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Thermophysical Properties of Acetophenone with Ethylchloroacetate at Temperatures of 303.15, 313.15 and 323.15 K

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ABSTRACT. Densities, viscosities, refractive indices and speed of sounds of the binary mixtures of Acetophenone with Ethylchloroacetate were measured over the entire mole fractions at (303.15, 313.15 and 323.15) K. From these experimental results, excess molar volume V^E, viscosity deviation $\Delta\eta$, refractive index deviation Δn_D, deviations in speed of sound Δu, deviations in isentropic compressibility Δk_s and excess intermolecular free length ΔL_f were calculated. The viscosity data have been correlated with the equations of Grunberg and Nissan, Hind et al., Tamura and Kurata, Katti and Chaudri, Sedgwick, Krishnan-Laddha and McAllister. The thermo physical properties under study were fit to the Jouyban-Acree model. The excess values were correlated using Redlich-Kister polynomial equation to obtain their coefficients and standard deviations. It was found that in all cases, the data obtained fitted with the values correlated by the corresponding models very well. The results are interpreted in terms of molecular interactions occurring in the solution.

Key words: Viscosity, Density, Refractive index, Ultrasonic velocity, Molecular interactions

INTRODUCTION

The thermodynamic and transport properties of liquids and liquid mixtures[1] are used to study the molecular interactions between the various components of the mixture and also to understand engineering applications concerning heat transfer, mass transfer, and fluid flow. In chemical process industries, materials are normally handled in fluid form, and as a consequence, the physical, chemical, and transport properties of fluids assume importance. Thus, data on some of the properties associated with the liquids and liquid mixtures like density, viscosity, refractive index and ultrasonic velocity find extensive application in solution theory and molecular dynamics.[2] Such results are necessary for interpretation of data obtained from thermo chemical, electrochemical, biochemical and kinetic studies.[3] Acetophenone is an important industrial chemical widely used as an ingredient of flavor and fragrance in soaps, detergents, cosmetics and perfumes. Ethylchloroacetate are used as a solvent in preparation of drugs, perfumes, cosmetics and pesticides. The acetophenone and ethylchloroacetate mixture is used as cosmetics, intermediates of drugs, perfumes, and pesticides. In our earlier paper, we had studied the transport properties of binary liquid mixtures.[4,5] In continuation of this research, we have reported density (ρ), viscosity (η), refractive index (n_D) and ultrasonic velocity (u) of pure acetophenone and ethylchloroacetate as well as for the binary system constituted by these two chemicals at temperatures of 303.15, 313.15 and 323.15 K. The viscosity values have been fitted to Grunberg and Nissan,[6] Hind et al.,[7] Tamura and Kurata,[8] Katti and Chaudhari,[9] Sedgwick,[10] McAllister[11] and Krishnan and Laddha model.[12] The Jouyban-Acree model[13] has also been extended to density, viscosity, refractive index and ultrasonic velocity of binary mixture. The deviation values have been fitted to Redlich-Kister type[14] equation. Literature survey showed that no measurements have been previously reported for the mixture studied in this paper.

EXPERIMENTAL SECTION

Materials

Acetophenone and ethylchloroacetate (Lobo Chemicals, India, and Mass fraction 0.998) were purified by using the methods described in the literature[15,16] and only middle fractions were used in the experiment. Binary mixtures are prepared by mixing appropriate volumes of the

