

Investigation of Temperature Dependent Dielectric Relaxation Studies of 4-Bromoacetanilide in Dilute Solution of Carbon Tetrachloride

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The changes in dielectric parameters of 4-bromoacetanilide in dilute solution of carbon tetrachloride with temperature variation are studied at a fixed frequency of 9.27 GHz. The investigation are being done for five different mole fractions of 4-bromoacetanilide in the microwave region. The values of dielectric constant (ϵ') and dielectric loss (ϵ'') are determined by using Heston *et al.* method. Permittivity at a static frequency (ϵ_0) and at an optical frequency (ϵ_∞) is calculated with the help of dipole meter and Abbe's refractometer, respectively. Higasi's method is used to calculate the values of relaxation times (τ_1 , τ_2 and τ_0). From these values, it is found that the relaxation time decreases systematically with the increase in temperature. The fall of τ_2 with the increase in temperature has been observed to be more remarkable in comparison to the value of τ_1 . It indicates that the rate of falls of the relaxation time related to intramolecular rotation with temperature is faster as compared to the internal group rotation.

Keywords: 4-Bromoacetanilide, Dielectric parameters, Relaxation time.

INTRODUCTION

At microwave frequency, the studies of dielectric parameters of polar compounds with non-polar solvents give an appropriate scope to understand the existence of solute-solute and solute-solvent interactions and self association in solution because microwaves have the capacity to detect the weaker molecular interaction [1]. Various researchers [2-5] had done such dielectric relaxation studies of polar molecules in non-polar solvents using microwave absorption techniques.

These studies are important to understand the molecular structure and molecular forces. The non-polar solvents reduce the internal field and change the viscosity to alter the relaxation time of the solute molecule. The dielectric relaxation studies of pure solutions provide meaningful information about molecular interaction present in systems in the microwave region. An investigation of dielectric properties of polar compounds consisting of one associative and other non-associative liquid provides valuable information about the molecular complex formation in the mixture. There are several factors like the molecular structure, temperature, *etc.* which may affect the strength of the molecular association [6]. The existence of hydrogen bond

provides noticeable changes in the dielectric properties of liquid mixtures, therefore the molecular interactions of such compounds in liquid mixtures can be strongly influenced by the aggregation phenomena. For mixtures, this phenomena help to understand some physical properties like refractive index, dielectric permittivity, density and molar volume.

The studies of dielectric relaxation of polar compounds with non-polar solvents may also help to verify that whether polar complex presence in a liquid mixture or not. When mixture of some polar compound dissolved in the dilute solutions of a non-polar solvent, its resultant complex dielectric constant will be the weighted sum of the complex dielectric constant of individual components [7,8] when dielectric constant, dielectric loss and relaxation time depends on the concentration of the components linearly, then there is no complex formation of inter association in a mixture. The development of polar complexes leads to relaxation times, significantly larger than those for uncomplex molecules [9]. Schallamach [10] has been noticed that there is a single relaxation time for a binary liquid mixture in which both components are either associated or non-associated but for a binary liquid mixture in which one component is associated and the other is non-associated, he

observed two distinct relaxation times. For polar solute system, the dipole moment values are determined and to minimize the dipole-dipole interaction the solution are made sufficiently dilute. The analysis of dielectric relaxation phenomena extends the major view point to investigate more about the structural behaviour of polar molecules in different non-polar solvents [11].

Determination of dielectric parameters and their variation with temperature can help in explaining the structural behaviour of the polar compounds. The relaxation studies of some biologically and industrially useful [12-16] polar molecules are of great interest as it provides proper information regarding the relaxation process in polar solutions and mixtures. This study can help to understand the nature of the molecular orientation process. Vishwam *et al.* [17] had also studied the dielectric behaviour of some hydrogen bonded polar liquid mixtures.

