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# Dielectric Investigations of Nonyl *p*-Hydroxy Benzoate at Room Temperature

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**ABSTRACT:** Plunger method in microwave frequency region, LCR bridge for static dielectric constant and Abbe's refractometer instruments for measurement of high frequency dielectric constant were used in order to calculate relaxations times. For dipole moments Higasi method was used in the dielectric study of the various concentrations of the binary system of nonyl *p*-hydroxy benzoates (9HB) and Isopropanol and benzene as solvent. There is an increase in the relaxation time as the concentration of the benzoate is increased in solute system in different concentrations of non-polar solvent benzene and there is a decrease in the dipole moment as the concentration of the benzoate is increased in solute system in non-polar solvent benzene and they are attributed to the formation of hydrogen bond between the solute systems.

**KEYWORDS:** Higasi method, Cole-Cole plot, Dipole moment, Relaxation time, Hydrogen bond

## I. INTRODUCTION

Studies of liquid mixtures have gained importance [1-3] because those provides one way of bringing together the molecules of different compounds and allow them to interact with one another. The physical forces responsible for such interactions cannot be studied when such molecules are isolated. Molecular mixtures bring about changes in thermodynamic properties like entropy, free energy and heat of mixing. Changes in physical properties like density, molar volume, viscosity, surface tension, refractive index, dielectric constant and speed of acoustic waves are manifestations of intermolecular interactions in liquid mixtures. Dielectric investigations of solutions containing varying amounts of interacting molecules help to detect the formation and composition of complexes in them. The nature of complex formation in binary mixtures is still far from clear. The Present trend is of liquid mixtures leading to formation of hydrogen bonding in the system due to solute-solvent interactions. Hydrogen bonding is a complex in the liquid state because of the uncertainty in identifying the particular bonds and the number of molecules involved. One of the physical parameters used for conformational analysis of any resultant structure is the net dipole moment besides relaxation time, thermodynamic parameters such as enthalpy and entropy values [4-6]. With this in view, the present investigation is aimed at studying the dielectric behaviour of binary mixtures of nonyl *p*-hydroxy benzoates (9HB) and Isopropanol in benzene which may provide useful information about the formation of complex in the mixture.

## II. RELATED WORK

Nonyl-*p*-Hydroxy benzoate is used in various fields' viz. food preservatives, cosmetic preservatives [7-9] and has biological importance. Alcohols have found various applications [10] and commercial use in medical and other fields, for example *n*-propyl alcohol is very effective against a broad spectrum of microorganisms including bacteria, fungi and viruses such as HIV, hepatitis-B, and respirator syncytial viruses. Also *n*-propyl alcohol is in use as the safest antiseptic compound for tropical use and is feedstock in the manufacture of insecticides. Most of the dielectric relaxation processes reported in the literature were studied for dilute solutions of polar substance in non-polar liquids. The non-polar liquid does not itself undergo relaxation but alters the relaxation time of the solute molecule by reducing the internal field and changing the viscosity. Relatively little work has been done on mixtures of polar components. The dielectric constants and losses of binary mixtures of polar components as functions of temperature and found two maxima for mixtures of an associated and a non-associated liquid but only one maximum for mixtures containing only associated liquids or non-associated liquids as investigated by Scballamach [11] . On the basis of this observation he suggested that dielectric relaxation involves the disturbance of a relatively large region in the liquid and that a mixture

of associated and non-associated liquids is not microscopically homogeneous. However, measurements at several microwave frequencies and at different temperatures have indicated that the dielectric behaviour of binary mixtures of non-associated pure liquids have been reported in the literature [9,11-15]. The dielectric absorption of some benzene derivatives and their binary mixtures has been studied by Boruah *et al* [16] at 3 cm. Investigation of dielectric relaxation in tributyl phosphate were carried out from susceptibility and conductivity measurement under microwave field by Bachhar *et al* [17]. wave length microwave region in the range of temperature from 25°C to 45°C. The relaxation time, dipole moment, and the thermodynamic parameters for the dielectric relaxation as well as for viscous process were measured. They discussed the results on the basis of molecular size, molecular polarity, molecular environment, molecular interaction, effect of localization of charge density and contribution arising from the formation of hydrogen bond structure. Dielectric Studies were carried out on Hydrogen Bonded Polar Binary Mixtures of Alkyl *p* –Hydroxy Benzoates and Isopropanol by Sreehari Sastry *et al.* [18].

