ACOUSTIC AND SPECTROSCOPIC STUDIES OF BINARY LIQUID MIXTURE OF ETHYL OLEATE WITH CYCLOHEXANONE IN THE TEMPERATURE RANGE OF 303.15K TO 318.15K

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Ultrasonic velocity, viscosity and density of Ethyl Oleate with Cyclohexanone have been determined at various temperatures in the range of 303.15K to 318.15K. The data is used to calculate the quantities like adiabatic compressibility, intermolecular free length and acoustic impedance along with their excess values and molar volume. The observed variations of these parameters with concentration and temperature are discussed in terms of the intermolecular interactions among the unlike molecules of the binary mixtures.

KEYWORDS: Ethyl Oleate, Cyclohexanone**,** ultrasonic velocity, viscosity, density, FTIR.

INTRODUCTION

Lagemann and Dunbar [1] were the first to point out the sound velocity method for qualitative determination of the degree of association in molecules of liquids. Intermolecular interactions play a vital role in liquid mixtures. They effect the arrangement, coordination and conformations of the molecules in solutions. Recent improvements have found the ultrasonic energy is very useful in medicine, engineering, agriculture and industry [2, 3] in understanding these interactions. Thermodynamic data is essential for many industrial applications such as in the oil and gas industries for flow assurance and oil recovery, in chemical industries for the design and separation processes, in pharmaceutical and polymer industries for solvent selection and emission control and recently in biotechnology for aggregation of proteins and several protein separations. Spectroscopic and acoustic properties of binary liquid mixtures containing polar and self-associated components exhibit significant deviations from ideality due to difference in size of molecules and possible interacting bonds between like and unlike molecules. In extension to the previous works, the research results of ultrasonic and spectroscopic properties of binary mixture of Ethyl Oleate with Cyclohexanone are presented. From the experimental values of ultrasonic velocity, viscosity and density the quantities like adiabatic compressibility, intermolecular free length, acoustic impedance and also their excess values with excess molar volume have been derived for interpretation of the molecular

interactions. Further, the infrared spectra of Ethyl Oleate with Cyclohexanone have also been presented for confirmation of the expected interactions.

MATERIALS AND EXPERIMENTS

All the materials procured of Sigma-Aldrich AR grade and glassware used of Borosilicate make. Organic liquid compounds Ethyl Oleate ($C_{20}H_{38}O_2$, 310.51g/mol) and Cyclohexanone ($C_6H_{10}O$, 98.15 g/mol) were procured and used directly without purification. The densities and viscosities of the liquid compounds were measured with specific gravity bottle and Ostwald viscometer respectively those are pre calibrated with 3D [4] water of Millipore to nearest unit. The time taken for flow of viscous fluid in Ostwald viscosity meter is measured with an accuracy of 0.01 sec. Borosilicate glassware, Japan make Shimadzu electronic balance of sensitivity +/–0.001g and constant temperature water bath of accuracy +/-0.1K were used while conducting the experiments. 2MHz ultrasonic interferometer model no. F-05 with least count of micrometer 0.001mm of Mittal Enterprises [5] was used for calculating velocities of stationary sound waves formed and all the tests were conducted as per ASTM standard [6] procedures. FTIR spectra were obtained with a Bruker ALPHA FT-IR spectrometer. Table 1 and table 2 represent the density, viscosity and velocity of ultrasonic wave of experimental as well as literature values for pure liquids of Ethyl Oleate and Cyclohexanone respectively. Table 3, Table 4 and Table 5 present the experimental, derived and excess values for the mixture of two liquids at the study temperatures as in the formulae given by the equations from 1 to 9.

Table 1. Comparison of experimental and literature values of density (ρ), viscosity (η) and velocity (U) of 2MHz ultrasonic wave of pure Ethyl Oleate

Parameter	303.15K		308.15K		313.15K		318.15K	
	Expt.	**Lite.**	**Expt.**	**Lite.**	**Expt.**	**Lite.**	**Expt.**	**Lite.**
Density**(ρ) kg/m^3**	**863.50**	863.20[4]	**859.34**	859.50[4]	**855.62**	855.80[4]	**852.04**	855.20[4]
Viscosity (η) Ns/m^2	**5.3101**	5.3094[4]	**4.7164**	4.7156[4]	**4.2163**	4.7300[3] 4.2137[4]	**3.7820**	3.7876[4]
Velocity (U) m/s	1368.16	1360.67[9]	1340.78	1342.98[9]	1324.00	1325.49[9]	1305.09	1308.17[9]

Table 2. Comparison of experimental and literature values of density (ρ), viscosity (η) and velocity (U) of 2MHz ultrasonic wave for pure Cyclohexanone.

