isentropic compressibility ΔK_S values of DEM + benzaldehyde is negative (Figs. 1 and 2) over the entire range of composition [26,27], shows that the chemical contribution between two components. This may be attributed by the dipole-dipole interaction. Keto-

esters and aldehydes both have the carbonyl group, hence they turn partially dipoles due to inductive effect, so the carbon atom of the ester group is attracted by the oxygen atom of the benzaldehyde, hence it involve dipole-dipole interaction and contract in volume. The observed value of ΔL_f , ΔV_f , $\Delta\pi_i$ (Figs. 3-5) reflect the same idea as obtained above. The $\Delta\pi_i$ values of the liquid mixture decreases and ΔV_f values increases with the increase in temperature for the two mixtures. The nature of interaction for the two mixtures decrease as the temperature is increased due to increase in thermal motion of interacting molecules.

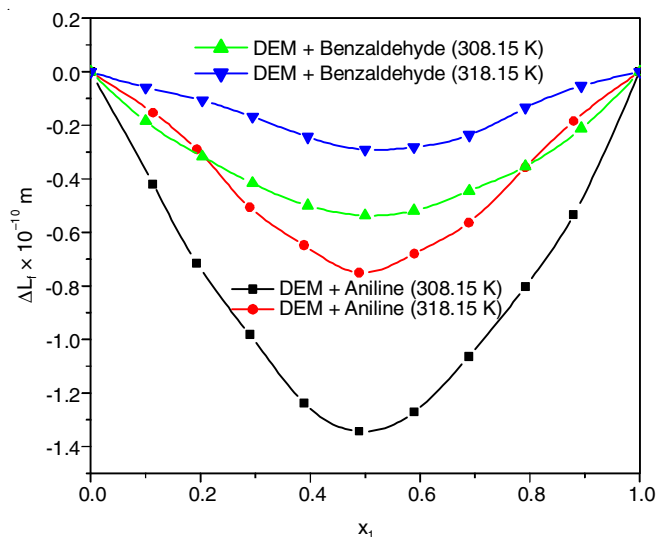


Fig. 3. ΔL_r for diethyl malonate with aniline and benzaldehyde at 308.15 and 318.15 K

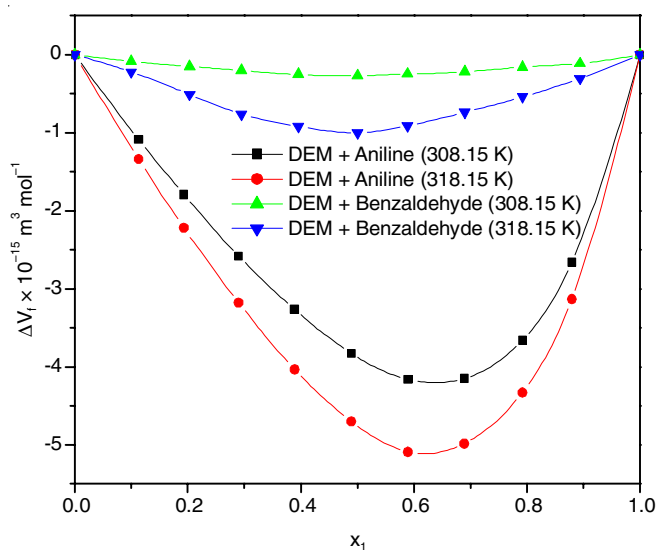


Fig. 4. ΔV_r for diethyl malonate with aniline and benzaldehyde at 308.15 and 318.15 K

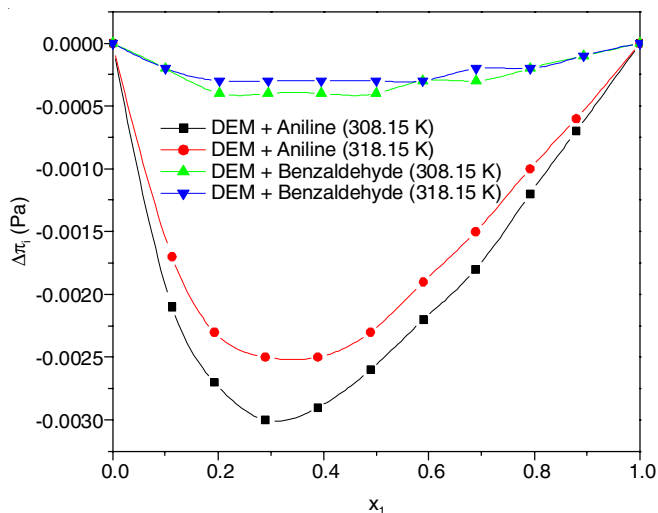


Fig. 5. $\Delta \pi_i$ for diethyl malonate with aniline and benzaldehyde at 308.15 and 318.15 K

