ijCEPr

International Journal of
Chemical, Environmental and
Pharmaceutical Research

Vol. 2, No.1, 5-11
January-April, 2011

Molecular Interaction Studies between H-Bonded Ternary Mixtures of *p*-Cresol with simple Aldehydes in Cyclohexane at Different Temperatures

M. Aravinthraj[1,*], S. Venkatesan[2] and M. Kamaraj[1]
[1]Department of Physics, Sacred Heart College, Tirupattur, Tamilnadu, India.
[2]Department of Chemistry, Sacred Heart College, Tirupattur, Tamilnadu, India.
*E-mail: mar.rebo84@gmail.com

Article History:
Received:12 February 2011
Accepted:27 February 2011

ABSTRACT

The ultrasonic velocity (U), Density (ρ) and Viscosity (η) have been measured for three ternary liquid mixtures of P-Cresol + Cyclohexane + Formaldehyde, P-Cresol + Cyclohexane + Acetaldehyde and P-Cresol + Cyclohexane + Benzaldehyde at 303K, 313K and 323K. The experimental data have been used to calculate the acoustical parameters such as adiabatic compressibility (β), free length (L_f), free volume (V_f), internal pressure (π_i), viscous relaxation time (τ) and Gibb's free energy (ΔG) were evaluated. The obtained results support the occurrence of dipole-dipole interactions and molecular association through intermolecular hydrogen bonding in these ternary liquid mixtures.

Key words: Ternary liquid mixture, Acoustical parameter, Molecular association, dipole-dipole interactions.

INTRODUCTION

The studies of multi-component (Binary and ternary liquid) mixtures and solutions have found wide applications in chemical, textile, leather and nuclear industries [1-3]. The study and understanding of thermo dynamical and transport properties of liquid mixtures and solutions are more essential for their application in these industries. It increases interest among several workers for the study of molecular interaction in binary [4, 5] and ternary [6, 7] liquid mixtures in recent past employing ultrasonic velocity measurement. This precisely helps to understanding the molecular interactions and structural behavior of molecules and their mixture.

In this paper, we report on the ultrasonic study of four ternary liquid mixtures (p-Cresol + Cyclohexane + Formaldehyde, p-Cresol + Cyclohexane + Acetaldehyde and p-Cresol + Cyclohexane + Benzaldehyde). The cyclohexane is a non-polar unassociated, inert hydrocarbons possesses globular structure [8], it has ring structure as benzene without any S electron and serves as reference point for comparison of the molecular interactions. Cresols are organic compounds which are methyl phenols. It has isomeric and methyl group substituted onto the benzene ring of a phenol molecule. It has three forms (ortho, meta and para), one of the o-,m- or p- positions relative to the OH group. The compounds are highly flammable moderately soluble in water and soluble in ethanol, ether, acetone cyclohexane and alkalies. Chemically these alkyl phenols undergo electro-philic substitution reactions at the condensation reaction with aldehydes, ketones [9]. The interactions between cresols and aldehydes may probably be dipole-dipole or may be due to intra molecular hydrogen bonding [10].

EXPERIMENTAL

The chemicals used in the present work were Analytical reagent (AR) grades with a minimum assay of 99.9%, obtained from E-Merck (Germany) and Loba chemicals; they are used without further purification. In all the systems, the various concentrations of the ternary liquid mixtures were prepared in terms of mole fraction, out of which the mole fraction of the second compound, cyclohexane (X_2=0.3) was kept fixed while the mole fraction of the remaining two (X_1 and X_3) were varied from 0.0 to 0.7. The ultrasonic velocity was measured by a single crystal interferometer with a high degree of accuracy operating frequency of 2MHz supplied by M/s. Mittal Enterprises, New Delhi. Water was circulated around the double walled sample holder to maintain the experimental temperatures. (Say 303K, 313K and 323K) The density of all compounds was measured by a 10 ml specific gravity bottle calibrated with double distilled water and acetone. An Ostwald's viscometer with 10ml capacity was used for the viscosity measurements of all the compounds. The viscometer was immersed in fresh conductivity water bath that can be operated at desired temperatures. The flow time of water (t_w) and the flow time of solution (t_s) were measured with a digital stop clock with an accuracy of 0.01s (RACER HS-10w).