### III. EXPERIMENTAL

Nonyl *p*-hydroxy benzoate (9HB) is used in various fields viz. food preservatives, cosmetic preservatives<sup>7-9</sup> [7-9] and has biological importance. 9HB of extra pure quality with 99.5% purity were supplied by M/s Frinton Laboratories Inc, USA. Both Isopropanol and Benzene were supplied by Qualigens. Both of them were ExcelaR grade and were double distilled before use. Benzene is used as solvent, the binary system of nonyl *p*-hydroxy benzoates (9HB) and Isopropanol is used as solute in the preparation of solution. 9HB is dissolved in Isopropanol in various concentrations, the various weights of 9HB for each concentration is given for 26.5 ml of Isopropanol, the mole fraction of 9HB in isopropanol from 0.01, 0.02, 0.03, 0.04 and 0.05 %, the weight of 9HB of 0.95925, 1.93808, 2.93709, 3.95691 and 4.99821 grams respectively. All the weights are measured using a single pan electronic balance Dhona make, model 100 DS with an accuracy of 0.01 mg. Each concentration of solute is diluted with benzene in volume fractions of 90% benzene and 10% solute, 80% benzene and 20% solute, 70% benzene and 30% solute, and 60% benzene and 40% solute. The dielectric studies are carried out using LCR bridge (static dielectric constant), the microwave bench adopting plunger<sup>3,10-14</sup> [3,19-23] method (in X-band) and Abbe's refractometer for refractive index (high frequency) at room temperature (300 °K)..

### IV. RESULTS AND DISCUSSION

Experimental results obtained from the optical, static and microwave frequencies for different concentrations of 9HB in isopropyl alcohol, and it is diluted in a non polar solvent medium as benzene. Since the solute (9HB+ Isopropanol) is a highly polar compound due to this reason highly saturated wave pattern is obtained in the measurement of complex dielectric constant at microwave frequency by using the plunger method. Due to the high loss nature of the sample it is not possible to measure the dielectric constant of high concentrations of the 9HB in isopropyl alcohol systems. In order to study the properties of these systems, we diluted the above system for different concentrations of non polar solvent medium benzene. Diluting the system in non polar solvent medium it is useful for studying the interaction between the 9HB and Isopropanol. From the standing wave pattern in the microwave frequency region, dilution studies of different concentrations of solute (9HB and Isopropanol) in non polar solvent medium is easy to obtain the dielectric constant and dielectric loss. The measurements are done at room temperature (300 °K).

The data obtained from different concentrations of the solute in a nonpolar solvent medium at static and optical frequency are used to evaluate the dipole moments of the solute using Higasi's method and this method is applicable only for dilute solutions. The average relaxation time is calculated from the Cole-Cole plot by plotting the real and imaginary parts of dielectric constant. From observing the relaxation time and the experimental dipole moment values there is increase in the dipole moment and relaxation values. The increase in the relaxation time values and it is due to probable formation of self associated groups in the solvent medium. The self associated groups are formed due to the formation of hydrogen bond between the 9HB and Isopropanol. Due to the presence of this self associated groups requires more energy (microwave) to relax, due to this relaxation time is increased. Due to the formation of hydrogen bond between the 9HB and Isopropanol, there is increase in the relaxation and dipole moment values.

When plunger is moved, the distance or the length of the column in the dielectric cell with respect to the mica sheet, voltage readings are noted. A typical data is shown in the table 1.