Subramanian and Satish [18] calculated the values of dielectric parameters (ϵ' , ϵ'' , ϵ_0 and ϵ_∞) to explore the dielectric behaviour of isobutanol and octanol with dibutyl phthalate at a fixed frequency 9.36 GHz at three different temperatures *viz.* 303, 308 and 313 K. They observed the linear variation of these dielectric parameters with the concentrations of mixture. They calculated the values of relaxation time (τ) using two different methods, named as: (i) Higasi's method and (ii) Cole-Cole method and compared their results. The values of dipole moment were also determined at different temperatures which helped to understand the molecular structure. The values of dielectric relaxation time (τ) and dipole moment (μ) of binary mixtures of *N*-methylformamide (NMF) and dimethyl sulphoxide (DMSO) in dilute solution of benzene were determined by Singh [19] at a fixed wavelength with temperature variation. He found that the values of relaxation time decrease with the increase in temperature, whereas dipole moment values increased as the temperature increase for a particular mixture. Similar results are obtained by many researchers [20,21] to understand the solute-solute and solute-solvent interactions among molecules [19,22-24]. The distraction in the specific structure in medium can be explained through thermodynamics of dielectric relaxation. Gedam and Suryavanshi [25] have done a dielectric investigation of polar liquids in the dilute solution of benzene at fixed microwave frequency and at a constant temperature 300 K. The values of dielectric loss and dielectric constant were measured by them. They observed that both the dielectric parameters are concentration dependent. They selected three different polar liquids *viz.*, acetone, pyridine and nitrobenzene for experiment and determined the values of ϵ' and ϵ'' in the dilute solution of non-polar solvent benzene for all three polar liquids using X-band microwave bench for different concentrations. The concentration of the solutions kept very dilute to minimize solute-solute interaction. These dielectric parameters helped to calculate the value of various relaxation times and dipole moments. The results are in good agreement with literature [25].

The dielectric behaviour of the molar concentrations of ternary mixtures of triethylamine with dimethyl phthalate was studied by Khan and Manian [26] in the dilute solution of benzene at a fixed microwave frequency 9.36 GHz for various concentrations at three different temperatures (303, 308 and 313 K). They determined various dielectric parameters (ϵ' , ϵ'' , ϵ_0 and ϵ_∞) and analyzed the results. They found that these

values are able to explain the molecular structure and molecular forces in the liquids and solutions. They dissolved the ternary mixture of polar solvent in non-polar liquids and determined relaxation parameters through which they obtained important information regarding solute-solute and solute-solvent interactions. They realized that at microwave frequencies such relaxation studies can explain the association of two polar molecules through hydrogen bonding. They calculated different relaxation times (τ_1 , τ_2 and τ_0) by both Cole-Cole method and Higasi's method and found that the values calculated by Higasi's method are lesser than that of Cole-Cole method. The rigid behaviour of molecules of solute may be responsible for such difference in values [26].

Dielectric studies of aromatic alcohol with three different amides *viz.* acetamide, *N*-methylacetamide and *N,N*-dimethylacetamide for binary and ternary systems in dilute solution of carbon tetrachloride was done by Vijayabalan & Sivagurunathan [27] for various molar ratios at a fixed frequency and constant temperature with the help of X-band microwave bench. In this relaxation study, they found that the values of all three relaxation times are higher for a particular molar ratio of binary and ternary mixture as compared to the other sets of molar ratios of binary as well as ternary mixtures of the given sample. Hydrogen bonding is attributed for this behaviour as it directly influences the relaxation times of samples. They also studied the association between free hydroxyl groups of benzyl alcohol with carbonyl group of acetamides [27]. The study in the present paper shows that *N*-methylacetamide has maximum ability of accepting proton and in *N,N*-dimethylacetamide has the minimum ability of accepting proton *i.e.* it varied with the substitution of methyl group. This investigation provides valuable information regarding solute-solute interaction in the system.

EXPERIMENTAL

4-Bromoacetanilide of AR grade was purchased from the Central Drug House, Delhi, India. Carbon tetrachloride was obtained from Merck and used without further purification. We have taken five different mole fractions of 4-bromoacetanilide (0.01, 0.015, 0.02, 0.025 and 0.03 mol) and dissolved them in 1 mol of carbon tetrachloride to make dilute solutions. The dielectric relaxation studies are performed at four different temperatures (303, 313, 323 and 333 K) and at a fixed frequency 9.27 GHz by using X-band microwave bench.

