

FTIR and ultrasonic studies on molecular interactions of ester with butanol in the temperature range 303.15–318.15 K

M. Srilatha^{1,*}, B. Venkateswara Rao² and B. Vijaya Saradhi²

¹Department of Engineering Chemistry, and

²Department of Civil Engineering, College of Engineering, Andhra University, Visakhapatnam 530 003, India

Ultrasonic velocity, viscosity and density of ethyl oleate in butanol have been determined at various temperatures in the range 303.15–318.15 K. These data are used to estimate adiabatic compressibility, intermolecular free length and acoustic impedance, molar volume and free volume along with their excess values. The observed variations in these parameters with different concentrations and temperatures are discussed in terms of the intermolecular interactions between the unlike molecules of the binary mixtures.

Keywords: Butanol, ethyl oleate, density, ultrasonic speed, viscosity.

THE knowledge of acoustical parameters is of importance in studying the molecular interactions and physico-chemical behaviour in binary liquid mixtures^{1,2}. The study of molecular interactions in the liquid mixtures is of considerable significance in the elucidation of the structural properties of the molecules. The intermolecular interactions influence the structural arrangement along with the shape of the molecules. In recent times, the ultrasonic technique is found to be one of the most powerful tools for studying the nature of molecular interactions in liquid mixtures. Acoustical parameters are used to understand different kinds of association, molecular packing, molecular motion, physico-chemical behaviour and various types of intermolecular interactions and their strengths, influenced by the size in pure components and in the mixtures. As a part of the current progressive and ongoing research^{3,4} on thermodynamic and acoustic properties of binary liquid mixtures, here we report the results of a study on a binary mixture of butanol and hexane over the entire range of composition at $T = 298.15, 303.15$ and 308.15 K. Employing the experimental values of ultrasonic velocity (U), viscosity (η) and density (ρ), the thermodynamic parameters such as adiabatic compressibility (β_{ad}) and intermolecular free length (L_f) have been estimated using standard relations.

Materials and experiments

All the materials were procured from Sigma-Aldrich, USA (AR grade) and glassware used of borosilicate make. Organic liquids ethyl oleate and butanol of AR grade were used directly without purification. The densities and viscosities of the liquid compounds were measured at different temperatures⁵ with a specific gravity bottle and Ostwald viscometer pre-calibrated with 3D water of Millipore to the nearest milligram per millilitre. The time taken for flow of viscous fluid in Ostwald viscosity meter was measured to the nearest 0.01 sec. An electronic digital stopwatch with an accuracy of ± 0.01 sec was used for flow-time measurement. Borosilicate glassware, Shimadzu electronic balance (Japan-make) of sensitivity $+0.001$ g and an electrically operated constant temperature water bath of accuracy $+0.1$ K were used to circulate water through the double-walled measuring cell made up of steel containing the experimental solution at the desired temperature while conducting the experiments. A 2 MHz ultrasonic interferometer, Mittal Enterprises (model no. F-05)⁵ with least count of micrometre 0.001 mm was used for calculating velocities of sound waves and all the tests were conducted according to ASTM standard procedures.

Theory and calculations

In order to examine the intermolecular interactions in liquid mixtures of ethyl oleate with butanol, experiments were conducted to find the density, viscosity and velocity of 2 MHz ultrasonic waves for pure liquids and binary liquid mixtures. The results of pure liquids were compared with the literature values for assessment. From the experimental data of binary mixtures, the derived and excess values were calculated at various mole fractions of ethyl oleate for understanding inter- and intramolecular interactions at each temperature. The derived and excess values were calculated as discussed below.

