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MOLECULAR INTERACTION ON BENZYL ALCOHOL WITH FORMAMIDES IN CARBON TETRACHLORIDE - DIELECTRIC RELAXATION STUDIES

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ABSTRACT

Molecular interaction on benzyl alcohol with formamides (formamide, N-methylformamide and N, N–dimethylformamide) in carbon tetrachloride for various molar ratios over the frequency at 9.36 GHz in 298K using X-band microwave bench technique (dielectric relaxation studies) were studied. The dielectric parameters like ε_0, ε_α, ε', and ε'' are calculated. In addition, the dielectric relaxation times ($\tau_{(1)}$, $\tau_{(2),}$ and $\tau_{(0)}$) have been calculated by employing Higasi's method and the activation energies ($\Delta F\tau$ and $\Delta F\eta$) have been determined using dielectric data. The most likely association between free hydroxyl groups of benzyl alcohol with the carbonyl group of formamides was studied. The result shows that the proton accepting ability of formamides is decreasing in the order: N-methylformamide> formamide> N, N-dimethylformamide and play an important role in determination of the strength of the hydrogen bond [O-H···C=O] is formed.

Key words: Molecular interaction, Hydrogen bonding, Benzyl alcohol, Amides, Dielectric relaxation

1. INTRODUCTION

The broad classifications of the methods to study H-bonding are chemical, crystallographic, spectroscopic and theoretical. The chemical methods are mainly concerned with the thermodynamic properties like enthalpy, entropy, heat of mixing etc., The crystallographic studies are mostly related to the conformation of the structure in H-bonded systems, by X-ray, neutron diffraction and electron diffraction methods. The spectroscopic effects consider the physical aspects of H-bonding like molecular potentials, barrier to internal rotation, the vibrational energies of the bonds which are modified by the potential field etc., The hydrogen bonds constitute a very interacting class of molecular interactions, which are of extreme fields of chemistry and molecular biology. The dielectric investigation of hydrogen–bonded compounds in non-polar solvent provides valuable information regarding molecular complex formation in solution. The study of the H-bonds of the types (O-H···C=O) occupies of position of importance as it relates to the study of biopolymers. Recently, dielectric relaxation behavior of mixtures of polar molecules under varying conditions of complexation temperature and environment factors has evoked considerable interest (Malathi et al., 2003).

They have often proved to be a powerful tool to give insights into the mechanisms of association and reorientation dynamics of dipolar liquids (Barthel and Gores 1994), (Grant et al., 1978), (Pethig 1979). Dielectric studies play a vital role in understanding the interaction between the components of a liquid mixture. The dielectric study includes dielectric relaxation, dielectric polarization and dipolar incremental. The dielectric relaxation study of solute-solvent mixtures in the microwave frequency range gives information about the molecular polarization of the system. This is related to the formation of multimers in dipoles and their rotations. The formation of hydrogen bonds leads to associate with restricted number of molecules called multimers. The dynamics properties of such liquids are often interpreted in terms of the duration of life and the motions of the multimers. At least three types of multimers can be occur in the case of monosubstituted amides namely the α-multimers, β-multimers and γ- multimeters. The relaxation times of such species are far and wide. When polar molecules are subjected to electromagnetic waves in microwave frequency region, they absorb considerable microwave energy there by perturbing dipole moment of molecules due to molecular rotations. The study of dielectric relaxation of polar liquids in non-polar, solvents from the microwave absorption studies gives valuable information about various types of the molecular associations present in the solutions as microwave can

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detect weak molecular interactions. Dielectric relaxation study can give precise information on the formation of H-bonded complex when a proton donor is mixed with a proton acceptor in non-polar solvent.

In our present investigation, the mixed solvents have been used a wide range of chemical, biological pharmaceutical, industrial, biophysics, condensed matter physics, and laboratory applications. Amides represent an important class of organic solvent due to their high polarity, strong solvating power and a large liquid range, which makes than industrial and technological important solvents and the amide linkage (-CO-NH-) is an important functional group in chemistry, pharmaceutical and material science (Greenberg et al.,2002) (Barthel and Gores 1994). They are the simplest molecules containing a peptide linkage and a study of their hydrogen bonding ability yields insight into the nature of protein structure (Jeffrey and Saeriger 1991). Amides have been the subject of extensive spectroscopic, physical, theoretical investigations to study intermolecular hydrogen bonding. The primary amides like formamide and acetamide are excellent proton donors as well as proton acceptors and hence are strongly associated through intermolecular hydrogen bond. Consequently, they exhibit physical properties which show strong dependence on the solvent environment, temperature and concentration. The secondary amides like as N-methylacetamide and N-methylformamide are also exhibit similar properties. On the other hand, the tertiary amides like N, N-dimethylformamide and N, N dimethylacetamide are poorly self associated and are not very sensitive to the choice of solvent. They are widely used as organic solvents, starting materials or intermediates in different industries (Synthetic polymers, manufacture of dyes, and synthesis of pesticides) (Manahan 1994) (Finlayson – Pitts and Pitts 1999) (Carter1994).