The values of dielectric constant (ϵ') and dielectric loss (ϵ'') are measured on X-band microwave bench using Heston *et. al* method [28] by the following equations:

$$\epsilon' = \left(\frac{\lambda_o}{\lambda_c} \right)^2 + \left(\frac{\lambda_o}{\lambda_d} \right)^2 \quad (1)$$

$$\epsilon'' = \frac{2}{\pi} \left(\frac{\lambda_g}{\lambda_d} \right) \left(\frac{\lambda_o}{\lambda_d} \right)^2 \frac{d\rho}{dn} \quad (2)$$

where

$$\rho = \frac{\sin \theta}{(2 - \cos^2 \theta)^{1/2}} \quad (3)$$

and

$$\theta = \frac{\pi \Delta x}{\lambda_g} \quad (4)$$

where, λ_0 is the free space wavelength, λ_c is the cut-off wavelength, λ_g is the wavelength in the empty waveguide and λ_d is the wavelength in dielectric medium. The values of all these wavelengths have been listed in Table-1. Also, the values of λ_d (dielectric wavelength) have been shown at four temperatures and five different weight fractions in Table-1.

' ρ ' denotes the inverse voltage standing wave ratio, Δx represents double minima width and 'n' is the number of minima. The values of dielectric permittivity at static frequencies (ϵ_0) and at optical frequencies (ϵ_∞) are determined by using a dipole meter and Abbe's refractometer, respectively [11]. All the measurements are performed at various temperatures using a constant temperature circulating water bath fitted with a thermostat having temperature stability of the order of $\pm 0.1^\circ\text{C}$.

For dilute solutions in non-polar solvents ϵ' , ϵ'' , ϵ_0 and ϵ_∞ can be expressed as linear functions of concentrations [29,30] in the following manner:

$$\begin{aligned} \epsilon' &= \epsilon'_1 + a' W_2 & (a) \\ \epsilon'' &= a'' W_2 & (b) \\ \epsilon_0 &= \epsilon_{10} + a_0 W_2 & (c) \\ \epsilon_\infty &= \epsilon_{1\infty} + a_\infty W_2 & (d) \end{aligned} \quad (5)$$

where subscript 1 refers to pure solvent, 2 refers to solute, 0 is for the zero frequency measurements in static field and ∞ is for the values at infinite frequency, W_2 represents the weight fraction of solute. The slopes of these linear equations are denoted as ϵ' , ϵ'' , ϵ_0 and ϵ_∞ , respectively.

The values of relaxation times (τ_1 , τ_2 and τ_0) are determined by the method given by Higasi *et al.* [31].

The relaxation time for overall molecular rotation (τ_1) is defined by:

$$\tau_1 = \frac{a''}{\omega(a' - a_\infty)} \quad (6)$$

while the relaxation time for intramolecular rotations (τ_2) is given by:

$$\tau_2 = \frac{(a_0 - a')}{\omega a''} \quad (7)$$

where ' ω ' is the angular frequency.

The most probable relaxation time (τ_0) is then obtained by employing the following relation:

$$\tau_0 = \sqrt{\tau_1 \tau_2} \quad (8)$$

The value of dipole moment (μ) of solute molecules is evaluated by using Higasi's method. According to this method the value of dipole moment is given by:

$$\mu = \left(\frac{27kTM_2}{4\pi N(\epsilon_{01} + 2)^2 d_1} \right)^{1/2} (a_0 - a_\infty)^{1/2} \quad (9)$$

where M_2 is the molecular weight of solute, d_1 is the density of solvent, k is the Boltzmann constant, N is the Avogadro's number and T is the temperature at which the experiment is performed. Thus, knowing the value of a_0 and a_∞ and μ can be obtained from eqn 9 at different temperatures.

RESULTS AND DISCUSSION

During the investigations of dielectric properties of 4-bromoacetanilide with carbon tetrachloride, it is noticed that all the dielectric parameters *i.e.* dielectric constant (ϵ'), dielectric loss (ϵ''), static permittivity (ϵ_0) and optical permittivity (ϵ_∞) are decreased with temperature and increased with the weight fractions of solute. The experimentally observed values of dielectric constant (ϵ'), dielectric loss (ϵ''), dielectric constant at static frequency (ϵ_0) and dielectric constant at optical frequency (ϵ_∞) at four different temperatures (303, 313, 323 and 333 K) and five different mole fractions (0.01, 0.015, 0.02, 0.025 and 0.03) of 4-bromoacetanilide at a fixed microwave frequency 9.27 GHz in the dilute solution of carbon tetrachloride are listed in Table-2.