***Table* 1.** Comparison of experimental and literature values of acetophenone and ethylchloroacetate at 303.15 K

Pure liquids	ρ/gcm^{-3}		η/(mPa·s)		n_D		u/ms^{-1}	
	lit	Exp	lit	Exp	lit	Exp	lit	Exp
Acetophenone	1.01943[18] 1.0199[19] 1.01944[20]	1.0196	1.513[18] 1.518[19]	1.5141	1.5221[28]	1.5221	1460[18]	1460
Ethyl chloroacetate	1.13929[21] 1.13929[22]	1.1395	0.969[21,22,25]	0.9691	1.4173[25]	1.4173	1229[21,22]	1228.3

liquid components in the specially designed glass bottles with air tight Teflon coated caps and mass measurements performed on a Shimadzu Corporation Japan type BL 2205 electronic balance, with a precision of ±0.01 mg. The required properties are measured on the same day immediately after preparing each composition. The uncertainty of the mole fraction is ±0.0001. The experimental values of density, viscosity, refractive indices and ultrasonic velocity for the pure liquids are compared, with the available literature values at T=303.15 K in *Table* 1.

Density

A double-arm pycnometer with a bulb of 25 cm^3 and a capillary of an internal diameter of about 1 mm is used to measure the densities (ρ) of pure liquids and binary mixtures. The pycnometer is calibrated by using conductivity water (having specific conductance less than 1×10^{-6} ohm^{-1}) with 0.9970 and 0.9940 g·cm^{-3} as its densities at T=(298.15 and 308.15) K, respectively. The pycnometer filled with air bubbles free liquids is kept in a thermostat with a thermal stability of ±0.01 K for over 30 min to attain thermal equilibrium. The precision of the density measurements was estimated to be ±0.0002 g·cm$^{-3.}$

Kinematic Viscosity

The kinematic viscosity of pure liquids and liquid mixtures were measured using Ostwald viscometer previously calibrated using water.[4,5] The time was measured with a precision of 0.01 s, and the uncertainty in the viscosity was estimated to be less than 0.0002 mPa·s. The kinematic viscosity was obtained from the working equation

$$\nu = at - b/t \quad (1)$$

where the two constants a and b were obtained by measuring the flow time t of benzene.

Refractive Index

Refractive indices were measured using thermostatically controlled Abbe refractometer with accuracy less than 0.0001 units. Water was circulated into the prism of the refractometer by a circulation pump connected to an external thermo stated water bath. Calibration was performed by measuring the refractive indices of doubly distilled water and propyl alcohol at defined temperatures. The sample mixture was directly injected in to the prism assembly of the instrument using a syringe. The solutions were pre thermo stated at the temperature of the experience before the experiments to achieve a quick thermal equilibrium.

Ultrasonic Velocity

The Speed of sound was measured by using a variable path, single crystal interferometer. (Mittal Enterprises, New Delhi) operating at a frequency of 2MHz, which was calibrated with water and benzene. The interferometer cell was filled with the test liquid, and the temperature of the solution was maintained constant within ±0.01 K by circulation of water from a thermostatically regulated water bath through the water jacketed cell. The uncertainty was estimated to be 0.1 ms^{-1}. The isentropic compressibility was calculated by the equation

$$k_s = 1/\rho u^2 \quad (2)$$

where ρ is the density of the mixture and u is the ultrasonic velocity of the mixture. The intermolecular free length (L_f) was calculated by the equation

$$L_f = K\times k_s^{1/2} \quad (3)$$

where K = ((91.368 + 0.3565T)×10^{-8}) is temperature dependent Jacobson's constant.

RESULTS AND DISCUSSION

The experimental values of densities (ρ), viscosities (η), refractive indices (n_D) and ultra sonic velocities (u) of acetophenone with ethylchloroacetate mixtures at various temperatures are listed in *Table* 2. Excess molar volumes V^E are calculated from the measured densities (ρ) by using equation