In this present paper, the proton acceptors like as formamide, N-methylformamide and N, N-dimethylformamide. Formamide is used as a solvent, softner, and chemical intermediate, and primarily for manufacturing sulfa drugs, synthesizing vitamins, softner for paper and fiber. N-methylformamide is mainly used as a reagent in various organic syntheses with limited applications as a highly polar solvent, precursor to methyl isocynamide, a ligand in coordination chemistry and an intermediate in the production of some pharmaceutical compounds. N, N-dimethylformamide is primarily used as an industrial solvent, and is used to process polymer fibers, films, and surface coating, to permit easy spinning of acrylic fibers, to produce wire enamels, and as a crystallization medium in the pharmaceutical industry. In addition, we used benzyl alcohol as a proton donor. It is used as a general solvent for ink, paints, lacquers and epoxy resin coatings, in soap, perfume, and flavor industries, dielectric solvent for the dielectrophoretic reconfiguration of nanowires.

Many workers have studied the dielectric behavior of alcohols and amides mixture with structure breaking and structure making properties. To detect the solute- solvent interactions through measurement of relaxation time in first time (Purcell and Smyth 1963). The dielectric relaxation data for N, N-substituted amides showed that these liquids in dilute non-polar solvents obey the simple Debye equation have been studied that the relaxation behavior of DMF with methanol in benzene solution by using dielectric method and the results indicate that the dielectric relaxation time is maximum at equimolar concentration (Sharma and Sharma 1985). Dielectric relaxation studies of dilute solutions of amides in 1,4–dioxane/benzene have been measured at 308 K using 9.37 GHz by using dielectric relaxation set up. This investigation reported that the relaxation behavior of amides in non-polar solvent, solute-solvent and solute-solute type of molecular association is proposed (Malathi et al., 2004). Dielectric studies of binary mixtures of ethyl alcohol with DMF in benzene at 9.88 GHz using standing microwave techniques and Gopala Krishna’s single frequency concentration variation method at different temperatures. This reported shows that the dielectric process can be treated as the rate process like the viscous flow process (Vimal Sharma et al., 2008). Finally the dielectric behavior of three amines with acrylonitrile at 9.35 GHz has been studied of 303K, it show that maximum relaxation time values at 1:1 complex ratio by both Higasi's and cole–cole method (Kalaivani et al., 2009).

The aim of the present investigation deals with dielectric behavior of benzyl alcohol with formamides (formamide, N-methylformamide and N, N–dimethylformamide) in CCl_4 solution in 9.36 GHz by using X-band microwave technique at 298 K.

2. EXPERIMENTAL

2.1. *Materials*

AnaloR variety of benzyl alcohol was purchased from sigma-Aldrich Bangalore, India. The formamides (formamide, N-methylformamide and N, N-dimethylformamide) and carbon tetrachloride were obtained from Sd-fine chemicals Limited, Mumbai, India. The above chemicals were used without further purification. The physical parameters of the entire chemicals are used to check against their literature.

2.2. *Methods*

The dielectric loss ($\varepsilon^{'}$) and dielectric loss at microwave frequency ($\varepsilon^{''}$) were measured using X-band microwave bench supplied by Vajenthura microwave products, Chengalpattu, India. This instrument was calibrated by using some standard liquids to know dielectric constant and dielectric loss ($\varepsilon^{'}$ and $\varepsilon^{''}$). The accuracy of $\varepsilon^{'}$ and $\varepsilon^{''}$ at 1% and 5% determination respectively. The commercial instrument “WTM dipolemeter DMOI” supplied by Wiser

Chaiftlich Technische Werkstatter, Germany operated at 220V and the dipolemeter based on the principle of heterodyne beat method was used for the measurement of the static permittivity (ε_α) at 298K. Refractive indices of all systems were measured by an Abbe's refractrometer, which in turn gives the static permittivity (ε_α) with an accuracy of ± 0.02%. The percentage deviation in the measurement of refractive index is about ± 0.02%.