Parameter	303.15K		308.15K		313.15K		318.15K	
	Expt.	**Lite.**	**Expt.**	**Lite.**	**Expt.**	**Lite.**	**Expt.**	**Lite.**
Density***(ρ)*** **kg/m^3**	949.8	936.50[7] 937.24[9] 944.40[10]	939.7	930.60[7] 931.20[8] 932.67[9] 939.60[10]	930.4	926.60[7] 927.27[9] 934.70[10]	921.9	923.4[8] 922.5[10]

Viscosity*(η)* Ns/m^2	1.7369	1.7372[7] 1.8470[9] 1.7572[10]	**1.6572**	1.6191[7] 1.6562[8] 1.6940[9] 1.6013[10]	1.5487	1.4308[7] 1.5460[9] 1.4561[10]	1.3811	1.3809[8] 1.3101[10]
Velocity*(U)* m/s	1409.5	1407.7[7] 1408.0[10]	1369.6	1397.4[7] 1358.0[8] 1362.0[10]	1347.2	1393.8[7] 1349.0[10]	1330.9	1275[8] 1372[10]

Table 3. Ultrasonic velocity *(U)*, Density *(ρ)*, Viscosity *(η)*, adiabatic compressibility *(β_{ad})*, inter molecular free length *(L_f)*, molar volume *(V_m)*, Rao's constant(R), Wada's constant(W) for binary mixture of Ethyl Oleate and Cylohexenone at different temperatures.

Mole fraction (X_1)	Velocity m/sec (U)	Density kg/m^3 (ρ)	Viscosity Nsm^{-2} (η)	Ad. Comp. 10^{-10} N^{-1}.m^2 (β_{ad})	Int. Mol. Free length 10^{-10} m (L_f)	Mol. Vol. (V_m)	Rao's Constnt (R)	Wada's Constant (W)
***T* = 303.15 K**								
0.0000	1409.5	949.8	1.7369	5.2995	4.7768	103.34	5.3779	8.1432
0.0543	1402.6	933.2	2.3319	5.447	4.8428	117.54	6.1072	9.2265
0.1256	1395.7	918.2	2.9269	5.5909	4.9063	135.95	7.0520	10.632
0.2232	1388.8	905.6	3.5219	5.7251	4.9649	160.73	8.3235	12.527
0.3650	1381.9	895.4	4.1169	5.8483	5.0180	196.18	10.142	15.243
0.5896	1375.0	881.1	4.7119	6.003	5.0840	253.51	13.085	19.625
1.0000	1368.1	863.5	5.3069	6.1873	5.1614	359.61	18.530	27.718
***T* = 313.15 K**								
0.0000	1369.6	939.7	1.6572	5.6731	4.9899	104.45	5.3839	8.1510
0.0543	1364.8	925.4	2.167	5.8014	5.0460	118.60	6.1064	9.2262
0.1256	1360.0	911.2	2.6768	5.9335	5.1031	137.14	7.0527	10.634
0.2232	1355.2	899.4	3.1866	6.054	5.1547	162.07	8.3249	12.531
0.3650	1350.4	890.1	3.6964	6.1608	5.2000	197.67	10.141	15.245
0.5896	1345.6	876.2	4.2062	6.3033	5.2598	255.27	13.081	19.624
1.0000	1340.8	859.3	4.7160	6.4733	5.3303	361.36	18.496	27.674
***T* = 308.15 K**								
0.0000	1347.2	930.4	1.5487	5.9220	5.1469	105.49	5.4079	8.1822
0.0543	1343.3	918.12	1.9947	6.0356	5.1960	119.61	6.1259	9.2521
0.1256	1339.5	905.2	2.4407	6.1570	5.2480	138.20	7.0711	10.66
0.2232	1335.6	894.8	2.8867	6.2645	5.2936	163.14	8.3392	12.552

0.3650	1331.8	885.9	3.3327	6.3641	5.3355	198.92	10.158	15.271
0.5896	1327.9	872.2	3.7787	6.5016	5.3929	256.77	13.1	19.652
1.0000	1324.1	855.6	4.2247	6.6663	5.4608	362.93	18.498	27.677
***T* = 318.15 K**								
0.0000	1330.9	921.9	1.3811	6.1239	5.2834	106.46	5.4357	8.2182
0.0543	1326.6	910.9	1.7813	6.2381	5.3324	120.62	6.1518	9.2863
0.1256	1322.3	899.6	2.1815	6.3576	5.3832	139.19	7.0913	10.687
0.2232	1318.0	890.2	2.5817	6.4667	5.4292	164.19	8.356	12.576
0.3650	1313.7	881.9	2.9819	6.5703	5.4726	200.11	10.172	15.292
0.5896	1309.4	868.5	3.3821	6.7156	5.5327	258.17	13.110	19.667
1.0000	1305.1	852.1	3.7823	6.8900	5.6041	364.42	18.485	27.662

Table 4. Free volume (V_f), acoustic impedance (Z), internal pressure(π), Gibb's energy (G^E), enthalpy(H) and relaxation time(τ) for binary mixture of Ethyl Oleate and Cylohexenone at different temperatures.