***Table* 2.** Densities ρ, viscosities η, refractive indices n_D and ultrasonic velocity u for the Acetophenone (1) + Ethylchloroacetate (2) mixture at T = (303.15, 313.15, and 323.15) K

x_1	ρ/gcm^{-3}	η/(mPa·s)	n_D	u/ms^{-1}
		303.15 K		
0.0000	1.1395	0.9691	1.4173	1228.3
0.0830	1.1284	1.0167	1.4264	1249.4
0.1674	1.1175	1.0654	1.4357	1270.4
0.2534	1.1066	1.1145	1.4452	1291.5
0.3409	1.0957	1.1640	1.4547	1312.6
0.4300	1.0848	1.2137	1.4642	1333.6
0.5206	1.0740	1.2630	1.4737	1354.7
0.6130	1.0631	1.3123	1.4833	1375.7
0.7070	1.0522	1.3621	1.4929	1396.8
0.8029	1.0413	1.4123	1.5024	1417.8
0.9005	1.0305	1.4629	1.5121	1438.9
1.0000	1.0196	1.5141	1.5221	1460.0
		313.15 K		
0.0000	1.1259	0.8342	1.4119	1197.6
0.0830	1.1155	0.8781	1.4212	1219.4
0.1674	1.1050	0.9221	1.4307	1241.2
0.2534	1.0946	0.9660	1.4402	1263.0
0.3409	1.0842	1.0099	1.4497	1284.8
0.4300	1.0738	1.0538	1.4593	1306.6
0.5206	1.0634	1.0978	1.4689	1328.4
0.6130	1.0529	1.1417	1.4785	1350.2
0.7070	1.0425	1.1857	1.4881	1372.0
0.8029	1.0320	1.2296	1.4977	1393.8
0.9005	1.0216	1.2736	1.5074	1415.6
1.0000	1.0112	1.3175	1.5172	1438.0
		323.15 K		
0.0000	1.1125	0.7009	1.4065	1168.8
0.0830	1.1021	0.7540	1.4156	1192.4
0.1674	1.0918	0.8072	1.4246	1215.9
0.2534	1.0814	0.8603	1.4337	1239.5
0.3409	1.0711	0.9135	1.4427	1263.1
0.4300	1.0608	0.9666	1.4518	1286.6
0.5206	1.0504	1.0198	1.4608	1310.2
0.6130	1.0401	1.0729	1.4699	1333.7
0.7070	1.0297	1.1261	1.4789	1357.3
0.8029	1.0194	1.1792	1.4880	1380.9
0.9005	1.0090	1.2324	1.4970	1404.4
1.0000	0.9987	1.2855	1.5061	1428.0

$$V^E = (x_1M_1 + x_2M_2)/\rho_m - (x_1M_1/\rho_1 + x_2M_2/\rho_2) \quad (4)$$

where ρ_m is the density of the mixture and M_1, ρ_1, x_1 and M_2, ρ_2, x_2 are molecular mass, density and mole fractions of pure acetophenone (1) and ethylchloroacetate (2), respectively. The uncertainty in the measurement of V^E was found to be ±0.0003 cm^3 mol^{-1}. The deviations in viscosities, Δη, are computed using the relationship

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \quad (5)$$

where η is the dynamic viscosity of the mixture and η_1, x_1 and η_2, x_2 are the viscosity and mole fractions of pure acetophenone (1) and ethylchloroacetate (2), respectively. The uncertainty in the measurement of Δη was found to be ±0.0001. The deviations of refractive index (Δn_D) over the entire mole fraction range are obtained by

$$\Delta n_D = n_D - (x_1n_{D1} + x_2n_{D2}) \quad (6)$$

The isentropic compressibility deviation (Δk_s) over the entire composition range are obtained by

$$\Delta k_s = k_s - (x_1k_{s1} + x_2k_{s2}) \quad (7)$$

k_{s1}, k_{s2}, and k_s refer to the isentropic compressibility of components 1 and 2 and the mixture, respectively.

The change of intermolecular free length (ΔL_f) on mixing are calculated by the equation

$$\Delta L_f = L_f - (x_1L_{f1} + x_2L_{f2}) \quad (8)$$

where L_{f1} and L_{f2} refer to the intermolecular free length of component 1 and 2.