2.3. *Preparation of solutions*

The proton donor (benzyl alcohol) and the proton acceptors (formamide, N-methylformamide and N, N-dimethyformamide) under study were separately dissolved at the same molar concentration ($0.3 mol^{-1}$) in carbon tetrachloride. The solutions were mixed in different proportions (3:1, 2:1, 1:1, 1:2, and 1:3). But with the total concentration was kept of a fixed value and were subjected to dielectric constant measurements. The maximum deviation for dielectric constant for all the system studied occurs at equimolar ratio of the solvent.

2.4. *Evaluation of dielectric parameters*

The values of dielectric parameters (ε_0, ε', ε'' and ε_α) are defined as following

$$\left.\begin{aligned} \varepsilon_0 &\Rightarrow \varepsilon_1 + a_0 W_2 \\ \varepsilon' &\Rightarrow \varepsilon_1 + a' W_2 \\ \varepsilon'' &\Rightarrow a''/W_2 \\ \varepsilon_\alpha &\Rightarrow \varepsilon_{1\alpha} + a_\alpha W_2 \end{aligned}\right\} \rightarrow (1)$$

In which subscript 1 refers pure solvent, 2 refers to the solute, 0 refers to the static frequency and α refers to the infinite or optical frequency measurements and W_2 is the weight fraction of the solute.

According to the method (Higasi et al., 1971), the dielectric relaxation times ($\tau_{(1)}$, $\tau_{(2)}$ and $\tau_{(0)}$) are given by

$$\tau_{(1)} \Rightarrow \frac{a''}{\omega(a' - a_\alpha)} \rightarrow (2)$$

$$\tau_{(2)} \Rightarrow \frac{a_0 - a'}{\omega a''} \rightarrow (3)$$

$$\tau_{(0)} \Rightarrow \sqrt{\tau_{(1)} \times \tau_{(2)}} \rightarrow (4)$$

Where, $\tau_{(1)}$ is the dielectric relaxation time due to the individual molecular rotation and $\tau_{(2)}$ is the dielectric relaxation time due to the overall molecular rotations and $\tau_{(0)}$ is the most probable relaxation time.

The molar free energy for the dielectric relaxation ($\Delta F\tau$) and the viscous flow process ($\Delta F\eta$) was using the equations given by (Glasstone et al., 1941).

$$\tau \Rightarrow \frac{h}{KT} \exp\left[\frac{\Delta F\tau}{RT}\right] \rightarrow (5)$$

$$\eta \Rightarrow \frac{Nh}{V} \exp\left[\frac{\Delta F\eta}{RT}\right] \rightarrow (6)$$

Where, h is the Plank's constant, K is the Boltzmann's constant, N is the Avogadro's number, and V is the molar volume.

3. RESULTS AND DISCUSSION

The systems selected were binary and ternary mixtures of proton donor (benzyl alcohol) with proton acceptors (formamide, N-methylformamide and N, N-dimethylformamide) in dilute solution of carbon tetrachloride of various molar ratios (ie, 1:0, 3:1, 2:1, 1:1, 1:2, 1:3 and 0:1) at 9.36 GHz in 298K. The dielectric parameters like ε_0, ε', ε'' and ε_α are calculated and the dielectric relaxation times ($\tau_{(1)}$, $\tau_{(2)}$ and $\tau_{(0)}$)are calculated by Higasi's method and activation energies (($\Delta F\tau$) and ($\Delta F\eta$)) for all the systems studied are given the table 1 and 2.

Tables reveals that the dielectric parameters, dielectric relaxation times and the activation energies are higher value for binary mixture of benzyl alcohol with CCl_4 (1:0) ratio than that of formamides (formamide, N-methylformamide and N, N-dimethylformamide) (0:1 ratio) system. This statement suggests that the benzyl alcohol molecules are heavier than that formamides molecule. The ternary systems of benzyl alcohol with formamides are significantly higher than that of binary systems because the hydrogen bond formed between hydrogen atom in the O-H group of benzyl alcohol molecules and oxygen atom in the C=O group of amides molecules (Malathi et al., 2003). The figure (1-3) shows that the molecular interaction between O-H group of benzyl alcohol and C=O group of formamides (formamide, N methylformamide and N, N-dimethylformamide).