The acoustical parameters were calculated from U , ρ and η as follows⁶⁻⁹. The experimental values for pure

*For correspondence. (e-mail: srilatha.manukonda@gmail.com)

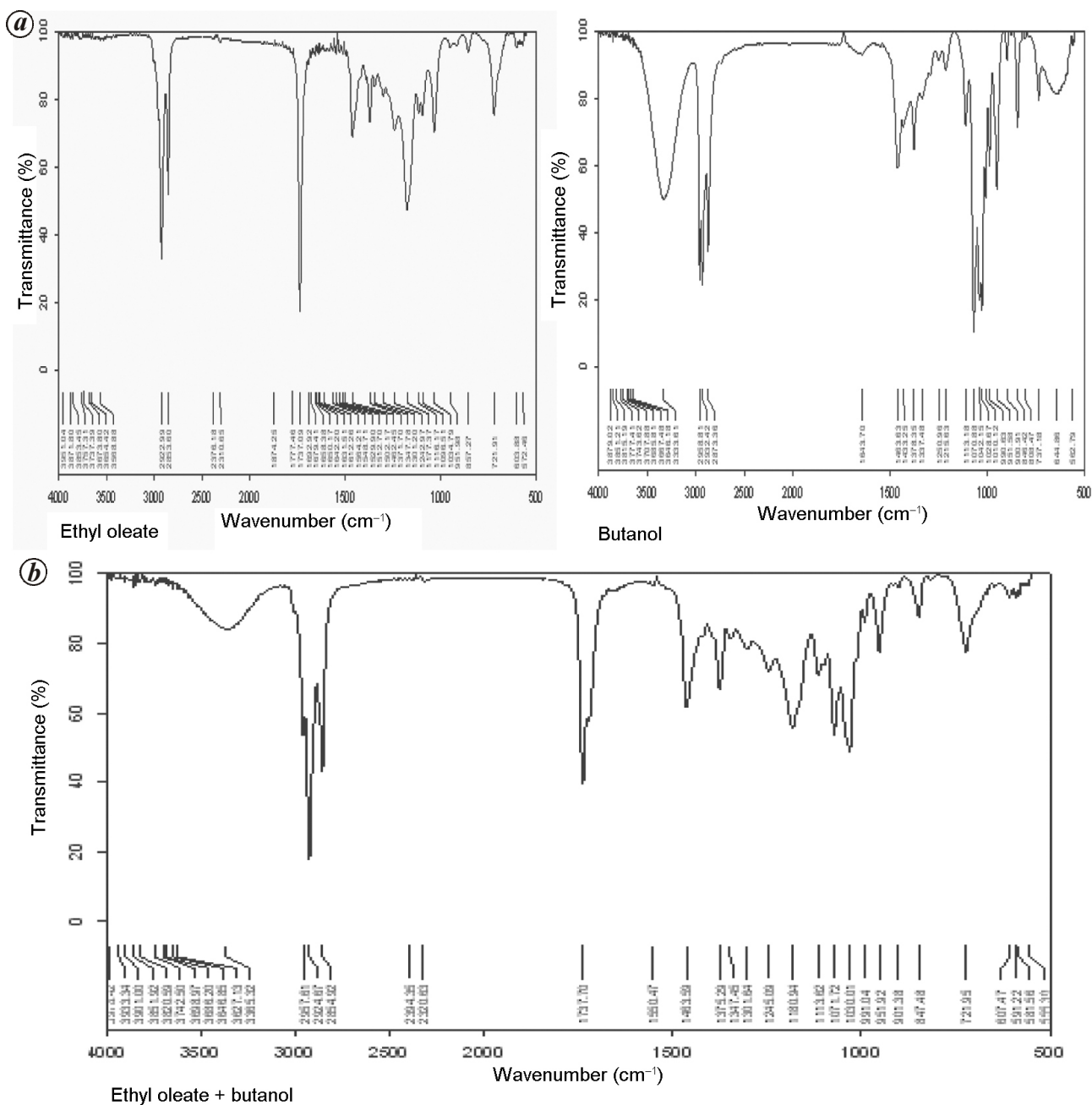


Figure 1. FTIR spectrum of (a) pure ethyl oleate and pure butanol and (b) ethyl oleate + butanol.

