

## Ultrasonic interaction studies between hydrogen bonded ternary mixtures of aniline with aliphatic ketones in cyclohexane at different temperatures

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### ABSTRACT

The ultrasonic velocity ( $U$ ), Density ( $\rho$ ) and Viscosity ( $\eta$ ) have been measured for three ternary liquid mixtures of Aniline + Cyclohexane + Ethyl methyl Ketone, Aniline + Cyclohexane + Dimethyl Ketone and Aniline + Cyclohexane + Iso-butyl methyl Ketone at 303K, 313K and 323K. The experimental data have been used to calculate the acoustical parameters such as adiabatic compressibility ( $\beta$ ), free length ( $L_f$ ), free volume ( $V_f$ ), internal pressure ( $\pi$ ), viscous relaxation time ( $\tau$ ) and Gibb's free energy ( $\Delta G$ ) were evaluated. The obtained results support the occurrence of molecular association through intermolecular hydrogen bonding in these ternary liquid mixtures.

**Key words:** Ternary liquid mixture, Acoustical parameter and Molecular association

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### INTRODUCTION

The study of molecular interaction plays a vital role in the development of molecular science. Molecular interactions and structural behavior of molecular and their mixtures can be identified using ultrasonic studies. Ultrasonic waves have been used by many scientists to investigate the nature of molecular interactions and physico-chemical behaviors of pure, binary and ternary liquid mixtures [1, 2].

Ultrasonic velocity together with density and viscosity data furnish wealth of information about the interaction between ions, dipoles, hydrogen bonding, multipolar and dispersive forces [3,4]. Aniline molecule is highly polar and self associated through hydrogen bonding of their amine group. Being aromatic, aniline with amino group is comparatively a strong electron donor. The H atoms in the  $\text{NH}_2$  group can also play the role of electron-acceptors centers [5]. It is used in the manufacturing of synthetic dyes, drugs and as an accelerator in vulcanization of rubber [6].

Dimethyl ketone, Ethyl methyl ketone and Iso butyl methyl ketone are all aprotic in nature and belong to the same ketone homologous series. Especially they have aliphatic nature and have common carbonyl (C=O) functional group [7].

In the present study aniline and ketones are mixed with cyclohexane and it forms three ternary mixture systems as follows.

1. Aniline + Cyclohexane + Di-methyl ketone
2. Aniline + Cyclohexane + Ethyl methyl ketone
3. Aniline + Cyclohexane + Iso-butyl methyl ketone.

For a better understanding of the physico-chemical properties and the molecular interactions between the participating components of these mixtures ultrasonic velocities and densities are measured at 303K, 313K and 323K over the entire concentration range, using the experimental data ultrasonic velocity (U), density ( $\rho$ ) and viscosity ( $\eta$ ) have been found. Various thermodynamic parameters like adiabatic compressibility, free length ( $L_f$ ), free volume ( $V_f$ ), internal pressure ( $\pi_i$ ), viscous relaxation time ( $\tau$ ) and Gibb's free energy ( $\Delta G$ ) were also been evaluated. The variations of these parameters with composition of the mixtures are discussed in terms of molecular interactions.

## MATERIALS AND METHODS

High purity Analytic reagent (AR) grades chemicals used in the present work were purchased from E-Merck (Germany) with of minimum assay of 99.9%. In all the systems, 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 in the liquid mixtures has been measured using a single crystal ultrasonic interferometer with an operating frequency of 2MHz supplied by M/s. Mittal Enterprises, New Delhi. 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 flow time of water ( $t_w$ ) and the flow time of solution ( $t_s$ ) were measured by a digital stop clock with an accuracy of 0.01s.

### Theory and calculation

Using the measured data of density ( $\rho$ ), velocity (U) and viscosity ( $\eta$ ), the acoustical parameters such as adiabatic compressibility ( $\beta$ ), free length ( $L_f$ ), free volume ( $V_f$ ), internal pressure ( $\pi_i$ ), viscous relaxation time ( $\tau$ ) and Gibb's free energy ( $\Delta G$ ) have been calculated using the following standard expressions [8, 9].