The decrease in the values of all these dielectric parameters (ϵ' , ϵ'' , ϵ_0 and ϵ_∞) with temperature indicates that the relaxation frequency of the molecules present in the system is higher than the applied microwave frequency (9.27 GHz). The decrease in the value of dielectric constant (ϵ') with temperature may be due to the random orientation of molecules because of their thermal motion. All these dielectric parameters (ϵ' , ϵ'' , ϵ_0 and ϵ_∞) are varying linearly with the weight fractions of the solute in carbon tetrachloride at a constant temperature. It indicates that there is no change in the nature of rotating molecular entities in solvent. This result confirms the applicability of Debye's theory [32,33]. It may also be due to the increase in hydrogen bonding in the solution as pure 4-bromo has a dipole-dipole interaction.

Fig. 1 represents the linear variation of dielectric constant (ϵ') with weight fractions of solute at all four temperatures. The slope of this graph gives the value of a' and Table-3 shows the values of a' at all temperatures. Similarly, Figs. 2-4 represent the linear variations of dielectric loss (ϵ''), static permittivity (ϵ_0) and optical permittivity (ϵ_∞) with weight fraction of solute respectively in the dilute solution of carbon tetrachloride at

TABLE-1
VALUES OF DIFFERENT WAVELENGTHS WHICH ARE USED TO MEASURE DIELECTRIC CONSTANT (ϵ') AND DIELECTRIC LOSS (ϵ'') OF 4-BROMOACETANILIDE AT DIFFERENT TEMPERATURES AND WEIGHT FRACTIONS OF SOLUTE IN DILUTE SOLUTION OF CARBON TETRACHLORIDE

Weight fraction	λ_d (cm) at 303	λ_d (cm) at 313	λ_d (cm) at 323	λ_d (cm) at 333	λ_g (cm)	λ_0 (cm)	λ_c (cm)
0.01373	2.362	2.369	2.375	2.382	4.482	3.241	5.060
0.02045	2.330	2.340	2.347	2.354	—	—	—
0.02709	2.306	2.314	2.326	2.336	—	—	—
0.03362	2.282	2.287	2.299	2.306	—	—	—
0.04007	2.258	2.2665	2.269	2.274	—	—	—

TABLE-2
EXPERIMENTAL VALUES OF DIFFERENT DIELECTRIC PARAMETERS OF 4-BROMOACETANILIDE IN DILUTE SOLUTION OF CARBON TETRACHLORIDE AT FREQUENCY 9.27 GHz USING X-BAND MICROWAVE BENCH AT DIFFERENT TEMPERATURES

Weight fraction	ϵ'	ϵ''	ϵ_0	ϵ_∞	ϵ'	ϵ''	ϵ_0	ϵ_∞
	Temperature: 303 K				Temperature: 313 K			
0.01373	2.293	0.020	2.439	2.116	2.281	0.018	2.421	2.102
0.02045	2.345	0.023	2.482	2.118	2.330	0.022	2.473	2.104
0.02709	2.386	0.026	2.524	2.121	2.371	0.024	2.508	2.107
0.03362	2.427	0.032	2.568	2.124	2.418	0.029	2.553	2.109
0.04007	2.471	0.032	2.618	2.125	2.455	0.031	2.603	2.112
	Temperature: 323 K				Temperature: 333 K			
0.01373	2.272	0.017	2.419	2.087	2.261	0.016	2.404	2.073
0.02045	2.316	0.020	2.459	2.089	2.306	0.019	2.442	2.075
0.02709	2.352	0.024	2.486	2.092	2.335	0.023	2.479	2.078
0.03362	2.397	0.026	2.542	2.095	2.385	0.025	2.532	2.081
0.04007	2.450	0.029	2.599	2.096	2.440	0.027	2.579	2.082