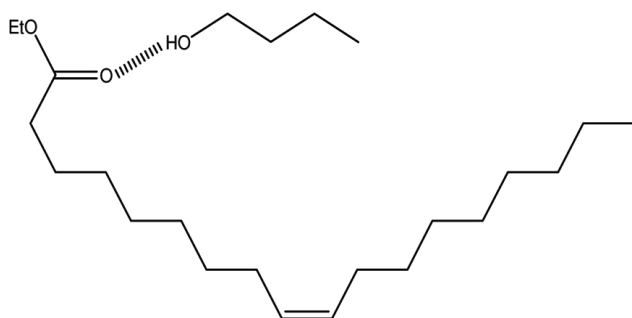


Figure 2. Hydrogen bonding between ethyl oleate and butanol.

liquids are compared with the literature values and given in Table 1 (refs 10–18).

Ultrasonic velocity

The relation used to determine the ultrasonic velocity (U ; $m\ s^{-1}$) is given by

$$U = f\lambda,$$

where f is the frequency of ultrasonic waves and λ is the wavelength.

Table 1. Experimental and literature values of density, viscosity and velocity of 2 MHz ultrasonic wave for pure butanol

Parameter	303.15 K		308.15 K		313.15 K		318.15 K	
	Experimental value	Literature value	Experimental value	Literature value	Experimental value	Literature value	Experimental value	Literature value
Density (ρ) kg/m ³	802.60	804.20 (ref. 10)	798.10	803.30 (ref. 10)	794.20	802.20 (ref. 10)	790.20	790.97 (ref. 11)
		801.90 (ref. 11)		798.25 (ref. 11)		794.60 (ref. 11)		787.20 (ref. 13)
		802.10 (ref. 12)		792.10 (ref. 13)		798.20 (ref. 12)		790.29 (ref. 16)
		802.20 (ref. 13)		802.10 (ref. 15)		792.30 (ref. 13)		
		804.40 (ref. 15)		798.12 (ref. 16)		798.50 (ref. 15)		
		802.01 (ref. 16)		798.00 (ref. 17)		794.31 (ref. 16)		
Viscosity (η) N.s/m ²	2.2223	2.2870 (ref. 12)	1.9536	2.0138 (ref. 12)	1.6806	1.7750 (ref. 13)	1.4859	1.5712 (ref. 13)
		2.2940 (ref. 13)		2.0123 (ref. 13)		1.6872 (ref. 14)		1.4879 (ref. 14)
		2.2243 (ref. 14)		1.9560 (ref. 14)		1.6302 (ref. 15)		
		2.1508 (ref. 15)		1.8643 (ref. 15)		1.8000 (ref. 18)		
		2.2900 (ref. 18)						
Velocity (U) m/s	1228.1	1220.60 (ref. 10)	1206.7	1212.00 (ref. 10)	1189.8	1188.80 (ref. 10)	1176.8	1170.00 (ref. 11)
		1224.60 (ref. 11)		1208.80 (ref. 11)		1189.50 (ref. 11)		1160.50 (ref. 13)
		1225.80 (ref. 13)		1202.30 (ref. 13)		1181.50 (ref. 13)		1173.38 (ref. 16)
		1229.10 (ref. 15)		1211.00 (ref. 15)		1198.40 (ref. 15)		
		1223.25 (ref. 16)		1206.56 (ref. 16)		1182.92 (ref. 16)		
				1207.76 (ref. 17)				

Table 2. Ultrasonic velocity, density, viscosity, adiabatic compressibility, intermolecular free length and molar volume values