### Adiabatic compressibility ( $\beta$ )

When acoustical wave passes through a medium, adiabatic compression and rarefaction takes place. This results in a change in pressure and a corresponding change in volume. Hence, the adiabatic compressibility is the fractional decrease of volume per unit increase of pressure, when no heat flows in or out. It can be calculated from the speed of sound (U) and the density of the medium ( $\rho$ ) using the equation of Newton and Laplace as

$$\beta = (U^2 \rho)^{-1} \dots \dots \dots (N^{-1} m^2) \quad (1)$$

**Intermolecular free length ( $L_f$ )**

In the liquid state of matter, molecules are loosely packed, leaving free space among them. The free length is the distance between the surfaces of the neighbouring molecules. Determination of intermolecular free length in liquids and in liquid mixtures has been a subject to a semi-empirical relation to achieve the concept of intermolecular free length in order to explain the ultrasonic velocity in liquids.

$$L_f = K_T \beta^{\frac{1}{2}} \dots \dots \dots (m) \quad (2)$$

Where  $K_T$  is a temperature dependent constant.

**Free volume ( $V_f$ )**

Free volume is one of the significant factors in explaining the free space and its dependent properties have close connection with molecular structure and it may show interesting features about interactions between liquid mixtures. This molecular interactions between like and dislike molecules are influenced by structural arrangements along with shape and size of the molecules. That is, the molecules of a liquid are not quite closely packed and there are some free spaces between the molecules for movement and this volume is called the free volume.

Based on the dimensional analysis, Suryanarayana *et al* [10] obtained a relation for free volume in terms of ultrasonic velocity (U) and the viscosity of the liquid ( $\eta$ ) as

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

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). K is a temperature independent constant.

**Internal pressure ( $\pi_i$ )**

The internal pressure is a measure of the resultant force of attraction and repulsion between the interacting components in the mixture. The internal pressure is the single factor which varies due to all type of solvent-solute, solute-solute and solvent-solvent interactions. On the basis of statistical thermodynamics, Suryanarayana *et al.* [11] derived an expression for the determination of internal pressure by the use of free volume concept as

$$\pi_i = bRT \left[ \frac{K\eta}{U} \right]^{\frac{1}{2}} \left[ \frac{\rho^{\frac{1}{3}}}{M^{\frac{1}{6}}} \right] \dots \dots (Pa) \quad (4)$$

Where b stands for the cubic packing factor which is assumed to be 2 for all liquids and solutions. K is the temperature independent constant, R is the gas constant, T is the absolute temperature,  $\eta$  is the viscosity, U is the ultrasonic velocity,  $\rho$  is the density and M is the effective molecular weight.

**Viscous Relaxation Time ( $\tau$ ):**

Relaxation time and absorption coefficient are directly correlated. The absorption of sound wave is the result of the time lag between the passing of the ultrasonic wave and the return of the molecules to their equilibrium position. It is calculated using the relation.

$$\tau = \frac{4}{3} \beta \eta \dots \dots \dots (s) \quad (5)$$

**Gibb's Free Energy ( $\Delta G$ )**

The Gibb's Free Energy of activation flow in the mixtures can be obtained on the basis of Eyring rate process theory [12] and it can be able to calculated from the relation,

$$-\Delta G = -KT \log \frac{h}{KT\tau} \dots (KJmol^{-1}) \quad (6)$$

Where K is Boltzmann's constant.

**RESULT AND DISCUSSION**

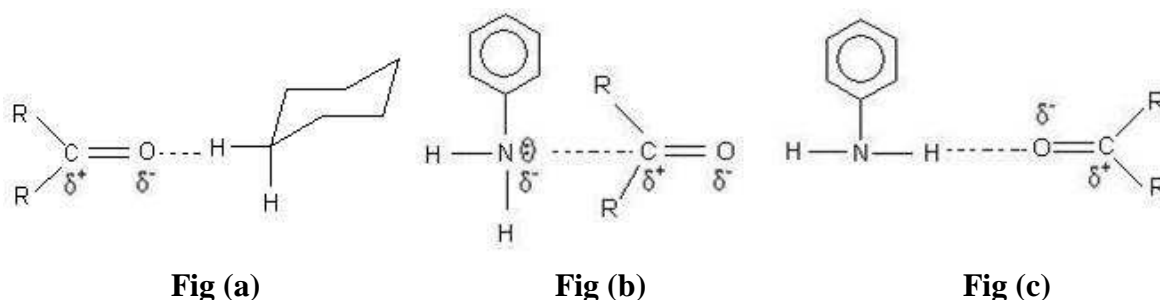
The values of density, viscosity and ultrasonic velocity of intermolecular H-bonded complexes of aniline with aliphatic ketones with different mole fractions in cyclohexane at 303K, 313K and 323K are presented in Table-1. The acoustical parameter viz., Adiabatic compressibility ( $\beta$ ), Free Length ( $L_f$ ), Free Volume ( $V_f$ ), thermo dynamical parameters viz., Internal pressure( $\pi_i$ ), Gibb's Free Energy ( $\Delta G$ ) and kinetic acoustical parameter like Viscous relaxation time ( $\tau$ ) are presented in Table 2 and 3.