Mole fraction (X_1)	Mole fraction (X_2)	Free Volume (V_f)	Acoustic Impedance (Z)	Internal pressure (n)	Gibb's Energy (G^E)	Enthalpy (H)	Relaxation time (τ)
***T* = 303.15 K**							
0.0000	1.0000	0.8025	1.3387	530.71	0.1102	54.842	1.2273
0.0543	0.9457	0.605	1.3089	535.12	0.1114	62.901	1.6936
0.1256	0.8744	0.5185	1.2815	511.31	0.1124	69.512	2.1819
0.2232	0.7768	0.4909	1.2577	465.71	0.1132	74.852	2.6884
0.3650	0.6350	0.5111	1.2374	402.31	0.1138	78.925	3.2102
0.5896	0.4104	0.5941	1.2115	322.52	0.1144	81.763	3.7714
1.0000	0.0000	0.8086	1.1814	230.53	0.115	82.898	4.3781
***T* = 308.15 K**							
0.0000	1.0000	0.8248	1.2870	530.77	0.1122	55.438	1.2535
0.0543	0.9457	0.6488	1.2630	528.25	0.1133	62.653	1.6762
0.1256	0.8744	0.5712	1.2392	500.32	0.1142	68.616	2.1177
0.2232	0.7768	0.5511	1.2189	452.98	0.1149	73.416	2.5722
0.3650	0.6350	0.5818	1.2020	389.71	0.1155	77.033	3.0364
0.5896	0.4104	0.6833	1.1790	311.47	0.1161	79.508	3.5350
1.0000	0.0000	0.9365	1.1521	222.41	0.1167	80.371	4.0704

T = 313.15 K							
0.0000	1.0000	0.8907	1.2534	522.26	0.1140	55.095	1.2228
0.0543	0.9457	0.7181	1.2334	516.07	0.1150	61.728	1.6052
0.1256	0.8744	0.6422	1.2125	486.45	0.1159	67.227	2.0037
0.2232	0.7768	0.6267	1.1951	439.09	0.1166	71.633	2.4112
0.3650	0.6350	0.6672	1.1798	376.77	0.1172	74.947	2.8280
0.5896	0.4104	0.7883	1.1582	300.62	0.1178	77.190	3.2757
1.0000	0.0000	1.0839	1.1329	214.65	0.1183	77.902	3.7551
T = 318.15 K							
0.0000	1.0000	1.0385	1.2270	501.05	0.1155	53.345	1.1277
0.0543	0.9457	0.8357	1.2084	495.68	0.1166	59.789	1.4816
0.1256	0.8744	0.7465	1.1895	467.80	0.1175	65.115	1.8492
0.2232	0.7768	0.7277	1.1733	422.60	0.1182	69.388	2.2260
0.3650	0.6350	0.7739	1.1586	362.87	0.1188	72.612	2.6123
0.5896	0.4104	0.9131	1.1372	289.77	0.1194	74.808	3.0284
1.0000	0.0000	1.2521	1.1121	207.27	0.1200	75.533	3.4747

Table 5. Excess velocity, excess adiabatic compressibility ($\Delta\beta_{ad}$), excess inter molecular free length (L_f^E), excess impedance (Z^E), excess molar volume (V_m^E), excess free volume, excess viscosity ($\Delta\eta$) for binary mixture of Ethyl Oleate and Cylohexenone at different temperatures.

(X_1)	U^E	$\Delta\beta_{ad}$	L_f^E	Z^E	V_m^E	V_f^E	$\Delta\eta$
T = 303.15 K							
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0543	-4.6500	0.0992	0.0451	-0.0213	0.2785	-0.1978	0.401
0.1256	-8.5989	0.1798	0.0812	-0.0374	0.4182	-0.2848	0.7415
0.2232	-11.459	0.2274	0.1022	-0.0459	0.1861	-0.3129	0.9881
0.3650	-12.490	0.2248	0.1009	-0.0439	-0.6889	-0.2936	1.0771
0.5896	-10.089	0.1800	0.0804	-0.0344	-0.9289	-0.2120	0.8700
1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
T = 308.15 K							
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0543	-3.2261	0.0845	0.0375	-0.0167	0.1152	-0.1821	0.3426
0.1256	-5.9634	0.1593	0.0702	-0.0308	0.2554	-0.2677	0.6334