Theory and Calculations

The longitudinal ultrasonic velocity (U), density (ρ) and viscosity (η) of unknown liquid mixtures at any experimental temperatures are calculated using the following relations. And the density, viscosity of water at different temperatures was taken from literature [11, 12]. The accuracy in the measurement of density in this method depends on the accuracy of the weight. The accuracy in the measurement of density is of the order of ± 0.1 kgm^{-3}. And the accuracy in the measurement of viscosity depends on the accuracy in the determination of time and density. The overall accuracy of the measurement of viscosity in this method is ± $0.001Nsm^{-1}$.

Ultrasonic velocity (U)

$$U = n\lambda \ldots\ldots\ldots\ldots\ldots\ldots.(ms^{-1}) \qquad (1)$$

Where n is the frequency of Ultrasonic wave in Hz, and λ is the wavelength of the Ultrasonic wave in solution under study in meter.

Viscosity (η_s)

$$\eta_s = \eta_w\left(\frac{\rho_s t_s}{\rho_w t_w}\right)\ldots\ldots\ldots\ldots(NSm^{-1}) \qquad (2)$$

Where η_w is the co-efficient of viscosity of water in Nsm^{-1}, ρ_w is the density of water in Kg/m^3, t_w is the flow of time for water in seconds, ρ_s is the density of solution in Kg/m^3 , and t_s is the flow of time solution in seconds.

Density (ρ_2)

$$\rho_2 = \frac{w_2}{w_1}\rho_1 \ldots\ldots\ldots\ldots\ldots..(Kg/m^3) \qquad (3)$$

Where w_1 & w_2 are weights of distilled water and experimental liquid and ρ_1 & ρ_2 are the densities of water and experimental liquid.

Using experimentally determined values of ultrasonic velocity (U), density (ρ) and viscosity (η), the following acoustic and thermodynamic parameters are evaluated.

Adiabatic compressibility (β)

$$\beta = (U^2\rho)^{-1}\ldots\ldots\ldots\ldots\ldots(N^{-1}\mathrm{m}^2) \qquad (4)$$

Where U is the velocity measured in meter/second and ρ is density measured in Kg/m^3.

Free Length (L_f)

$$L_f = K_T\beta^{\frac{1}{2}}\ldots\ldots\ldots\ldots\ldots\ldots.(m) \qquad (5)$$

Where K_T is Jacob's constant and β is the adiabatic compressibility of a liquid mixtures measured in $N^{-1}m^2$.

Free Volume (V_f)

Suranarayana et al [13] obtained a formula for free volume in term of the ultrasonic velocity (V) and the viscosity of the liquid (η) as,

$$V_f = \left[\frac{M_{eff}U}{K\eta}\right]^{\frac{3}{2}}\ldots\ldots\ldots\ldots\ldots..(m^3mol^{-1}) \qquad (6)$$

Where M_{eff} is the effective molecular weight ($M_{eff} = \sum m_i x_i$ in which m_i and x_i are the molecular weight and the mole fraction of the individual constituents respectively) and K is a temperature independent constant equal to 4.28×10^9 for all liquids.

Internal Pressure (π_i)

On the basis of statistical thermodynamics, Surayanarayana [14] derived an expression for the determination of internal pressure through the use of the concept of free volume

$$\pi_i = bRT\left[\frac{K\eta}{U}\right]^{\frac{1}{2}}\left[\frac{\rho^{\frac{1}{3}}}{M^{\frac{7}{6}}}\right]\ldots\ldots\ldots\ldots\ldots(Pa) \qquad (7)$$

Where T is absolute temperature in Kelvin, R is the universal gas constant and *b* is the cubic packing fraction factor is assumed to be '2' for all liquid systems.

Viscous relaxation time (τ)

$$\tau = \frac{4}{3}\beta\eta\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(s) \qquad (8)$$

Where η is the viscosity of the solution in Nsm^{-2} and β is adiabatic compressibility in $N^{-1}m^2$

Gibb's free energy (ΔG)

The Gibb's free energy of activation flow in the mixtures can be obtained on the basis of Erying rate process theory and it can be able to calculate from the relation,

$$-\Delta G = -KTlog\left(\frac{h}{KT\tau}\right)\dots\dots\dots(KJmol^{-1}) \quad (9)$$

Where τ is the viscous relaxation time measured in meters, K is the Boltzmann's constant, h is a Plank's constant and T is the temperature measured in kelvin.