**Table- 1: values of Density ( $\rho$ ), Viscosity ( $\eta$ ) and Velocity (U) at 303K, 313K and 323K**

Mole fraction		Density ( $\rho$ ) (Kgm <sup>-3</sup> )			Viscosity ( $\eta$ ) (10 <sup>-3</sup> Nsm <sup>-2</sup> )			Velocity (U) (ms <sup>-1</sup> )		
X <sub>1</sub>	X <sub>3</sub>	303K	313K	323K	303K	313K	323K	303K	313K	323K
Aniline + Cyclohexane + Di methyl ketone										
0.000	0.701	813.5	808.1	796.1	0.4655	0.4146	0.3449	1174.0	1130.2	1099.6
0.100	0.602	823.9	819.2	810.0	0.5462	0.4866	0.4340	1200.7	1170.1	1119.7
0.204	0.501	833.7	828.6	822.4	0.6743	0.5907	0.5122	1250.1	1210.0	1159.7
0.303	0.404	844.1	838.7	834.2	0.8377	0.6933	0.6278	1323.5	1300.3	1258.7
0.405	0.303	850.4	846.7	841.8	0.9692	0.8600	0.7036	1350.4	1326.1	1297.2
0.501	0.205	858.8	854.7	849.5	1.2269	1.0202	0.8632	1376.4	1349.3	1312.3
0.602	0.101	867.1	861.6	856.4	1.5080	1.2035	0.9653	1424.2	1376.3	1360.9
0.702	0.000	881.1	876.9	872.0	2.0254	1.6714	1.3058	1442.1	1402.1	1387.0
Aniline + Cyclohexane + Ethyl methyl ketone										
0.000	0.703	817.3	812.3	807.9	0.5138	0.4672	0.4108	1195.9	1171.5	1138.5
0.100	0.606	826.7	822.0	817.2	0.6097	0.5429	0.4734	1241.3	1197.0	1154.4
0.202	0.504	835.1	830.3	825.9	0.6882	0.6179	0.5446	1286.7	1242.5	1175.6
0.302	0.401	843.8	839.4	834.2	0.8452	0.7489	0.6297	1355.5	1327.2	1267.8
0.403	0.304	850.8	846.0	841.2	0.9412	0.7876	0.6902	1380.5	1339.5	1293.8
0.501	0.199	858.8	853.6	849.1	1.2101	0.9675	0.8383	1410.6	1381.4	1327.2
0.605	0.101	864.7	860.9	855.0	1.4611	1.1786	0.9795	1450.5	1410.1	1354.8
0.700	0.000	874.1	869.9	864.8	1.8849	1.4387	1.1979	1465.3	1438.6	1375.2
Aniline + Cyclohexane + Iso butyl methyl ketone										
0.000	0.701	818.4	814.0	810.0	0.6171	0.5379	0.4924	1176.3	1162.0	1137.0
0.103	0.603	825.0	820.7	816.6	0.7213	0.6334	0.5628	1229.5	1207.1	1171.4
0.201	0.504	833.0	829.0	823.8	0.8249	0.6904	0.6126	1269.3	1238.8	1199.6
0.305	0.402	840.3	835.6	831.8	0.9757	0.8195	0.7023	1307.2	1277.3	1236.1
0.401	0.304	847.6	843.9	839.4	1.0383	0.8919	0.7627	1335.8	1303.4	1255.5
0.500	0.203	858.1	852.2	848.8	1.3159	1.0518	0.9071	1387.3	1361.1	1278.1
0.598	0.102	864.3	859.2	855.7	1.5323	1.2203	0.9725	1477.2	1414.8	1351.5
0.701	0.000	875.8	870.6	865.8	1.8381	1.4501	1.1889	1484.3	1429.9	1379.1