Mole fraction (X_1)	Mole fraction (X_2)	Ultrasonic velocity (m/s)	Density (kg/m ³)	Viscosity (N.S m ⁻²)	Adiabatic compressibility (10 ⁻¹⁰ m ² /N (β_{ad}))	Intermolecular free length (10 ⁻¹⁰ m)	Molecular volume
303.15 K							
0.0000	1.0000	1228.1	802.6	2.2223	8.2610	5.9639	92.349
0.0488	0.9511	1251.4	814.2	2.7367	7.8423	5.8108	105.21
0.1137	0.8862	1274.7	825.4	3.2512	7.4552	5.6656	122.38
0.2043	0.7956	1298.1	836.5	3.7657	7.0942	5.5267	146.35
0.3393	0.6606	1321.4	847.0	4.2801	6.7609	5.3953	182.21
0.5621	0.4378	1344.8	856.3	4.7946	6.4574	5.2728	241.76
1.0000	0.0000	1368.1	863.5	5.3091	6.1869	5.1612	359.60
308.15 K							
0.0000	1.0000	1206.7	798.1	1.9536	8.6048	6.1454	92.870
0.0488	0.9511	1229.0	809.4	2.4140	8.1789	5.9914	105.85
0.1137	0.8862	1251.4	820.6	2.8744	7.7817	5.8441	123.12
0.2043	0.7956	1273.7	831.7	3.3348	7.4107	5.7031	147.22
0.3393	0.6606	1296.1	842.4	3.7952	7.0665	5.5691	183.24
0.5621	0.4378	1318.4	851.9	4.2556	6.7528	5.4440	243.02
1.0000	0.0000	1340.8	859.3	4.7160	6.4733	5.3302	361.28
313.15 K							
0.0000	1.0000	1189.8	794.2	1.6806	8.8945	6.3077	93.326
0.0488	0.9511	1212.1	805.2	2.1032	8.4520	6.1487	106.41
0.1137	0.8862	1234.5	816.4	2.5258	8.0365	5.9957	123.77
0.2043	0.7956	1256.9	827.6	2.9484	7.6479	5.8490	147.99
0.3393	0.6606	1279.3	838.4	3.3710	7.2875	5.7095	184.17
0.5621	0.4378	1301.7	847.9	3.7936	6.9602	5.5798	244.24
1.0000	0.0000	1324.0	855.62	4.2162	6.6662	5.4607	362.91
318.15 K							
0.0000	1.0000	1176.8	790.2	1.4859	9.1381	6.4539	93.799
0.0488	0.9511	1198.1	801.2	1.8686	8.6938	6.2951	106.96
0.1137	0.8862	1219.5	812.4	2.2514	8.2759	6.1419	124.43
0.2043	0.7956	1240.9	823.7	2.6341	7.8835	5.9945	148.76
0.3393	0.6606	1262.3	834.8	3.0169	7.5174	5.8537	185.06
0.5621	0.4378	1283.7	844.5	3.3997	7.1856	5.7230	245.33
1.0000	0.0000	1305.0	852.6	3.7824	6.8860	5.6025	364.20