RESULT AND DISCUSSION

The experimentally measured values of density, viscosity and ultrasonic velocity of the three ternary systems of p-Cresol + Cyclohexane + Formaldehyde, p-Cresol + Cyclohexane + Acetaldehyde and p-Cresol + Cyclohexane + Benzaldehyde at 303K, 313K and 323K are presented in Table-1. The systems taken for the present study is basically an H-Bonded complexes of phenol (p-cresol) with aldehydes in cyclohexane. Here mole fraction of cyclohexane is fixed as 0.3 and the mole fraction of the other two components are varied from 0.0 to 0.7.

Acoustical parameters such as adiabatic compressibility (β), intermolecular free length (L_f), free volume (V_f), internal pressure (π_i), kinetic parameter like viscous relaxation time (τ) and thermo dynamical parameter like Gibb's Free Energy (ΔG) were calculated from the measured ultrasonic velocity, density and viscosity using standard equations given in theory and calculations.

From the measured data, it is observed that there is a sudden fall of density in all the systems for second molar concentration, and from the second concentration onwards p-cresol is made to dissolve in cyclohexane for which aldehydes are used as doped. When aldehyde and cyclohexane are taken without p-cresol, the density is found to be higher. This serves the possibility of greater interaction between the aldehydes and solvent due to dipole-dipole interaction as well as possibility of weak H-bond shown in Figure (a).

The solvent has hydrogens placed at the 1,3-diaxial position, such that any of these hydrogens are free to interact with non-bonding electrons (or) n-electron from the carbonyl oxygen of aldehydes Figure (b). This serves to the increasing density with the increasing of aldehydes concentration. From the second molar concentration onwards a gradual increasing trend in density is observed. Figure (a) shows the interaction between Oxygen of the p-cresol and flagpole Hydrogen of the Cyclohexane. This dipole-dipole interaction serves for the gradual increase in density.

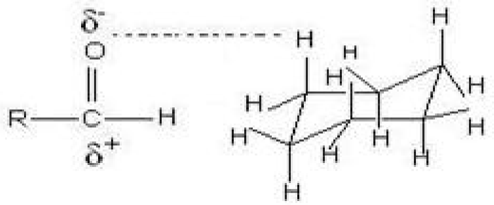

Fig. (a)

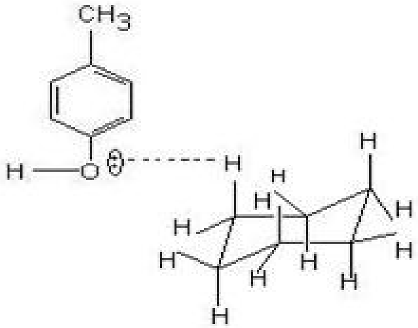

Fig. (b)

From the second concentration onwards the influence of non bonding electron is frankly reduced when p-cresol is began to introduce. Another kind of factor is observed when the mole fraction of aldehydes is taken as 0(zero), and the mole fraction of p-cresol is taken as 0.7, here also the density is greater because of the possible interaction between the non bonding electrons of oxygen and the cyclohexane. The mole fraction of cyclohexane is taken as 0.3 M, which is constant for the overall experiment. From the second concentration onwards p-cresol and aldehydes are mixed with different molar concentration in cyclohexane, and then increasing trend is observed in density, viscosity and velocity. This is because of more hydrogen bonding and dipole-dipole interaction observed as shown in Fig (c) and Fig (d) respectively. This behavior at such concentrations is same as the ideal mixtures behavior can be attributed to intermolecular interactions in the systems studied [16]. It is evident from table-1.

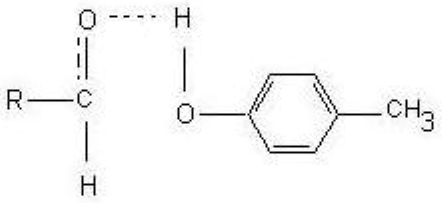

Fig. (c)

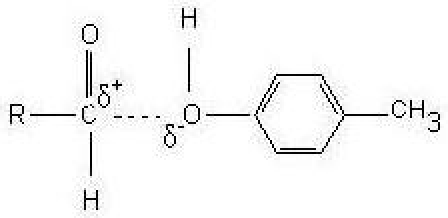

Fig. (d)

It is found that from the table-2, the adiabatic compressibility and intermolecular free length decreases with increase in concentration of p-cresol in all the three systems. This shows an inverse behavior as compared to ultrasonic velocity. This indicates that there is a significant interaction between solute and solvent molecules. It can be taken as the indication of complex formation.