The excess molar volumes, deviations of viscosity, deviations of refractive index, isentropic compressibility deviation and change of intermolecular free length were fitted to a Redlich-Kister[14] equation of the type

$$Y = x_1x_2 \Sigma A_i(x_1 - x_2)^i \quad (9)$$

where Y is either V^E, Δη, Δn_D, Δu, Δk_s or ΔL_f and n is the degree of polynomial. Coefficients Ai were obtained by fitting equation (9) to experimental results using a least-squares regression method. In each case, the optimum number of coefficients was ascertained from an examination of the variation in standard deviation (S). S was calculated using the relation

$$S(Y) = [\Sigma(A_{exp} - A_{cal})^2/(N - n)]^{1/2} \quad (10)$$

where N is the number of data points and n is the number of coefficients. The calculated values of coefficients along with the standard deviation (S) are given in *Table* 3. Interaction parameters and standard deviations of the McAllister model and Krishnan and Laddha model for the viscosity of acetophenone and ethylchloroacetate mixtures at (303.15, 313.15, and 323.15) K are presented in *Table* 4 and 5. Interaction parameters and standard deviations of the Grunberg and Nissan, Hind, Tamura and Kurata, Katti and Chaudri, and Sedgwick models are presented in *Table* 6. Constants and standard deviations of the Jouban-

***Table* 3.** Parameters and standard deviations (S) of Redlich-Kister equation for Acetophenone (1) + Ethylchloroacetate (2) mixture at T = (303.15, 313.15, and 323.15) K

Functions	A_0	A_1	A_2	A_3	A_4	A_5	S
			303.15 K				
V^E/cm^{3}mol^{-1}	0.1163	0.0029	0.1982	-0.0059	-0.3076	0.0017	0.0023
Δη/mPa·s	0.0382	-0.0053	0.0259	0.0141	-0.0624	-0.0091	0.0006
Δn_D	0.0073	0.0003	0.0016	-0.0029	-0.0087	0.0026	0.00005
$\Delta k_s \times 10^{-11}$ m^2 N^{-1}	-4.0532	0.1622	-6.1812	3.2228	9.9599	-3.3248	0.09078
$\Delta L_f \times 10^{-11}$ m	-0.1559	0.0035	-0.2337	0.1113	0.3791	-0.1126	0.0003
Δu/ms^{-1}	21.054	0.6787	30.084	-10.077	-49.782	9.1405	0.0412
			313.15 K				
V^E/cm^{3}mol^{-1}	0.1234	0.0022	0.3023	0.0159	-0.4146	-0.0193	0.0037
Δη/mPa·s	0.0437	0.0013	0.0656	-0.0177	-0.1065	0.016	0.0009
Δn_D	0.0081	0.0003	0.0079	-0.0021	-0.0156	0.0018	0.00012
$\Delta k_s \times 10^{-11}$ m^2 N^{-1}	4.7724	0.1629	-6.8648	4.8216	11.332	-4.9091	0.1019
$\Delta L_f \times 10^{-11}$ m	-0.1794	0.0022	-0.2481	0.1755	0.4164	-0.175	0.0034
Δu/ms^{-1}	21.09	1.4383	23.853	-23.738	-43.841	21.984	0.04579
			323.15 K				
V^E/cm^{3}mol^{-1}	0.1327	0.0061	0.3376	-0.0067	-0.456	-0.001	0.0048
Δη/mPa·s	0.0531	0.0019	0.0784	-0.0238	-0.1281	0.0212	0.0011
Δn_D	0.009	0.0004	0.0133	-0.0042	-0.0218	0.0037	0.0001
$\Delta k_s \times 10^{-11}$ m^2 N^{-1}	-6.0458	0.3084	-9.3651	5.0749	15.001	-5.2911	0.1362
$\Delta L_f \times 10^{-11}$ m	-0.2236	0.0068	-0.3393	0.1655	0.548	-0.169	0.0045
Δu/ms^{-1}	23.553	0.8343	34.63	-10.496	-56.665	9.3741	0.0618