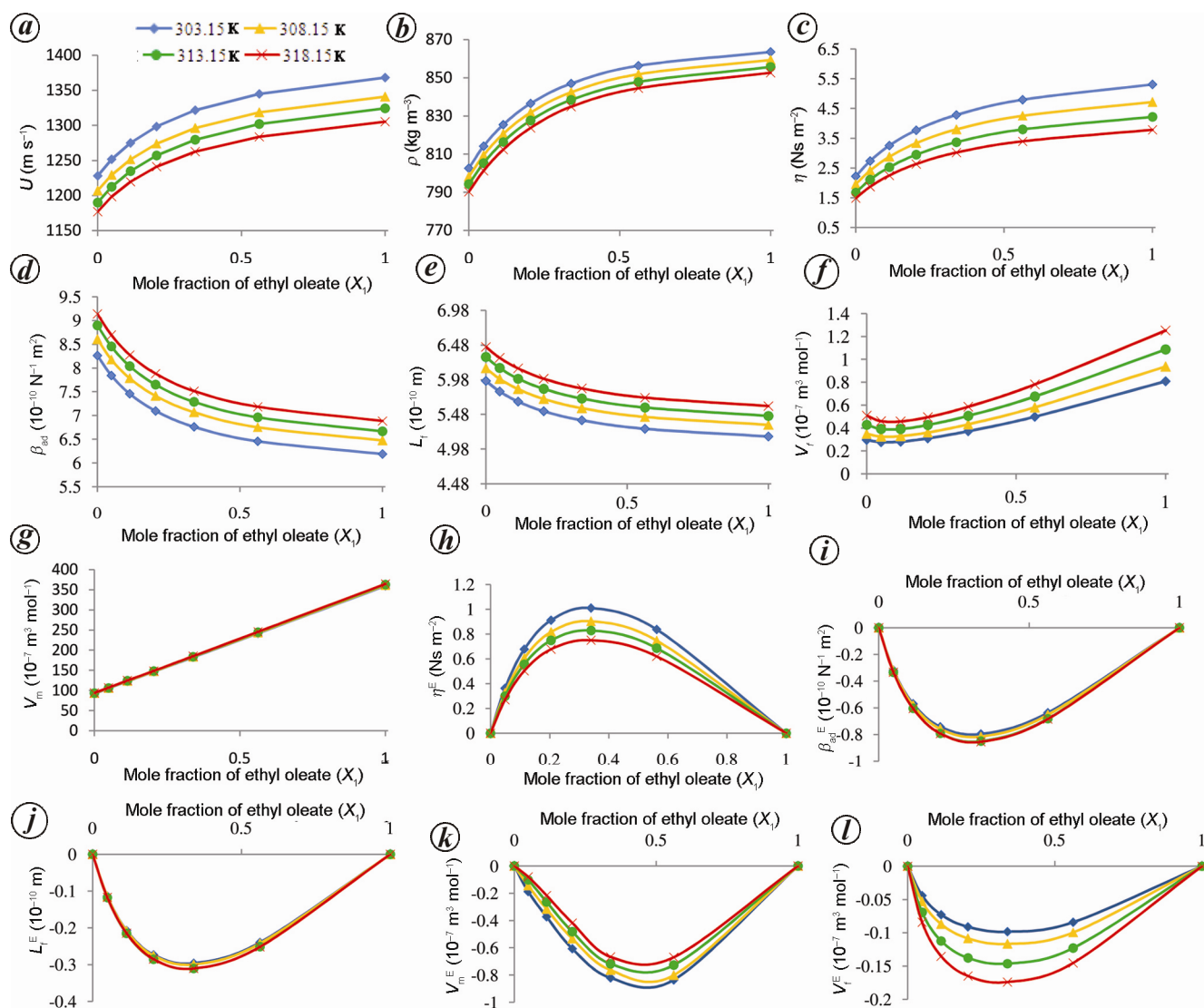


Figure 3. *a*, Ultrasonic velocity versus mole fraction of ethyl oleate (X_1); *b*, Density versus mole fraction of ethyl oleate (X_1); *c*, Viscosity versus mole fraction of ethyl oleate (X_1); *d*, Adiabatic compressibility versus mole fraction of ethyl oleate (X_1); *e*, Intermolecular free length versus mole fraction of ethyl oleate (X_1); *f*, Free volume versus mole fraction of ethyl oleate (X_1); *g*, Molar volume versus mole fraction of ethyl oleate (X_1); *h*, Deviation in viscosity versus mole fraction of ethyl oleate (X_1); *i*, Excess adiabatic compressibility versus mole fraction of ethyl oleate (X_1); *j*, Excess intermolecular free length versus mole fraction of ethyl oleate (X_1); *k*, Excess molar volume versus mole fraction of ethyl oleate (X_1); *l*, Excess molar volume versus mole fraction of ethyl oleate (X_1).

Adiabatic compressibility

The intermolecular association or dissociation leads to structural arrangement of the constituent particles. This structural change of the molecules takes place due to the existence of electrostatic field between interacting molecules that may affect the adiabatic compressibility (β_{ad} ; $\text{kg}^{-1} \text{ms}^2$) which is defined as

$$\beta_{ad} = (1/U^2\rho),$$

where U is the ultrasonic velocity and ρ is the density of the solution.

Internal pressure

On the basis of statistical thermodynamics, Suryanarayana¹⁹ derived an expression for the determination of internal pressure (π) through use of the concept of free volume

$$\pi = bRT (\beta_{ad}\eta/U)^{1/2} (\rho^{2/3}/M_{\text{eff}})^{7/6},$$

where b is packing factor whose value here is 2, R the universal gas constant, T the absolute temperature, β_{ad} the adiabatic compressibility, η the viscosity, ρ the density and M_{eff} is the effective molecular weight.