According to Iloukhani and Rezaei-Sameti [28] negative values of $(\Delta\eta)$ arise for system of dissimilar molecular sizes due to dispersion forces, accountable for the interactions and positive values of $(\Delta\eta)$ responsible for the interaction forces through hydrogen bonding. Positive deviations are due to the substitute of like molecules [29]. ΔZ behaves in an opposite manner to ΔL_r , positive and negative deviation of the mixtures indicate the extent of association or dissociation between the mixing components [30–33].

The observed values of $\Delta\eta$ and ΔZ are positive over the entire range (Figs. 6 and 7) that strongly supported to the above idea. Between the two mixtures aniline liquid mixture has the highest negative value than benzaldehyde, which indicates strong specific interaction between mixing liquids and obviously hydrogen bond is strong intermolecular force than dipole-dipole interaction:

DEM + aniline > DEM + benzaldehyde

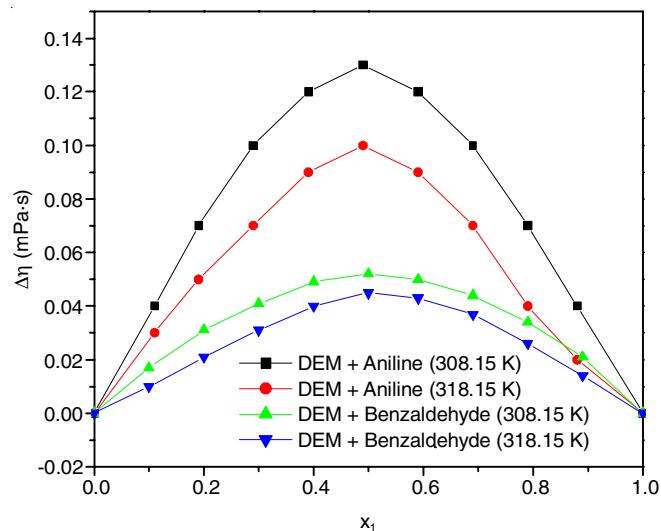


Fig. 6. $\Delta\eta$ for diethyl malonate with aniline and benzaldehyde and at 308.15 and 318.15 K

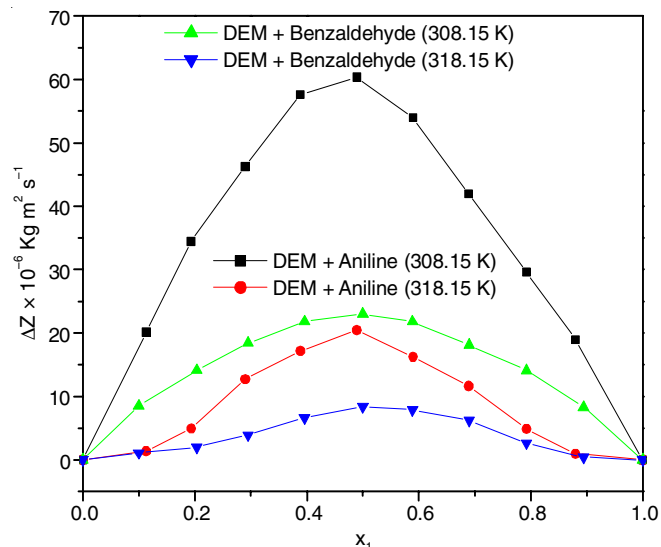


Fig. 7. ΔZ for diethyl malonate with aniline and benzaldehyde at 308.15 and 318.15 K

Conclusion

This work has determined the intermolecular interaction for the mixtures of diethyl malonate with aniline and benzaldehyde. The magnitude of V^E , ΔK_S , ΔL_r , ΔV_f , $\Delta \pi_i$, $\Delta \eta$ and ΔZ have been interpreted in terms of different molecular interaction between these molecules. The experimental excess properties for DEM with aniline shows existence of strong hydrogen bond between them, there is dipole-dipole interaction between DEM and benzaldehyde. The intermolecular interaction decreases with increasing temperature.

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