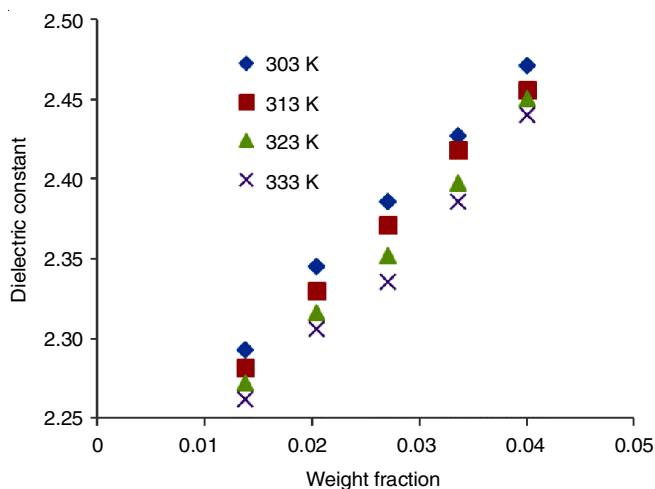


Fig. 1. Graph between dielectric constant and weight fraction

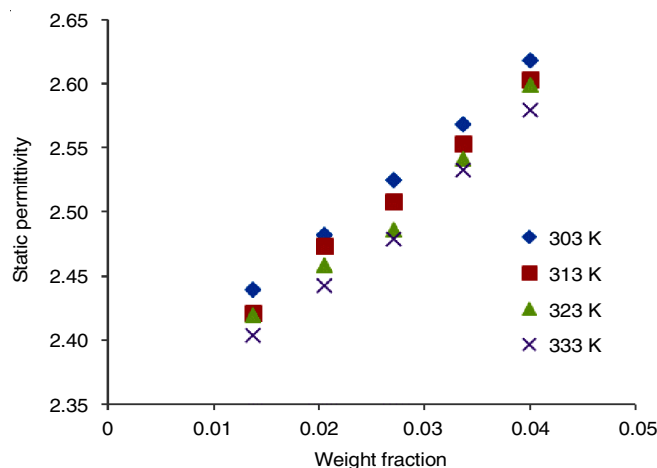


Fig. 3. Graph between dielectric permittivity at static frequency and weight fraction

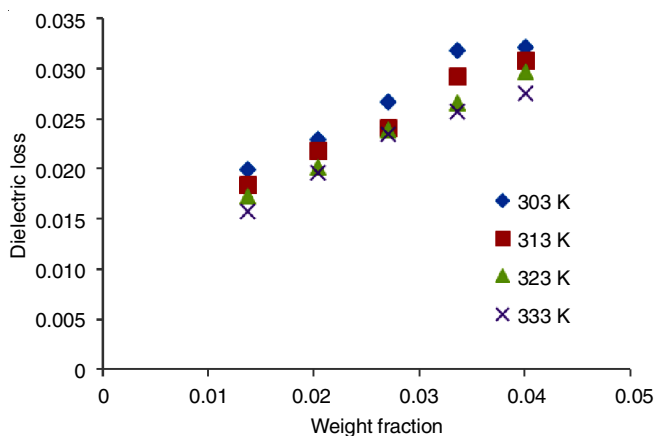


Fig. 2. Graph between dielectric loss and weight fraction

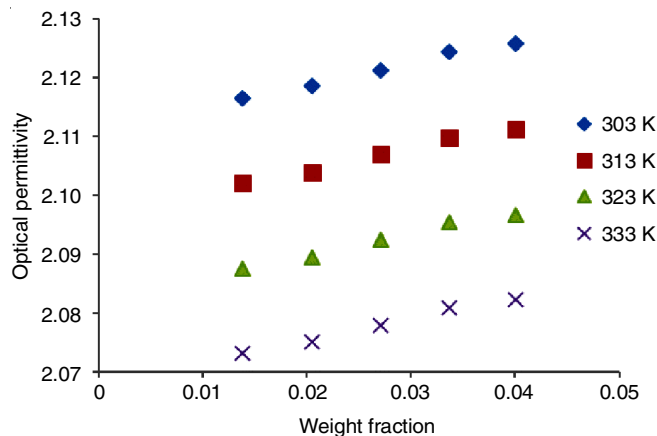


Fig. 4. Graph between dielectric permittivity at optical frequency and weight fraction

TABLE-3
VALUES OF SLOPES, RELAXATION TIMES AND DIPOLE MOMENT OF 4-BROMOACETANILIDE IN THE DILUTE SOLUTION OF CARBON TETRACHLORIDE AT FOUR DIFFERENT TEMPERATURES