Table 3. Free volume, acoustic impedance and internal pressure values

Mole fraction (X_1)	Mole fraction (X_2)	Free volume	Acoustic impedance	Internal pressure
303.15 K				
0.0000	1.0000	0.2959	0.9856	797.650
0.0488	0.9511	0.2768	1.0189	747.702
0.1137	0.8862	0.2814	1.0522	672.292
0.2043	0.7956	0.3095	1.0858	578.118
0.3393	0.6606	0.3713	1.1192	470.105
0.5621	0.4378	0.4995	1.1515	352.702
1.0000	0.0000	0.8081	1.1813	230.570
308.15 K				
0.0000	1.0000	0.3497	0.9630	764.052
0.0488	0.9511	0.3252	0.9947	717.378
0.1137	0.8862	0.3293	1.0268	645.901
0.2043	0.7956	0.3611	1.0593	556.014
0.3393	0.6606	0.4321	1.0918	452.611
0.5621	0.4378	0.5799	1.1231	339.919
1.0000	0.0000	0.9361	1.1521	222.467
315.15 K				
0.0000	1.0000	0.4291	0.9449	722.890
0.0488	0.9511	0.3917	0.97604	682.746
0.1137	0.8862	0.3919	1.0078	617.237
0.2043	0.7956	0.4259	1.0402	532.927
0.3393	0.6606	0.5064	1.0725	434.794
0.5621	0.4378	0.6763	1.1037	327.095
1.0000	0.0000	1.0872	1.1329	214.436
318.15 K				
0.0000	1.0000	0.5077	0.9299	692.051
0.0488	0.9511	0.4598	0.9599	655.309
0.1137	0.8862	0.4574	0.9907	593.488
0.2043	0.7956	0.4951	1.0221	513.157
0.3393	0.6606	0.5867	1.0537	419.240
0.5621	0.4378	0.7812	1.0840	315.786
1.0000	0.0000	1.2520	1.1127	207.357

Free length

Jacobson²⁰ introduced the concept of free length in liquids. He suggested the following relation to calculate the intermolecular free length (L_f ; m)

$$L_f = (K/U\rho^{1/2}),$$

where U is the ultrasonic velocity of the liquid, ρ the density of liquid and K is the Jacobson temperature-dependent constant defined as

$$K = (93.875 + 0.345T) \times 10^{-8}.$$

Acoustic impedance

The ultrasonic velocity is influenced by the acoustic impedance (Z), which is given by the relation

$$Z = U\rho$$

where U and ρ are as stated above.

Free volume

The ultrasonic velocity and effective molecular weight are influenced by the free volume (V_f) which is given by the relation

$$V_f = [M_{\text{eff}} U/K\eta]^{3/2}.$$

where M_{eff} , U , K and η are as stated above.

Experiments were conducted to find the density, viscosity and velocity of 2 MHz ultrasonic wave for pure liquids and mixtures of butanol with ethyl oleate by varying the mole fraction and temperature. Table 1 provides the experimental values of density (ρ), viscosity (η) and the velocity (U) of 2 MHz sound wave for pure butanol at different temperatures. The literature values^{10–18} for pure liquids coincide with the experimental values at all temperatures. All the experimental values decreased with increase in temperature for pure butanol. Tables 2–4 provide the experimental, derived and excess values respectively. Figure 1 *a* and *b* shows the FTIR spectra for

Table 4. Excess adiabatic compressibility ($\Delta\beta_{ad}$), excess intermolecular free length (L_f^E), excess impedance (Z^E), excess molar volume (V_m^E), excess free volume (V_f^E) and excess viscosity ($\Delta\eta$) values