0.2232	-7.9422	0.2014	0.0885	-0.0379	0.0180	-0.2988	0.8435
0.3650	-8.6501	0.1945	0.0854	-0.0356	-0.8920	-0.2839	0.9187
0.5896	-6.9782	0.1572	0.0687	-0.0283	-1.0240	-0.2075	0.7411
1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
T = 313.15 K							
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0543	-2.5809	0.0728	0.0319	-0.0135	-0.0243	-0.1832	0.2990
0.1256	-4.7688	0.1406	0.0613	-0.0256	0.0407	-0.2729	0.5524
0.2232	-6.3477	0.1749	0.0761	-0.0311	-0.3288	-0.3075	0.7353
0.3650	-6.9078	0.1685	0.0733	-0.0293	-1.2124	-0.2945	0.8002
0.5896	-5.5657	0.1387	0.0601	-0.0238	-1.2226	-0.2169	0.6448
1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
T = 318.15 K							
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.0543	-2.8757	0.0719	0.0313	-0.0122	-0.0839	-0.2146	0.2676
0.1256	-5.3116	0.136	0.059	-0.0228	-0.1497	-0.3192	0.4943
0.2232	-7.0667	0.1696	0.0733	-0.0277	-0.5924	-0.3591	0.6577
0.3650	-7.6848	0.1639	0.0709	-0.026	-1.4935	-0.3433	0.7152
0.5896	-6.1846	0.1369	0.059	-0.0215	-1.4247	-0.2522	0.5756
1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

THEORY AND CALCULATIONS

In order to examine the inter molecular interactions in liquid mixtures of Ethyl Oleate with Cyclohexanone, experiments were conducted to find the density, viscosity and velocity of 2MHz ultrasonic waves for pure liquids and for binary liquid mixtures. The results of pure liquids are compared with 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 the inter and intra molecular interactions at each temperature. The derived and excess values are calculated by using the fallowing relations.

Adiabatic compressibility (β_{ad})

Adiabatic compressibility the parameter which represents the ability to change volume of a liquid sample is

$$\beta_{ad} = (\rho\, U^2)^{-1} \qquad \ldots(1)$$

Intermolecular free length (L_f)

The formula for outer to outer distance between the interacting molecules

$$L_f = K\sqrt{\beta_{ad}} \quad \ldots(2)$$

Molar volume of the binary liquid mixture (V_m)

The molar volume of the system at every mole fraction for the mixture is given by

$$V_m = M_{eff}/\rho_{mix} \quad \text{where} \quad M_{eff} = M_1 X_1 + M_2 X_2 /(X_1 + X_2) \quad \ldots(3)$$

Free volume (V_f)

The free volumes of the binary mixtures have been computed using its relationship with the ultrasonic velocity and viscosity as given below

$$V_f = \left(\frac{MU}{K\eta}\right)^{\frac{3}{2}} \quad \ldots(4)$$

where k is a constant, which is independent of temperature and its value is 4.28×10^9 for all liquids.

Specific acoustic impedance (Z)

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

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

Excess thermodynamic parameters

With the help of excess parameters the extent of deviation from the ideal behavior of binary mixture can be estimated. The difference between the thermodynamic function of mixing for a real system and the value corresponding to a perfect solution at the same temperature, pressure and composition is called the thermodynamic excess function, denoted by Y^E.

Excess value Y^E for each parameter can compute by using the general formula

$$Y^E = Y - (Y_1 X_1 + Y_2 X_2) \quad \ldots(6)$$

where Y is the parameter under consideration, X_1 and X_2 are mole fractions of two liquids Ethyl Oleate and other organic compound under consideration respectively of the binary system, Cyclohexonone here and E represent 'excess'.

Deviation in adiabatic compressibility ($\Delta\beta_{ad}$)

The difference of the adiabatic compressibility of the mixture and the sum of the fractional contributory adiabatic compressibilities of the two liquids individually is the deviation in adiabatic compressibility. At a given mole fraction it is given by

$$\Delta\beta_{ad} = \beta_{ad} - (\beta_{ad1} X_1 + \beta_{ad2} X_2) \quad \ldots(7)$$

Excess free length (L_f^E)

The excess free length can be calculated with formula

$$L_f^E = L_f - (L_{f1} X_1 + L_{f2} X_2) \quad \ldots(8)$$

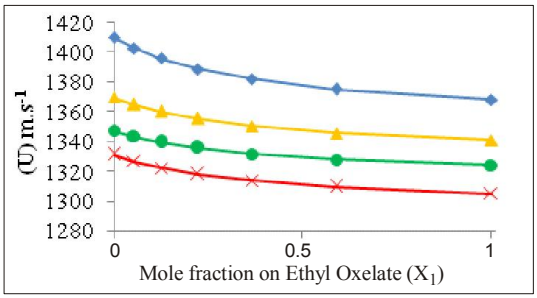
Excess acoustic impudence (Z^E)