***Table* 4.** Parameters of the McAllister and standard deviation for the kinematic viscosity Acetophenone (1) + Ethylchloroacetate (2) mixtures at T = (303.15, 313.15, and 323.15) K

Temperature	McAllister constants		
T/K	A	B	S
303.15	1.35267	1.18349	0.0000
313.15	1.17775	1.03051	0.0000
323.15	1.11675	0.94779	0.0002

***Table* 5.** Parameters of the Krishnan - Laddha and standard deviation for the kinematic viscosity Acetophenone (1) + Ethylchloroacetate (2) mixtures at T = (303.15, 313.15, and 323.15) K

Temperature	Krishnan - Laddha Constants			
T/K	A_0	A_1	A_2	S
303.15	-0.05721	0.00929	0.00279	0.0531
313.15	-0.06446	0.01234	-0.00254	0.0088
323.15	-0.10102	0.02491	-0.00652	0.0193

Acree model of the acetophenone and n-alkyl acetate at (303.15, 313.15, and 323.15) K are presented in *Table* 7.

The excess molar volumes with the mole fraction (x_1) of acetophenone and ethylchloroacetate at (303.15, 313.15 and 323.15) K are represented in *Fig*. 1. This shows that the excess molar volumes are always positive for all the studied temperatures. The sign of excess volume of a system depends on the relative magnitude of expansion/contraction on mixing of two liquids. If the factors causing expansion dominate the factors causing contraction, then V^E becomes positive.[17,26] The factors that are responsible for expansion in volume are as follows. (1) Loss of dipolar association (2) the geometry of molecules structure, which does not allow fitting of one component in to other component. (3) Steric hindrance, which opposes the proximity of the constituent molecules. The experiment in present investigation indicates that the factors which are

***Table* 6.** Interaction parameters (G_{12}), (T_{12}), (H_{12}), (C) and (K_{12}) for Acetophenone and Ethylchloroacetate mixtures at 303.15, 313.15 and 323.15 K

T/K	G_{12}	S	T_{12}	S	H_{12}	S	C	S	K_{12}	S
303.15	0.1088	0.032	-1.2708	0.026	1.0502	0.045	0.0155	0.017	-0.0766	0.0203
313.15	0.1254	0.021	-1.1008	0.035	0.9166	0.036	0.0200	0.011	-0.1385	0.049
323.15	0.2046	0.023	-1.0160	0.018	0.8519	0.027	0.0243	0.025	-0.1331	0.051

***Table* 7.** Parameters and standard deviations of Jouyban-Acree model for Acetophenone (1) + Ethylchloroacetate (2) mixture at T = (303.15, 313.15, and 323.15) K

Physical Properties	T/K	A_0	A_1	A_2	A_3	A_4	S
	303.15	41.031	-5.50518	-39.919	-14.446	159.42	0.0011
ρ/g·cm^{-3}	313.15	47.782	-4.1707	-44.918	-28.834	201.87	0.0012
	323.15	79.947	-8.7436	-76.941	-60.423	349.56	0.0018
	303.15	-1.587	0.0072	1.3252	0.2613	-6.0984	0.00003
η/mPa·s	313.15	-1.5767	0.0145	1.4543	0.1762	-6.8706	0.00003
	323.15	-1.6796	0.0089	1.7603	0.2385	-7.9688	0.00003
	303.15	2.363	0.0046	-1.6297	-0.702	4.772	0.00007
n_D	313.15	2.7273	-0.0357	-2.2016	0.5171	8.5964	0.00005
	323.15	3.0375	-0.0359	-2.7709	-0.9381	12.105	0.00008
	303.15	10.004	-0.3656	-9.4081	-3.8573	40.564	0.02852
u/ms^{-1}	313.15	10.912	-0.2224	-9.4937	-7.4843	39.912	0.02892
	323.15	13.272	-0.532	-12.351	-5.2151	54.0	0.03320