From Table-1, it is found that Ultrasonic Velocity ( $U$ ), Density ( $\rho$ ), Viscosity ( $\eta$ ) are increasing with increase in mole fraction of aniline. The increase in velocity, perhaps, is due to the structural changes occurring in the mixture, resulting in weakening of intermolecular forces. In addition, ultrasonic Velocity ( $U$ ) decreases with increase in temperature at all noted concentration. This is because, the increase in spacing between the molecules and the increase in entropy of its structural arrangement. The decrease in density and viscosity with temperature indicates the decrease in intermolecular forces due to increase in thermal vibrations of the system, which causes an increase in volume expansion and hence increase in free path length.

In all the cases values of the aliphatic ketones without aniline is much lower than that of the aniline without ketones. Which means, when mole fraction of aniline increases the increasing trend is observed in density, viscosity and velocity, it reveals that the addition of aniline increases the effective molecular space. The increased space is due to the substitution of a cyclic molecule (aniline) by replacing another cyclic molecule (cyclohexane). The observed trend may be due to the polar nature of the added components [13]. In the first concentration, a weak dipolar interaction is observed between ketones and cyclohexane as shown in fig a, from the second concentration onwards, aniline is purposefully introduced, because aniline is having a relatively higher dielectric constant [14,15] and basically electron donor nature, when it mixes with aliphatic ketones exhibits significant interactions like a dipole-dipole interaction and hydrogen bonding between participating molecules shown in fig b and fig c respectively.



From Table 2, it is found that the adiabatic compressibility ( $\beta$ ) and Free Length ( $L_f$ ) decrease with increasing concentration of aniline. There is a decreasing trend observed for adiabatic compressibility ( $\beta$ ) which is just the inverse to ultrasonic velocity ( $U$ ). The decrease in adiabatic compressibility ( $\beta$ ) shows the decrease in interaction between solute and solvent molecules with the decrease concentration of ketones, which shows a possible formation of complex between these two entities. This clustering between aniline and ketones molecule is broken up by the interacting molecule, cyclohexane. This lessened force of interaction between solute & solvent will consequently decrease free length in the mixture. It implies that, there is a significant interaction between solute and solvent molecule, which is found, decreased when the concentration of solute is lessened. On the other hand, if there is a decrease in free length ( $L_f$ ) with increase in ultrasonic velocity ( $U$ ), which is also in accordance with the expected decrease in compressibility following an increase in ultrasonic velocity ( $U$ ).

**Table- 2: Values of Adiabatic compressibility ( $\beta$ ) Free length ( $L_f$ ) and Free Volume( $V_f$ ) at 303K, 313K and 323K**