The dielectric constant (ε_0) is the one of the important factor in the dielectric relaxation study. The dielectric constant of monomethyl amides is found to be considerably higher than those of the unsubsituted amides. This can be explained as due to the fact that the monosubsituted amides are associated as chain polymers, the two amino hydrogen's in the unsubstituted amides permit a larger variety of associated forms, even if it be assumed that only one hydrogen atom of the amino group is involved in hydrogen bonding at any given time. The possible associated forms range from the extended type of increased moment, involving the trans hydrogen atom through intermediate random forms which would involve a considerable degree of mutual cancellation among the concentrations made by each molecule to the net moment of the polymeric group and finally to a dimer of small or zero moment. The observed dielectric constant is largely determined by the composite effect of all of these associated groups, and hence is smaller for the unsubstituted amide. In the dimethyl amides, association through hydrogen bonding is no longer possible, and hence a large decreased in dielectric constant occurs (Gordon Leader and James Gormadey 1951). Finally we expects the dielectric constant (ε_0) value of primary amide is much higher than tertiary amide which is mainly due to small size of primary amide molecules and the formation of large range H-bonded linear structures Via strong interactions between C=O and N-H groups. The primary amide molecules possess three hydrogen bond donors and three acceptors that help informing strong H-bonded molecular connectivity with parallel dipolar ordering. In the formation the enolate ion is favorable as $-CH_3$ present on C=O group shows hyper conjugation which promotes the self-association of tertiary amide molecules through H-bonded formation, tertiary amide shows the absence of H-bonded intermolecular association whereas of tertiary amide have comparatively low. The ε_0 value of primary

Table 1. The dielectric parameters of benzyl alcohol with formamides in carbon tetrachloride system at 298K.

ε_1 = 2.218 and $\varepsilon_{1\alpha}$ = 2.0851

System	Ratio benzyl alcohol: Formamide	Dielectric constant (ε_0)	Dielectric constant at static permittivity (ε_α)	Dielectric loss (ε')	Dielectric loss at microwave frequency (ε'')
N-methyl formamide + benzyl alcohol	1:0	2.6593	2.1101	2.4803	**0.1762**
	3:1	2.8146	2.1213	2.4451	**0.1861**
	2:1	2.8247	2.1286	2.4421	**0.1922**
	1:1	2.8938	2.1389	2.4060	**0.2141**
	1:2	2.8142	2.1272	2.4438	**0.1841**
	1:3	2.8021	2.1213	2.4486	**0.1840**
	0:1	2.6001	2.1081	2.4890	**0.1386**
Formamide + benzyl alcohol	1:0	2.6593	2.1101	2.4803	**0.1762**
	3:1	2.7249	2.1200	2.4630	**0.1829**
	2:1	2.7488	2.1256	2.4570	**0.1915**
	1:1	2.8043	2.1359	2.4153	**0.1989**
	1:2	2.7414	2.1213	2.4603	**0.1844**
	1:3	2.7145	2.1193	2.4699	**0.1818**
	0:1	2.5190	2.1012	2.4913	**0.1269**
N, N-methyl formamide + benzyl alcohol	1:0	2.6593	2.1101	2.4830	**0.1762**
	3:1	2.7045	2.1110	2.4690	**0.1803**
	2:1	2.7245	2.1114	2.4687	**0.1864**
	1:1	2.7624	2.1138	2.4207	**0.1956**
	1:2	2.6794	2.1112	2.4807	**0.1828**
	1:3	2.6609	2.1108	2.4810	**0.1783**
	0:1	**2.4017**	**2.0921**	**2.5016**	**0.0852**

Table: 2. Dielectric relaxation times and activation energies of benzyl alcohol with formamides in carbon tetrachloride system at 298K.