The addition of interacting molecules breaks up the molecular clustering of the other, releasing several interaction, because p-cresol has the possibility of forming intermolecular H-bonding with such carbonyl oxygen. However free volume increases with increase in molar concentration of p-cresol, the p-cresol system has the lone pair of electron in oxygen of p-cresol system interact with Hydrogen in hydroxyl group of another p-cresol system. This is clearly observed in the increasing trend in free volume with increasing molar concentration of p-cresol. At the same time the mole fraction of doped is purposely decreased, because this factor holds well only from the introduction of p-cresol.

From Table-3 it is found that, internal pressure decreases with increase in concentration of p-cresol, because the hydrogen releasing ability of p-cresol is becoming low. The decreasing density with the increase in concentration of p-cresol and the decreasing concentration of aldehydes is contributed from the above explanation. Also a decrease in internal pressure with the decrease in size of the alkyl group of aldehyde is observed. This is naturally due to the increasing available space with the decreasing size of alkyl group.

However, the viscous relaxation time increases with increasing molar concentration of p-cresol as shown in Table -3 and a decrease in all these values is also noted with the increasing temperature. This is clearly due to the increasing free space between the molecules and the weakening of intermolecular forces. Generally, viscous relaxation time is of the order of 10^{-12} s, for identifying structural relaxation between the component molecules. This shows the presence of molecular interactions [17], Gibb's free energy confirms the same, which indicates the need for smaller time for the cooperative process or the rearrangement of the molecules in the mixtures.

Table -1: Density (ρ), Viscosity (η) and Velocity (U) of p-Cresol + Cyclohexane + Formaldehyde, p-Cresol + Cyclohexane + Acetaldehyde and p-Cresol + Cyclohexane + Benzaldehyde at 303K, 313K and 323K

Mole fraction		Density (ρ)			Viscosity (η)			Velocity (U)		
		(Kg/m^3)			(10^{-3}Nsm^{-2})			(ms^{-1})		
X_1	X_2	303K	313K	323K	303K	313K	323K	303K	313K	323K
p-Cresol + Cyclohexane + Formaldehyde										
0.000	0.705	887.7	883.8	880.3	1.704	1.339	1.106	1310.1	1306.5	1298.1
0.100	0.606	846.2	841.1	837.0	2.890	2.344	1.829	1323.9	1318.7	1309.9
0.197	0.498	870.6	866.1	861.2	2.955	2.389	1.853	1338.1	1330.0	1318.9
0.301	0.403	871.3	867.9	863.3	3.112	2.398	1.899	1345.9	1339.2	1323.2
0.399	0.297	873.0	867.9	864.0	3.075	2.435	1.911	1357.3	1348.9	1336.8
0.497	0.195	875.1	870.3	866.8	3.232	2.458	2.023	1364.5	1353.1	1349.3
0.607	0.104	877.0	873.5	869.8	3.345	2.701	1.985	1374.7	1363.4	1354.8
0.705	0.000	879.0	875.2	870.6	3.538	2.901	2.189	1361.0	1357.3	1349.3
p-Cresol + Cyclohexane + Acetaldehyde										
0.000	0.701	871.2	868.1	863.9	1.415	1.050	0.880	1309.3	1300.5	1295.1

0.102	0.604	842.1	837.5	830.3	2.190	1.845	1.407	1319.8	1310.1	1301.9
0.199	0.501	867.1	852.8	847.7	2.503	2.106	1.524	1326.9	1320.1	1312.1
0.305	0.398	868.3	863.3	859.5	2.621	2.161	1.577	1333.8	1328.6	1322.4
0.398	0.301	870.3	866.8	862.3	2.859	2.223	1.590	1349.7	1337.5	1331.6
0.504	0.200	874.4	869.3	865.8	3.105	2.236	1.777	1356.4	1350.0	1339.6
0.601	0.098	875.0	872.5	867.2	3.256	2.388	1.954	1360.0	1354.0	1347.8
0.700	0.000	879.0	875.5	869.6	3.506	2.896	2.185	1360.1	1355.5	1350.9
p-Cresol + Cyclohexane + Benzaldehyde										
0.000	0.700	870.2	868.5	867.3	1.024	0.885	0.773	1307.9	1304.5	1300.3
0.101	0.603	838.2	835.2	832.3	1.216	1.004	0.890	1310.7	1296.0	1289.1
0.203	0.501	858.8	855.2	851.8	1.426	1.193	1.046	1321.7	1305.8	1295.0
0.299	0.404	860.1	859.8	857.4	1.711	1.362	1.199	1329.7	1310.7	1302.2
0.403	0.308	869.3	867.2	865.9	1.950	1.604	1.315	1338.4	1318.8	1309.4
0.503	0.199	870.0	868.9	864.6	2.427	2.025	1.560	1345.2	1331.9	1320.5
0.599	0.104	874.4	872.9	871.8	2.879	2.463	1.758	1350.4	1333.6	1326.0
0.703	0.000	878.0	876.9	872.0	4.096	2.850	2.223	1359.5	1351.4	1328.6