Mole fraction		Adiabatic compressibility ( $\beta$ ) ( $10^{-10} \text{ N}^{-1} \text{ m}^2$ )			Free length ( $L_f$ ) ( $10^{-10} \text{ m}$ )			Free Volume ( $V_f$ ) ( $10^{-7} \text{ m}^3 \text{ mol}^{-1}$ )		
$X_1$	$X_3$	303K	313K	323K	303K	313K	323K	303K	313K	323K
Aniline + Cyclohexane + Di methyl ketone										
0.000	0.701	8.9195	9.6864	10.387	5.8947	6.1429	6.3612	2.3085	2.5946	3.2816
0.100	0.602	8.4192	8.9155	9.8455	5.7270	5.8934	6.1931	2.0008	2.2891	2.5442
0.204	0.501	7.6749	8.2418	9.0411	5.4680	5.6663	5.9347	1.6574	1.9250	2.2368
0.303	0.404	6.7631	7.0517	7.5663	5.1329	5.2413	5.4291	1.3972	1.8071	1.9973
0.405	0.303	6.4484	6.7160	7.0594	5.0120	5.1150	5.2441	1.2470	1.4519	1.8982
0.501	0.205	6.1465	6.4264	6.8351	4.8933	5.0035	5.1602	0.9737	1.2465	1.5362
0.602	0.101	5.6857	6.1265	6.3042	4.7063	4.8854	4.9557	0.8118	1.0819	1.4808
0.702	0.000	5.4572	5.8006	5.9605	4.6108	4.7536	4.8187	0.5800	0.7417	1.0568
Aniline + Cyclohexane + Ethyl methyl ketone										
0.000	0.703	8.5550	8.9698	9.5484	5.7730	5.9113	6.0989	2.6145	2.9235	3.3972
0.100	0.606	7.8505	8.4897	9.1818	5.5302	5.7509	5.9807	2.2171	2.4988	2.9064
0.202	0.504	7.2328	7.8013	8.7598	5.3081	5.5128	5.8417	2.0231	2.2565	2.5101
0.302	0.401	6.4505	6.7632	7.4576	5.0129	5.1330	5.3900	1.6715	1.9418	2.3514
0.403	0.304	6.1676	6.5872	7.1017	4.9017	5.0657	5.2598	1.5166	1.8938	2.1913
0.501	0.199	5.8519	6.1391	6.6856	4.7746	4.8904	5.1034	1.1191	1.5170	1.7714
0.605	0.101	5.4968	5.8411	6.3714	4.6275	4.7702	4.9820	0.9162	1.2123	1.5069
0.700	0.000	5.3284	5.5545	6.1142	4.5560	4.6517	4.8805	0.6615	0.9650	1.1871
Aniline + Cyclohexane + Iso butyl methyl ketone										
0.000	0.701	8.8313	9.0980	9.5496	5.8655	5.9534	6.0993	2.7337	3.2984	3.6451
0.103	0.603	8.0186	8.3612	8.9241	5.5891	5.7072	5.8962	2.2877	2.7048	3.0869
0.201	0.504	7.4509	7.8602	8.4342	5.3876	5.5336	5.7321	1.9410	2.4442	2.7869
0.305	0.402	6.9639	7.3350	7.8674	5.2085	5.3455	5.5361	1.5610	1.9587	2.3505
0.401	0.304	6.6122	6.9744	7.5571	5.0753	5.2124	5.4258	1.4542	1.7607	2.1050
0.500	0.203	6.0555	6.3333	7.2119	4.8570	4.9671	5.3005	1.0664	1.4504	1.6478
0.598	0.102	5.3025	5.8145	6.3973	4.5449	4.7593	4.9922	0.9234	1.2179	1.5985
0.701	0.000	5.1825	5.6173	6.0728	4.4932	4.6779	4.8639	0.7004	0.9452	1.2059

From Table-2 is also found that there is a decrease in free volume, with decrease in concentration of ketones which is depicted in table 3. The values of free volume also reported to be a temperature dependent. Carbonyl group in ketones, being polar, results in intermolecular H-bonding between H of the aniline and O of carbonyl ketones. Because of the H-bonding, the molecules must be arranged such that the void spaces may be available, due to which the solute is more compressible. On the other hand cyclohexane is non-polar, having no possibility of forming H-bond.

Table-3 is evident of the value of internal pressure increases with decrease in mole fraction of ketones, the increase in internal pressure may be due to the strengthening of cohesive force. When the temperature is increased, there is a tendency for the solute molecules to move away from each other, reducing the interactions, which may further reduce the cohesive forces. As it is known that pressure of the system is inversely proportional to the volume of the same, this

decrease in internal pressure with increase temperature leads to increase in free volume simultaneously.

**Table- 3: Internal pressure ( $\pi_i$ ), Viscous Relaxation Time ( $\tau$ ) and Gibb's Free Energy ( $\Delta G$ ) at 303K, 313K and 323K**