X_1	$\Delta\beta_{ad}$	L_f^E	Z^E	V_m^E	V_f^E	$\Delta\eta$
303.15 K						
0.0000	0.0000	0.000	0.0000	0.0000	0.0000	0.0000
0.0488	-0.3172	-0.1138	0.0236	-0.1877	-0.0441	0.3636
0.1137	-0.5697	-0.2069	0.0442	-0.3718	-0.0728	0.6776
0.2043	-0.7429	-0.2731	0.0602	-0.6061	-0.0910	0.9126
0.3393	-0.7962	-0.2961	0.0671	-0.8211	-0.0984	1.0104
0.5621	-0.6376	-0.2398	0.0558	-0.8357	-0.0843	0.8370
1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
308.15K						
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0488	-0.3216	-0.1141	0.0224	-0.1440	-0.0531	0.32532
0.1137	-0.5803	-0.2084	0.0422	-0.3158	-0.0871	0.6061
0.2043	-0.7581	-0.2756	0.0576	-0.5329	-0.1085	0.8163
0.3393	-0.8146	-0.2995	0.0645	-0.7645	-0.1167	0.90363
0.5621	-0.6532	-0.2428	0.0537	-0.8014	-0.0995	0.7483
1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
313.15K						
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0488	-0.3335	-0.1174	0.0219	-0.1009	-0.0695	0.2985
0.1137	-0.6041	-0.2154	0.0415	-0.2623	-0.1122	0.5563
0.2043	-0.7907	-0.2854	0.0568	-0.4825	-0.1378	0.7491
0.3393	-0.8501	-0.3104	0.0638	-0.7174	-0.1462	0.8292
0.5621	-0.6808	-0.2514	0.0530	-0.7271	-0.1230	0.6866
1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
318.15K						
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0488	-0.3339	-0.1171	0.0211	-0.0801	-0.0843	0.2702
0.1137	-0.6052	-0.2147	0.0400	-0.2148	-0.1351	0.5035
0.2043	-0.7933	-0.2849	0.0548	-0.4165	-0.1650	0.6779
0.3393	-0.8550	-0.3107	0.0617	-0.6674	-0.1740	0.7502
0.5621	-0.6848	-0.2516	0.0512	-0.6699	-0.1454	0.6211
1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

pure liquid and mixture respectively. Figure 2 shows the expected hydrogen bonding. Figure 3 *a-l* shows the variation of all the thermo-acoustic quantities with mole fraction of ethyl oleate.

FTIR studies

There is a characteristic absorption at 1737 cm^{-1} , which is attributed to the stretching frequency of the C=O bond of the ester. The absorption band at 3333 cm^{-1} is attributed to the stretching frequency of free O-H bond in alcohol^{21,22}. The band at 2958 cm^{-1} refers to the stretching frequency of C-H bond. The frequency range at 1070 and 1042 cm^{-1} belongs to C-O stretching.

The absorption band at 3365 cm^{-1} is attributed to the stretching frequency of free O-H bond in alcohol. The band at 1737 cm^{-1} attributed to the stretching frequency of C=O bond of the ester.

The hydrogen bond formation between the proton donor O-H of butanol and carbonyl group (C=O) of ethyl oleate using FTIR spectroscopy^{23,24}.

Results and discussions

Velocity of 2 MHz ultrasonic wave in pure liquids, density and viscosity of ethyl oleate and butanol were measured with pre-calibrated interferometer, density bottle and viscosity meter respectively, to the nearest milligram in the temperature range 303.15–318.15 K. The density of ethyl oleate and butanol decreased with increase in temperature. Viscosity and velocity of ultrasonic wave also decreased with increase in temperature. Ultrasonic velocity is increased with increase in mole fraction X_1 of ethyl oleate. Adiabatic compressibility is decreased with increase in mole fraction and increased with increase in temperature at constant mole fraction. Intermolecular free

length also followed the same trend as adiabatic compressibility. Negative excess molar volume represents considerable interactions among the molecules of the binary mixtures²⁵.

Conclusion

The miscible organic compounds ethyl oleate and butanol showed negative values of excess intermolecular free length, deviation in adiabatic compressibility, excess free volume and excess molar volume. This may provide an information about strong interactions in this binary mixture. It is expected that the intermolecular interactions are due to the strong hydrogen bonding. FTIR spectra confirm the hydrogen bond.

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