Table -2: Adiabatic compressibility (β), Free Volume (V_f), and Free Length (L_f) of p-Cresol + Cyclohexane + Formaldehyde, p-Cresol + Cyclohexane + Acetaldehyde and p-Cresol + Cyclohexane + Benzaldehyde at 303K, 313K and 323K

Mole fraction		Adiabatic compressibility (β)			Free length (L_f)			Free Volume (V_f)		
		(10^{-10} $N^{-1}m^2$)			(10^{-10}m)			(10^{-7} m^3 mol^{-1})		
X_1	X_2	303K	313K	323K	303K	313K	323K	303K	313K	323K
p-Cresol + Cyclohexane + Formaldehyde										
0.000	0.705	6.5630	6.6280	6.7406	5.0564	5.0814	5.1243	0.1719	0.2457	0.3241
0.100	0.606	6.7417	6.8365	6.9626	5.1248	5.1607	5.2080	0.0908	0.1236	0.1776
0.197	0.498	6.4143	6.5264	6.6750	4.9988	5.0423	5.0993	0.1045	0.1424	0.2059
0.301	0.403	6.3355	6.4244	6.6158	4.9680	5.0027	5.0767	0.1158	0.1700	0.2368
0.399	0.297	6.2174	6.3316	6.4759	4.9215	4.9665	5.0227	0.1461	0.2054	0.2915
0.497	0.195	6.1368	6.2751	6.3367	4.8895	4.9443	4.9684	0.1719	0.2561	0.3414
0.607	0.104	6.0332	6.1580	6.2630	4.8480	4.8979	4.9395	0.2121	0.2886	0.4538
0.705	0.000	6.1412	6.2017	6.3090	4.8912	4.9153	4.9576	0.2670	0.3584	0.5419
p-Cresol + Cyclohexane + Acetaldehyde										
0.000	0.701	6.6950	6.8109	6.9006	5.1070	5.1510	5.1848	0.3690	0.5713	0.7399
0.102	0.604	6.8164	6.9559	7.1049	5.1531	5.2055	5.2610	0.2160	0.2763	0.4108
0.199	0.501	6.5500	6.7283	6.8512	5.0514	5.1197	5.1662	0.1998	0.2569	0.4137
0.305	0.398	6.4735	6.5618	6.6524	5.0218	5.0559	5.0907	0.2143	0.2846	0.4533
0.398	0.301	6.3074	6.4486	6.5395	4.9570	5.0121	5.0473	0.2182	0.3139	0.5154
0.504	0.200	6.2175	6.3114	6.4360	4.9215	4.9585	5.0072	0.2265	0.3683	0.5137
0.601	0.098	6.1789	6.2513	6.3472	4.9062	4.9349	4.9726	0.2509	0.3968	0.5324

0.700	0.000	6.1495	6.2163	6.3011	4.8945	4.9210	4.9545	0.2703	0.3583	0.5438
p-Cresol + Cyclohexane + Benzaldehyde										
0.000	0.700	6.7172	6.7653	6.8191	5.1155	5.1337	5.1541	1.5849	1.9652	2.3944
0.101	0.603	6.9437	7.1278	7.2292	5.2010	5.2695	5.3068	1.2331	1.6165	1.9208
0.203	0.501	6.6652	6.8571	6.9996	5.0956	5.1685	5.2219	0.9860	1.2654	1.5216
0.299	0.404	6.5750	6.7692	6.8774	5.0610	5.1352	5.1761	0.7586	1.0451	1.2539
0.403	0.308	6.4210	6.6292	6.7353	5.0014	5.0818	5.1223	0.6322	0.8285	1.1041
0.503	0.199	6.3511	6.4868	6.6325	4.9741	5.0270	5.0831	0.4594	0.5938	0.8670
0.599	0.104	6.2709	6.4413	6.5228	4.9426	5.0093	5.0409	0.3587	0.4447	0.7310
0.703	0.000	6.1616	6.2441	5.3932	4.8993	4.9320	4.5837	0.2141	0.3655	0.5946