System	Ratio benzyl alcohol: formamide	Relaxation time τx10^{-12} (PS)			Free energy of activation ($\Delta F\tau$) KJ/mole	Viscous flow ($\Delta F\eta$) KJ/mole
		$\tau_{(1)} \times 10^{-12}$ (PS)	$\tau_{(2)} \times 10^{-12}$ (PS)	$\tau_{(0)} \times 10^{-12}$ (PS)		
N-methyl formamide + benzyl alcohol	1:0	11.4547	15.6006	13.3678	11.1743	**12.6610**
	3:1	15.2301	30.9666	21.7169	12.3969	**13.2969**
	2:1	16.4460	31.7307	22.8439	12.5244	**13.7244**
	1:1	24.8823	35.5408	29.7378	13.1889	**14.0889**
	1:2	15.9158	30.8424	21.1559	12.4474	**13.3474**
	1:3	14.7880	29.9459	21.0437	12.3176	**13.2176**
	0:1	08.7246	12.4983	10.4423	10.5520	**12.2743**
Formamide + benzyl alcohol	1:0	11.4547	15.6006	13.3678	11.1743	**12.6610**
	3:1	13.5864	22.3339	17.4194	11.8413	**13.0413**
	2:1	15.0512	25.9033	19.7452	12.1576	**13.1576**
	1:1	21.2174	30.5040	25.4404	12.7956	**13.8956**
	1:2	13.9611	23.7706	18.2171	11.9542	**13.1642**
	1:3	13.0322	20.9930	16.5340	11.7099	**12.9099**
	0:1	07.8847	11.4141	09.4866	10.3102	**11.5102**
N, N-methyl formamide + benzyl alcohol	1:0	11.4547	15.6006	13.3678	11.1743	**12.6610**
	3:1	12.5001	20.3649	15.9550	11.6201	**12.5201**
	2:1	12.9267	21.4017	16.6329	11.7255	**12.8451**
	1:1	17.5632	27.2462	21.8753	12.4152	**13.5491**
	1:2	12.0537	16.9485	14.2931	11.3430	**13.0412**
	1:3	11.7221	15.7370	13.5820	11.2144	**12.4141**
	0:1	**04.7683**	**11.2414**	**07.3213**	**09.5047**	**11.3214**

amide is in between the ε_0 values of secondary and tertiary amide (Sengwa et al., 2009).

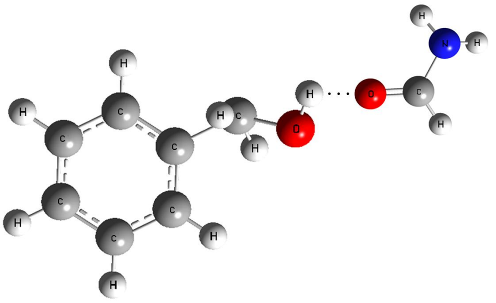


Figure.1. Molecular interaction between benzyl alcohol and formamide

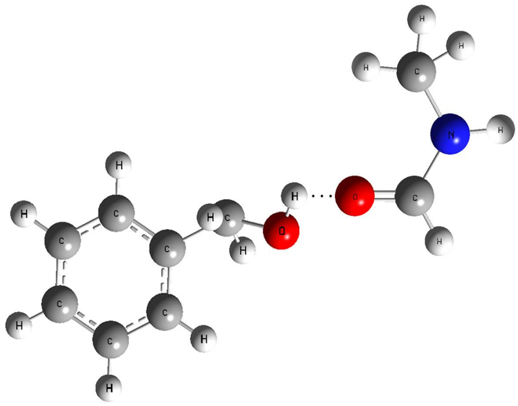


Figure .2. Molecular interactions between benzyl alcohol and N-methylformamide

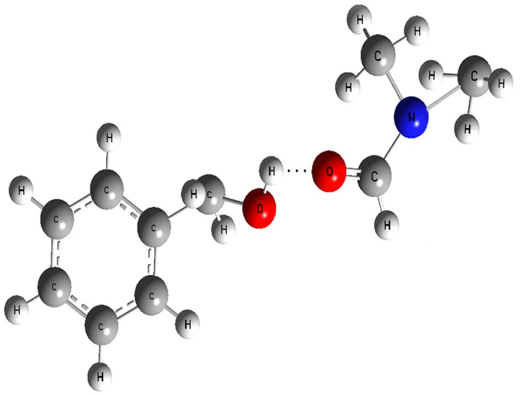


Figure. 3. Molecular interactions between benzyl alcohol and N, N-dimethylformamide

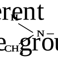
Table 2 shows that the values of $\tau_{(2)}$ is significantly higher than the value of $\tau_{(1)}$ and $\tau_{(0)}$ for the binary and ternary mixtures of benzyl alcohol with amides (formamide, N-methylformamide and N, N-dimethylformamide) in CCl_4 for all the studied systems. The results of above statement suggest that the contribution of intermolecular or overall molecular relaxation $\tau_{(2)}$ is longer than that the individual molecular relaxation $\tau_{(1)}$ and the most probable molecular rotation $\tau_{(0)}$. The increase in relaxation time may be due to the increase in effective radius of the rotating unit. This may be due to the fact the CH_3 group attached to the same carbon atom to which the OH group is attached. This increases the size of the molecule and may cause hindrance to the group rotation, which may give rise to increased relaxation time (Kalaivani et al., 2009) & (Manjunath and Sannappa 2008).