Excess acoustic impedance can be calculated by the relation

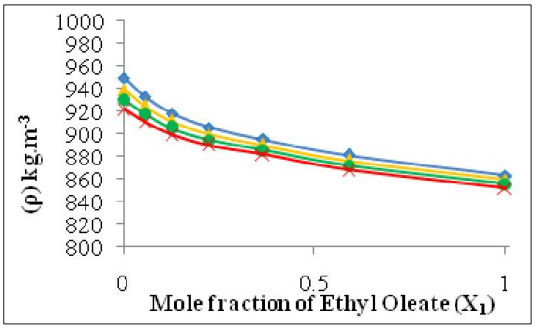
$$Z^E = Z - (Z_1 X_1 + Z_2 X_2) \quad \ldots(9)$$

Results and discussion

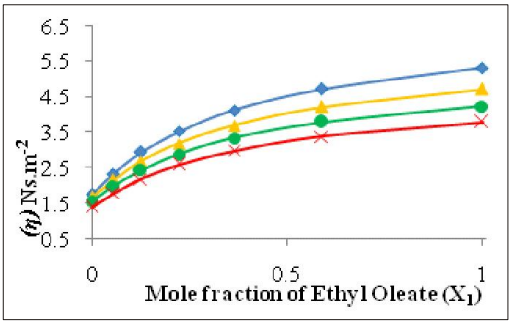
In order to examine the inter molecular interactions in liquid mixtures of Ethyl Oleate with Cyclohexanone, experiments were conducted to find the density, viscosity and velocity of 2MHz ultrasonic waves for pure liquids and for liquid mixtures. The results of pure liquids are compared with literature values. The experimental values are coinciding with the values from the previous studies. The derived, excess values were calculated at various mole fractions of Ethyl Oleate for understanding the inter and intra molecular interactions at each temperature. Graphs were drawn for variation of the experimental and derived quantities with mole fraction of Ethyl Oleate at all the study temperatures as shown in graph 1 to graph 12. From the above studies it can be concluded that there exists a dipole-dipole interactions among the unlike molecules.



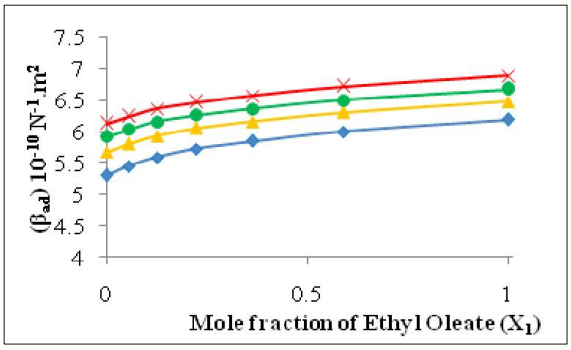
Graph 1. Ultrasonic Velocity Vs Mole fraction of Ethyl Oleate (X_1)



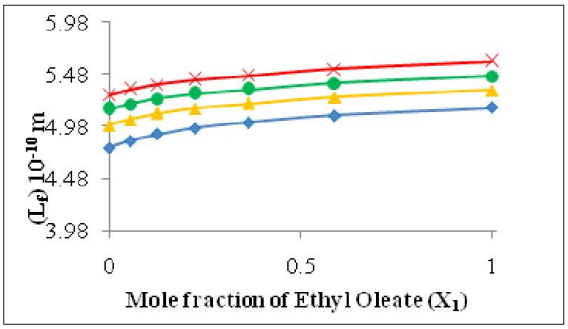
Graph 2. DensityVs Mole fraction of Ethyl Oleate (X_1)



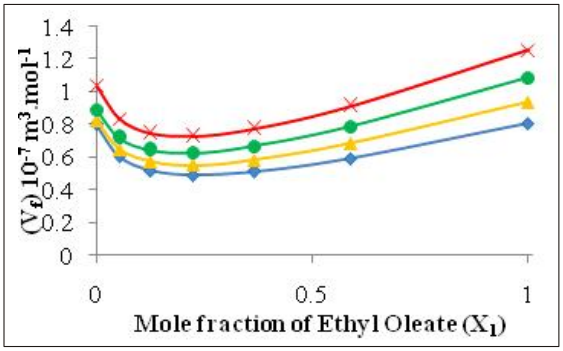
Graph 3. ViscocityVs Mole fraction of Ethyl Oleate (x_1)