Mole fraction		Internal pressure ( $\pi_i$ ) ( $10^{-6}$ Pa)			Viscous Relaxation Time ( $\tau$ ) ( $10^{-12}$ s)			Gibb's Free Energy ( $\Delta G$ ) ( $10^{-20}$ KJ mol $^{-1}$ )		
$X_1$	$X_3$	303K	313K	323K	303K	313K	323K	303K	313K	323K
Aniline + Cyclohexane + Di methyl ketone										
0.000	0.701	3.8933	3.7281	3.4130	0.5536	0.5354	0.4776	0.2272	0.2345	0.2259
0.100	0.602	4.0045	3.8143	3.6546	0.6131	0.5784	0.5697	0.2457	0.2489	0.2601
0.204	0.501	4.1712	3.9520	3.7403	0.6900	0.6491	0.6174	0.2672	0.2706	0.2756
0.303	0.404	4.3175	3.9458	3.8026	0.7554	0.6518	0.6333	0.2836	0.2714	0.2806
0.405	0.303	4.3591	4.1316	3.7638	0.8333	0.7701	0.6622	0.3014	0.3027	0.2892
0.501	0.205	4.6031	4.2259	3.9255	1.0054	0.8741	0.7866	0.3355	0.3264	0.3225
0.602	0.101	4.7578	4.3053	3.8619	1.1432	0.9831	0.8113	0.3588	0.3485	0.3285
0.702	0.000	5.1745	4.7520	4.2072	1.4737	1.2926	1.0377	0.4049	0.3998	0.3762
Aniline + Cyclohexane + Ethyl methyl ketone										
0.000	0.703	3.3605	3.2245	3.0560	0.5860	0.5587	0.5229	0.2375	0.2425	0.2435
0.100	0.606	3.5208	3.3704	3.1923	0.6381	0.6145	0.5795	0.2529	0.2603	0.2634
0.202	0.504	3.5962	3.4544	3.3221	0.6636	0.6427	0.636	0.2601	0.2687	0.2814
0.302	0.401	3.7922	3.5949	3.3587	0.7269	0.6753	0.6461	0.2766	0.278	0.2844
0.403	0.304	3.8751	3.585	3.4019	0.7739	0.6917	0.6535	0.2879	0.2825	0.2866
0.501	0.199	4.2379	3.8138	3.6089	0.9441	0.7919	0.7472	0.3241	0.3079	0.3126
0.605	0.101	4.4689	4.0587	3.7575	1.0708	0.9179	0.8321	0.3469	0.3356	0.3334
0.700	0.000	4.9268	4.3302	4.0254	1.3391	1.0655	0.9765	0.3876	0.3636	0.3644
Aniline + Cyclohexane + Iso butyl methyl ketone										
0.000	0.701	2.8437	2.6616	2.5660	0.7266	0.6525	0.6269	0.2765	0.2716	0.2786
0.103	0.603	3.0481	2.8726	2.7396	0.7711	0.7061	0.6696	0.2873	0.2864	0.2914
0.201	0.504	3.2561	3.0057	2.8650	0.8195	0.7235	0.6889	0.2934	0.2909	0.2969
0.305	0.402	3.5379	3.2679	3.0658	0.9059	0.8014	0.7367	0.3166	0.3101	0.3098
0.401	0.304	3.6595	3.4235	3.2142	0.9154	0.8293	0.7685	0.3185	0.3166	0.318
0.500	0.203	4.1125	3.6948	3.5315	1.0624	0.8881	0.8722	0.3455	0.3294	0.3425
0.598	0.102	4.3543	3.9549	3.6024	1.0833	0.946	0.9295	0.3491	0.3413	0.3548
0.701	0.000	4.8397	4.3623	4.0072	1.2701	1.0861	0.9626	0.3779	0.3672	0.3616

In addition free volume and internal pressure are behaving opposite to each other, since the lone pair electrons in aniline are dominant in dipole-dipole interaction, when aniline in the mixture leads to a compact structure due to the presence of dipolar type interaction. This contributes to decrease in free volume and this can easily form complex structures so that increasing trend observed for internal pressure [16].

The viscous relaxation time ( $\tau$ ) increases with increasing concentration of aniline and decrease with increase in temperature and in mole fraction of ketones at any concentration. The increase in viscous relaxation time ( $\tau$ ) and Gibb's free energy value confirms the availability of intermolecular interaction. The reduction of  $\Delta G$  indicates the need for smaller time for the



cooperation process of the rearrangement of the molecules in the mixtures, decreases the energy with increase in temperature leads to dissociation [17, 18].

## CONCLUSION

The ultrasonic studies provide a comprehensive investigation of molecular association between aniline with Dimethylketone, Ethyl methyl ketones, Iso-butyl methyl ketones in cyclohexane arising from the dipole-dipole and H-bonding between the solute and solvent molecules and the order of interaction is found to be Dimethylketone > Ethyl methyl ketones > Iso-butyl methyl ketones.

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