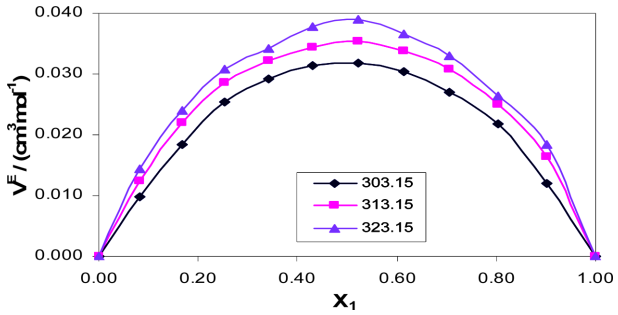

***Fig*. 1.** Excess molar volume (V^E) for acetophenone (1) + ethylchloroacetate (2) at ◆, 303.15 K; ■, 313.15 K; ▲, 323.15 K.

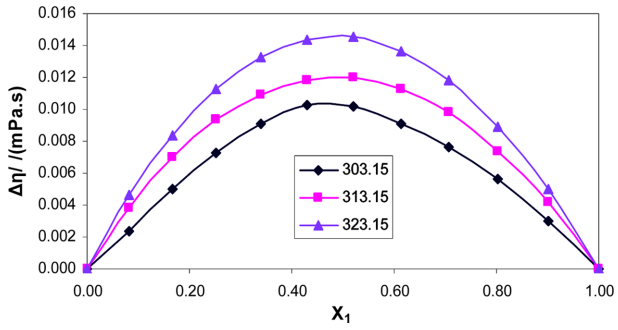

***Fig*. 2.** Viscosity Deviation (η) for acetophenone (1) + ethylchloroacetate (2) at ◆, 303.15 K; ■, 313.15 K; ▲, 323.15 K.

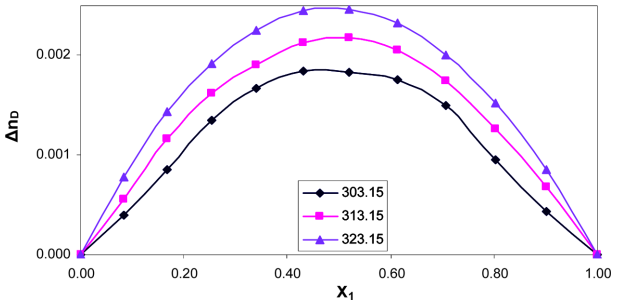

***Fig*. 3.** Refractive index deviation (Δn_D) for acetophenone (1) + ethylchloroacetate (2) at ◆, 303.15 K; ■, 313.15 K; ▲, 323.15 K.

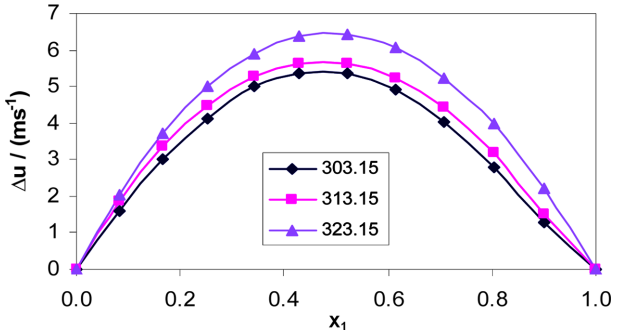

***Fig*. 4.** Excess ultrasonic velocity deviation (Δu) for acetophenone (1) + ethylchloroacetate (2) at ◆, 303.15 K; ■, 313.15 K; ▲, 323.15 K.

responsible for expansion in volume are dominant over the entire composition range in all the studied temperatures of acetophenone with ethylchloroacetate.