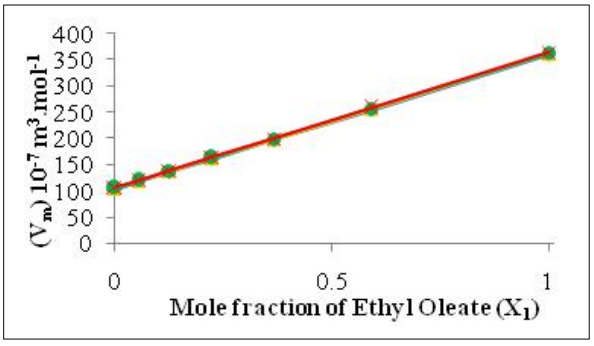
Graph 4. Adiabatic Compressibility Vs Mole fraction of Ethyl Oleate (X_1)



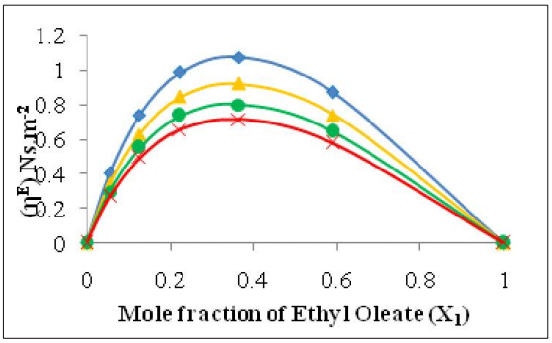
Graph 5. Intermolecular Free Length Vs Mole fraction of Ethyl Oleate (X_1)



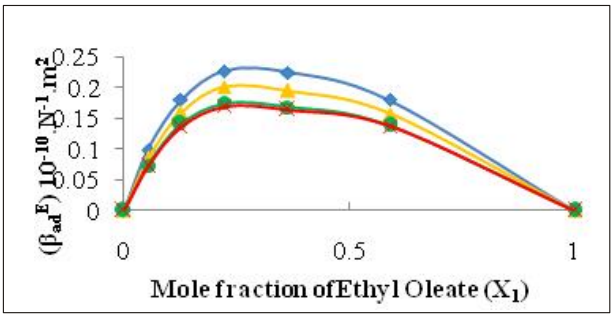
Graph 6. Free volume Vs Mole fraction of Ethyl Oleate (X_1)



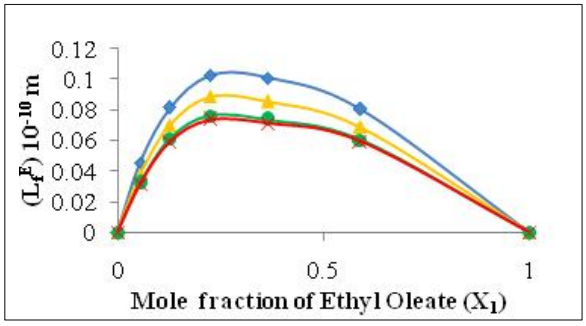
Graph 7. Molar Volume Vs Mole fraction of Ethyl Oleate (X_1)



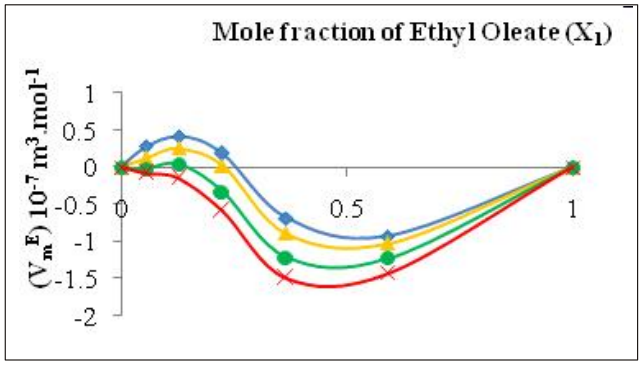
Graph 8. Deviation in Viscosity Vs Mole fraction of Ethyl Oleate (X_1)



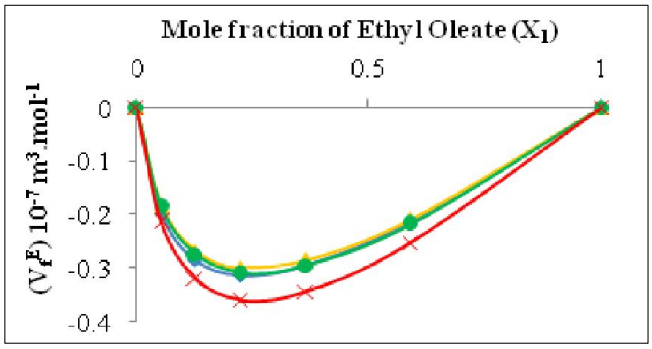
Graph 9. Excess adia. Compressibility Vs Mole fraction of Ethyl Oleate (X_1)