Temperature (K)	Slope				Relaxation times (pico sec)			Dipole moment (Debye)
	a'	a''	a_0	a_∞	τ_0	τ_1	τ_2	μ
303	6.643	0.505	6.746	0.368	2.20	1.38	3.51	2.67
313	6.636	0.490	6.733	0.369	2.14	1.34	3.41	2.71
323	6.630	0.470	6.720	0.368	1.94	1.29	2.93	2.75
333	6.620	0.449	6.695	0.368	1.88	1.23	2.87	2.79

all four temperatures. The slopes of these graphs give the values of a'' , a_0 and a_∞ respectively and these slope values are also mentioned in Table-3 at all temperatures. By using the slope values a' , a'' , a_0 and a_∞ , we have determined the values of all the relaxation times (τ_1 , τ_2 and τ_0) and dipole moment (μ) of 4-bromoacetanilide in the dilute solution of carbon tetrachloride at different temperatures. The calculated values of relaxation times and dipole moments are shown in Table-3.

The relaxation time depends upon shape and size of rotatory molecular entities in the solution. Table-3 shows that the values of τ_1 , τ_2 and τ_0 . From this table, it is found that the relaxation time values decrease systematically with increase in temperature [34]. Two reasons may be possible for such result, first is increase in molar volume due to temperature and second is increase in size of dipole with temperature. It may also possible that as temperature increases, viscosity of solution decreases as a result of which the frictional resistance for dipolar orientation decreases. Both these factors may be responsible for such decrease in relaxation time with temperature. It is also observed that the values of relaxation time due to intramolecular rotation of molecules (τ_2) are higher than the values of relaxation time due to overall rotation of molecules (τ_1). This difference in values of relaxation time indicates that an additional intramolecular relaxation process exist with the overall relaxation process *i.e.* it confirms the presence of two relaxation mechanisms in the system. This result also suggests that the rate of fall of τ_2 with temperature is more considerable than τ_1 which shows that the relaxation time due to intramolecular rotation falls off at higher rate with temperature as compared to the relaxation times due to intermolecular rotation. It indicates that in polar liquids, the relaxation process of intramolecular rotation dominates over intermolecular rotation [35].

The self association between molecules of 4-bromoacetanilide exist in the dilute solution of carbon tetrachloride. A weak hydrogen bonding takes place among the molecules of 4-bromoacetanilide so that it shows a weak molecular association between its self molecules. The H-bond is formed only between -NH groups and =OCH₃ group among the molecules of 4-bromoacetanilide. As we know that when a chemical compound has higher number of electronegative oxygen atoms, it shows quite strong hydrogen bonding among its molecules. From the chemical structure of 4-bromoacetanilide, it can be seen that it does not has more electronegative oxygen atom and this may be the reason that it has weak hydrogen bonding between its self molecules. The weak hydrogen bonding may affect the relaxation time of the system. The increased alkyl chain length may also be responsible for weak bonding as the linear increase in the chain length of solvent directly proportional to the strength of hydrogen bonding.

Dipole moment is a factor which may affect the relaxation mechanism of any molecules. The dipole moment values (Table-3) are not directly related with the values of relaxation time. It is suggested that the intramolecular interaction and some other factors like shape and size of the molecules may influence the values of relaxation times. In present case, the values of dipole moments are increased with the increase in temperature. It may be because the length of dipole increases with temperature. This suggests the existence of solute-solvent

[36] type of molecular association for 4-bromoacetanilide in dilute solution of carbon tetrachloride.

Conclusion

From this study, it could be concluded that various dielectric parameters (ϵ' , ϵ'' , ϵ_0 and ϵ_∞) of 4-bromoacetanilide in the dilute solution of carbon tetrachloride measured at a fixed frequency using standard method shows some significant changes in their values with variation in the temperature and mole fractions. The values of relaxation times (τ_1 , τ_2 and τ_0) and dipole moments (μ) are determined by the method given by Higasi *et al.* for all the samples. These relaxation mechanisms help to explain the solute-solute and solute-solvent molecular interactions among the molecule at microwave frequency. The calculated values of dipole moment agrees well with the literature values.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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