Viscosity deviations are positive over the entire range of composition for the binary mixture of acetophenone + ethylchloroacetate, which indicates that the interaction between binary mixtures is strong.[27] Experimental values of mixture viscosities and the viscosities of their components are used to evaluate the interaction parameters of various models like G_{12}, T_{12}, H_{12}, K_{12} and C values for the studied system are recorded in *Table* 6. The results of refractive index deviations versus x_1 at (303.15, 313.15 and 323.15) K for the system of acetophenone and ethylchloroacetate are shown in *Fig*. 3. In *Fig*. 3, the positive values are observed for the system of acetophenone with ethylchloroacetate.

The system of acetophenone and ethylchloroacetate shows negative deviations through Δk_s isotherms over the entire range of mole fractions (*Fig*. 5) and exhibiting a

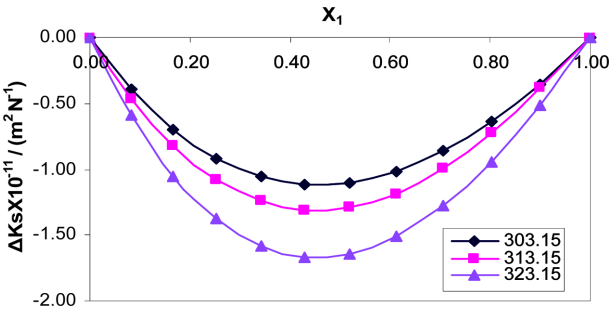

***Fig.* 5.** Excess isentropic compressibility (Δk_s) for acetophenone (1) + ethylchloroacetate (2) at ◆, 303.15 K; ■, 313.15 K; ▲, 323.15 K.

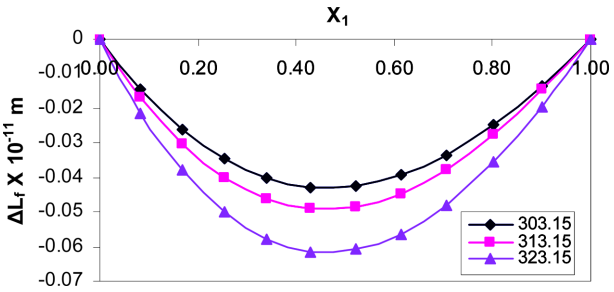

***Fig.* 6.** Excess inter molecular free length (ΔL_f) for acetophenone (1) + ethylchloroacetate (2) at ◆, 303.15 K; ■, 313.15 K; ▲, 323.15 K.

clear minima at the mole fraction around x_1=0.45, indicating that the maximum interactions are at that mole fraction range in every system. From *Fig*. 4, it is observed that Δu values are positive while values are negative. Such a trend of positive deviation in speed of sound and negative deviation in isentropic compressibility is quite common. The excess free length is negative over the whole mole fraction range for binary mixtures at different temperatures, *Fig*. 6. This indicates that due to intermolecular interaction, the structural readjustment occurs in the liquid mixtures towards the less compressible phase of fluid.[23,24]

CONCLUSIONS

Experimental data of the density, viscosity, refractive index and ultrasonic velocity of acetophenone and ethylchloroacetate mixture have been measured at 303.15, 313.15 and 323.15 K. These data have used to compute the excess molar volumes, viscosity deviations, refractive index deviations, compressibility deviation and change in intermolecular free length for mixtures of acetophenone and ethylchloroacetate. Negative deviations were observed for Δk_s and ΔL_f. The positive deviations were observed for V^E, $\Delta\eta$, Δn_D and Δu. The results are analyzed in the sight of molecular interactions between the components. It may be concluded that the interactions resulting in the interstitial accommodation of ethylchloroacetate into acetophenone are the predominant factor over dipole-dipole interaction. The intermolecular interactions between acetophenone and ethylchloroacetate mixture lead to weak dispersive type. The viscosity data are correlated with the Grunberg and Nissan, Hind, Tamura and Kurata, Katti and Chaudri, Sedgwick, Krishnan- Laddha and McAllister. It has been concluded that the Jouyban Acree model is very well suited for correlating the thermo physical properties of the studied binary mixture.

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