Graph 10. Excess Intermolecular Free Length Vs Mole fraction of Ethyl Oleate (X_1)



Graph 11. Excess Molar Volume Vs Mole fraction of Ethyl Oleate (X_1)



Graph 12. Excessmolar volume Vs Mole fraction of Ethyl Oleate (X_1)

In the above system the experimental values are changing gradually from values of selected carbonyl compounds Cyclohexanone at X_1 is 0.0000 to the values of Ethyl Oleate at X_1 is 1.0000. Except acoustic impedance, internal pressure, enthalpy and relaxation time all the parameters are increasing with the increase in temperature whereas all the parameters except acoustic impedance and internal pressure remaining all the parameters are increasing with the increase in mole fraction of Ethyl Oleate. The adiabatic compressibility, intermolecular free length, Gibbs free energy and enthalpy are increasing with increase in the mole fraction of Ethyl Oleate. Rao's constant and Wada's constant as a function of mole fraction of Ethyl Oleate are represented in the graphs. From the figures, it is observed that Rao's constant and Wada's constant increase with increase in the mole fraction of Ethyl Oleate and is almost independent of temperature at particular mole fraction of Ethyl Oleate.

The excess quantities excess velocity (U^E), excess adiabatic compressibility($\Delta\beta_{ad}$), excess inter molecular free length (L_f^E), excess impedance (Z^E), excess molar volume(V_m^E), excess free volume (V_f^E), excess viscosity ($\Delta\eta$) and excess internal pressure (Π^E) were calculated for Cyclohexanone and presented as in the relevant tables. FT-IR figures were shown for binary mixture system. The variation of excess parameters with mole fraction of Ethyl Oleate X_1 is shown in graphs.

The excess quantities excess molar volume, excess free volumes are negative and excess viscosity, excess internal pressures are positive. The negative values of excess molar volume represent the strong interactions [11-19] for the present studies between Ethyl Oleate and Cyclohexanone which is supported by the positive values of deviation in viscosity and internal pressure. The values of excess molar volume are changing from positive to negative with increase in mole fraction of Ethyl Oleate (X_1) in case liquid mixture of Cyclohexanone which yields that there exists a strong dipole-dipole interactions due to the increase in concentration of the common compound Ethyl Oleate.

FT IR 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 band at 2935cm^{-1} referred to the stretching frequencies of aliphatic C-H bond. The strong band at 1703 cm^{-1} confirmed the stretching frequency of C=O group in the molecule. Fig. 1 and fig. 2 shows the peaks of IR values for pure liquids and liquid mixture respectively.