Table -3: Internal Pressure (π_i), Viscous Relaxation Time (τ) and Gibb's free energy (ΔG) of p-Cresol + Cyclohexane + Formaldehyde, p-Cresol + Cyclohexane + Acetaldehyde and p-Cresol + Cyclohexane + Benzaldehyde at 303K, 313K and 323K

Mole fraction		Internal pressure (π_i)			Viscous Relaxation Time (τ)			Gibb's Free Energy (ΔG)		
		(10^{-6} Pa)			(10^{-12}s)			(10^{-20} KJ mol^{-1})		
X_1	X_2	303K	313K	323K	303K	313K	323K	303K	313K	323K
p-Cresol + Cyclohexane + Formaldehyde										
0.000	0.705	14.090	12.472	11.342	1.4911	1.1836	0.9943	0.4077	0.3898	0.3686
0.100	0.606	15.867	14.397	12.878	2.5982	2.1373	1.6985	0.5086	0.4948	0.4722
0.197	0.498	14.769	13.754	11.907	2.5280	2.0793	1.6497	0.5036	0.4897	0.4666
0.301	0.403	12.561	11.314	9.971	2.6295	2.0545	1.6758	0.5017	0.4874	0.4696
0.399	0.297	10.921	9.529	8.429	2.5493	2.0563	1.6505	0.5051	0.4876	0.4667
0.497	0.195	9.357	8.127	6.977	2.6448	2.0565	1.7094	0.5118	0.4857	0.4735
0.607	0.104	7.818	6.712	6.034	2.6908	2.2184	1.6581	0.5149	0.5018	0.4676
0.705	0.000	6.829	5.661	4.915	2.8985	2.3989	1.8416	0.5284	0.5165	0.4879
p-Cresol + Cyclohexane + Acetaldehyde										
0.000	0.701	8.691	7.495	6.854	1.2634	0.9539	0.8101	0.3776	0.3435	0.3289
0.102	0.604	9.684	8.887	7.742	1.9907	1.7115	1.3336	0.4602	0.4531	0.4254
0.199	0.501	9.630	8.997	7.603	2.1867	1.8898	1.3923	0.4773	0.4717	0.4337
0.305	0.398	8.883	7.948	6.873	2.2624	1.8907	1.3992	0.4834	0.4718	0.4347
0.398	0.301	8.345	7.268	6.096	2.4048	1.9118	1.3870	0.4945	0.4739	0.4330
0.504	0.200	7.719	6.539	5.837	2.5746	1.8818	1.5252	0.5069	0.4709	0.4514
0.601	0.098	6.924	5.931	5.356	2.6831	1.9910	1.6542	0.5144	0.4815	0.4671
0.700	0.000	6.782	5.664	4.907	2.8753	2.4005	1.8364	0.5269	0.5166	0.4873
p-Cresol + Cyclohexane + Benzaldehyde										
0.000	0.700	3.464	3.220	3.012	0.9178	0.7988	0.7035	0.3196	0.3102	0.3016
0.101	0.603	3.669	3.344	3.150	1.1262	0.9543	0.8582	0.3568	0.3436	0.3401
0.203	0.501	4.013	3.682	3.453	1.2676	1.0910	0.9767	0.3782	0.3687	0.3651
0.299	0.404	4.379	3.605	3.362	1.5007	1.2301	1.0996	0.4089	0.3912	0.3881

0.403	0.308	4.679	4.269	3.875	1.6696	1.4183	1.1814	0.4283	0.4179	0.4019
0.503	0.199	5.203	4.773	4.193	2.0560	1.7520	1.3800	0.4661	0.4575	0.4320
0.599	0.104	5.663	5.265	4.458	2.4073	2.1157	1.5297	0.4947	0.4929	0.4519
0.703	0.000	6.737	5.632	4.771	3.3652	2.3731	1.5989	0.5555	0.5145	0.4605

CONCLUSION

From the experimental and calculated parameters, there is a significant interactions observed only after introducing p-Cresol. This increases density, viscosity and velocity of all the liquid mixtures. This increasing trend is due to the intermolecular hydrogen bonding between carbonyl oxygen of aldehydes with hydroxyl hydrogen in p-cresol and dipole- dipole interaction between the same compounds with carbonyl carbon of aldehydes and hydroxyl oxygen. Hence this investigation provides comprehensive idea about the molecular interactions between solute and solvent. The order of interactions is found to be Formaldehyde > Acetaldehyde > Benzaldehyde.

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[IJCEPR-151/2011]