The values of viscous flow ($\Delta F\eta$) are greater than that of free energy of activation ($\Delta F\tau$) for all the systems are studied. This is in agreement with the fact that the processes of viscous flow which involve both the rotational and translational form of motion, faced greater inference from neighbors than the dielectric relaxation takes place by rotational only (Malathi et al., 2003). Further the values of dielectric relaxation times ($\tau_{(1)}$, $\tau_{(2)}$, and $\tau_{(0)}$) and activation energies ($\Delta F\tau$ and $\Delta F\eta$) show a larger values for the 1:1 molar ratio of benzyl alcohol with formamides (formamide, N-methylformamide and N,N-dimethylformamide) compared to the other higher molar ratios (ie, 3:1, 2:1, 1:2 and 1:3]. The above results suggest that the 1:1 complex formation is predominant in these systems (Kalaivani et al., 2009).

In this present investigation reports that, the dielectric relaxation times and activation energies of the proton donor (benzyl alcohol) and proton acceptors (formamide, N-methylformaide and N, N-dimethylformamide) in carbon tetrachloride at 298K by using X- band microwave bench at 9.36 GHz. The observed result are indicates that the proton accepting ability of the systems are in the decreasing order: N-methylformamide>formamide> N, N-dimethylformamide. This indicates the proton accepting ability of N-methylformamide with benzyl alcohol system is significantly higher than that of the formamide and N, N-dimethylformamide with benzyl alcohol systems.

The above mentioned order is formed due to the following factors; they are

(i) The formation of hydrogen bonds leads to associate with restricted number of molecules called multimers. The dynamic properties of such liquids are often interpreted in terms of the duration of life and the motions of the multimers. At least three types of multimers can occur in the case of monosubstituted amides namely the α-multimers, β-multimers and γ-multimers. The relaxation times of such species are far and wide. So this above reason the N-methylformaide with benzyl alcohol system is more predominant than other systems (Malathi et al., 2003).

(ii) The dielectric relaxation model and comparative study, among all the system N- methylformamide (NMF) shows a very different behavior. This is due to the asymmetric of the  group. The addition of two –CH_3 groups with N-group leads to reduction in the correlation factor (Kumbharkhane et al., 1991).

(iii) The proton accepting ability of secondary amides is higher than that of tertiary amides. This is probably due to the electron contribution of the methyl group to the C=O group is significantly greater than that of the dimethyl group and the higher value of relaxation time for secondary amides with alcohols systems may probably be due to comparatively large inductive effect (Garcia et al., 1997).

(iv). The micelle formations in tertiary amide could be due to the effects like ion-solvent interactions hydrogen bonding and dispersion forces even though the dielectric constant of tertiary amide is much lower than primary and secondary amide. It could be suggested that the micelle formation in tertiary amide is due to the orientation the opposite end of the solvent dipoles, which cause ion-solvent interaction. It could also be suggested that in tertiary amide the ability of nitrogen to participate in resonance with the carbonyl group is more likely than secondary amide and primary (Salim Akhter et al., 1999).

(v). The normal behavior of the dimethylamides can be accounted for by the absence of the amino hydrogen’s for the bonding to oxygen’s of neighboring molecules; the moderate increases for formamide suggest a variety of associated forms with either or both of the two amino hydrogen and the very effects in N-methylamides result from chain wise association by hydrogen bonding with the single amino hydrogen such that adjacent molecular dipole moments are nearly parallel (Bass et al., 1964).

Finally, the observed values of this investigation suggest that the proton accepting ability of formamides varying with substitution of methyl group, it concluded that the N-methylformamide has higher proton accepting ability than that of formamide and N, N-dimethylformamide and is in the order: N-methylformamide>formamide> N, N dimethylformamide has briefly explained by the above factors. Therefore the strongest intermolecular hydrogen bond formed between O-H group of benzyl alcohol and C=O group of N-methylformamide are higher than that of O-H group of benzyl alcohol and C=O group of formamide and weakest between the O-H group of benzyl alcohol and C=O group of N, N-dimethylformamide.

4. CONCLUSION

In our present investigation concludes that the hydrogen bonded complexes of benzyl alcohol with formamides (formamide, N-methylformamide and N, N-dimethylformamide) have been studied in carbon tetrachloride using dielectric relaxation method. It may concluded that the proton accepting ability of formamides is varying with substitution of methyl group and provide valuable information of solute-solute interaction system of the specified complexes.

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