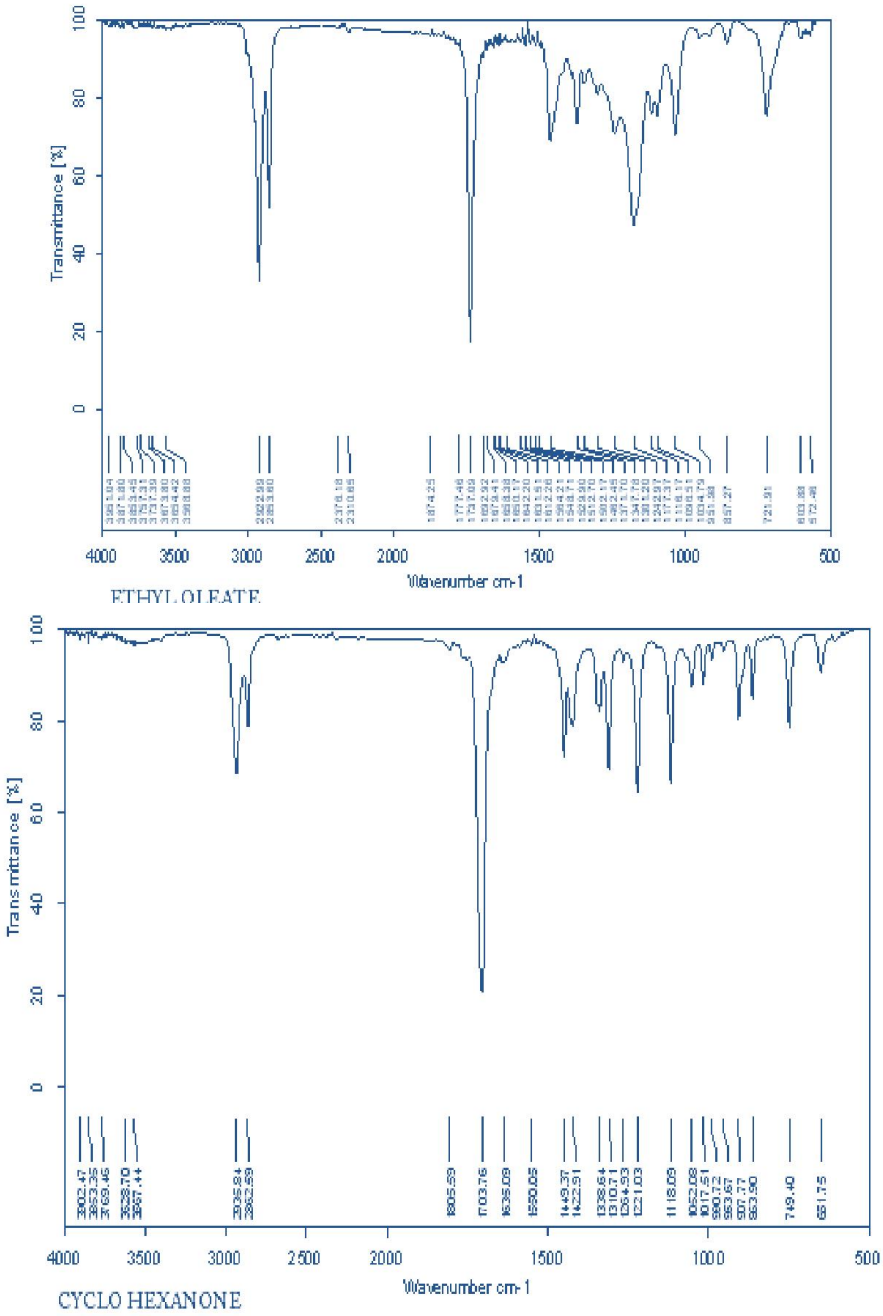


Fig. 1. FTIR spectrum of pure Ethyl Oleate and pure Cyclohexanone

The absorption band at 1735cm^{-1}, which is attributed to the stretching frequency of the C=O bond of the ester. The band at 2925cm^{-1} referred to the stretching frequencies of C-H bond. The strong band at 1714cm^{-1}confirmed the stretching frequency of C=O group in the carbonyl compound [11].

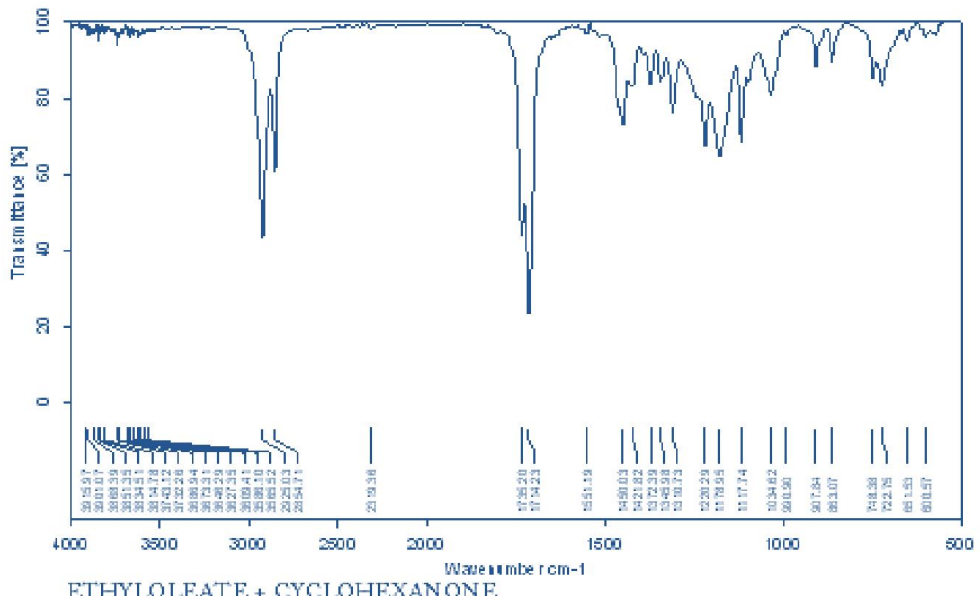


Fig. 2. FTIR spectrum of Ethyl Oleate + Cyclohexanone

Conclusions

The miscible organic binary liquid mixture of Ethyl Oleate and Cyclohexanone are showing negative values of Excess molar volume (VmE) and Excess free volume (V_f^E) this may give an information about the considerable interactions among the molecules of the between this binary mixture. So we concluded that interactions are may be due to dipole-dipole interactions. FTIR spectra also confirms the same.

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