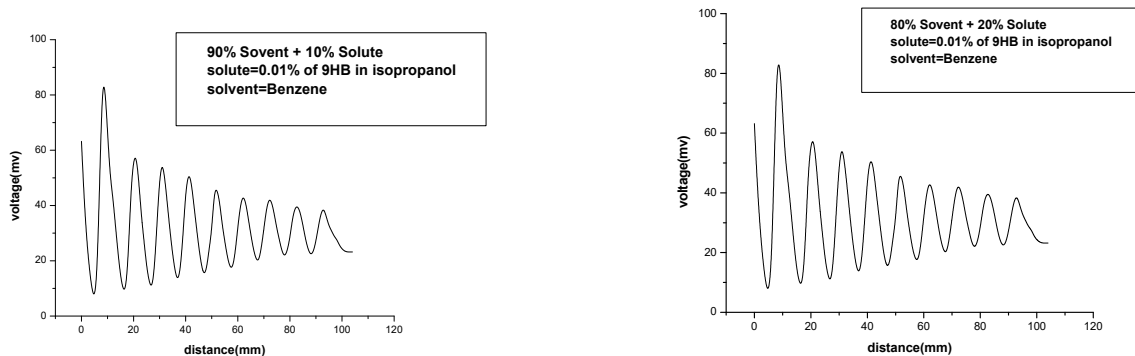
**Table 1:** Micro wave bench readings obtained by moving plunger in 0.01% of Nonyl P-hydroxy benzoate (9HB) in Isopropanol taken as solute and benzene as solvent for concentration of 90% benzene and 10% solute.

Distance (mm)	Voltage (mV)	Distance (mm)	Voltage (mV)	Distance (mm)	Voltage (mV)	Distance (mm)	Voltage (mV)	Distance (mm)	Voltage (mV)
1	63.2	22	58.1	43	58.1	64	41.3	85	37.4
2	43.1	23	48.9	44	48.9	65	35.5	86	32.8
3	27.9	24	36.3	45	36.3	66	29.5	87	27.8
4	17.4	25	27.2	46	27.2	67	24.2	88	24
5	9.7	26	18.1	47	18.1	68	20.5	89	22.1
6	6.3	27	12.2	48	12.2	69	19.8	90	23.2
7	15.2	28	10	49	10	70	24.4	91	27.1
8	49.2	29	16.9	50	16.9	71	31.5	92	32.8
9	83.6	30	33.7	51	33.7	72	38.8	93	37.6
10	84	31	49.7	52	49.7	73	42.6	94	39
11	66.5	32	55.8	53	55.8	74	41.3	95	35.8
12	50.9	33	49.7	54	49.7	75	36.9	96	32.6
13	43.4	34	40.2	55	40.2	76	31.5	97	30.8
14	33.4	35	30.5	56	30.5	77	27.4	98	28.8
15	23.1	36	22.4	57	22.4	78	23.2	99	27.7
16	14.4	37	15.7	58	15.7	79	21.5	100	25.6
17	9.3	38	12.7	59	12.7	80	23.6	101	24.3
18	9.6	39	17.2	60	17.2	81	28.4	102	23.5
19	22	40	29.8	61	29.8	82	35.4	103	23.2
20	45.7	41	44.8	62	44.8	83	39.3	104	23.2
21	56.8	42	51.5	63	51.5	84	39.8	105	23.2

Distance means length of liquid column in dielectric cell with respect to mica film as reference.

In order to calculate the relaxation times, the plunger readings at microwave frequency region, LCR for static dielectric constant and Abbe's refractometer for measurement of high frequency dielectric constant using the refractive index were used. All these data were used to fit in an Argand diagram to calculate the relaxation times. It is observed a distribution of relaxation times as witnessed from Cole-Cole arc plots instead of Debye arc plots. Although Cole-Cole [24] equations which signify the distribution of relaxation times, eventually a macroscopic relaxation time (predominant relaxation time) and dipole moments were measured and the data is given in Table 1.

The representative dielectric data at microwave frequency for one concentration (0.01% to 0.05% of 9HB in isopropanol taken as solute and benzene as solvent for concentration of 90% benzene and 10% solute) is given in Table 1. and typical graphs for the concentrations are given in Fig. 1.





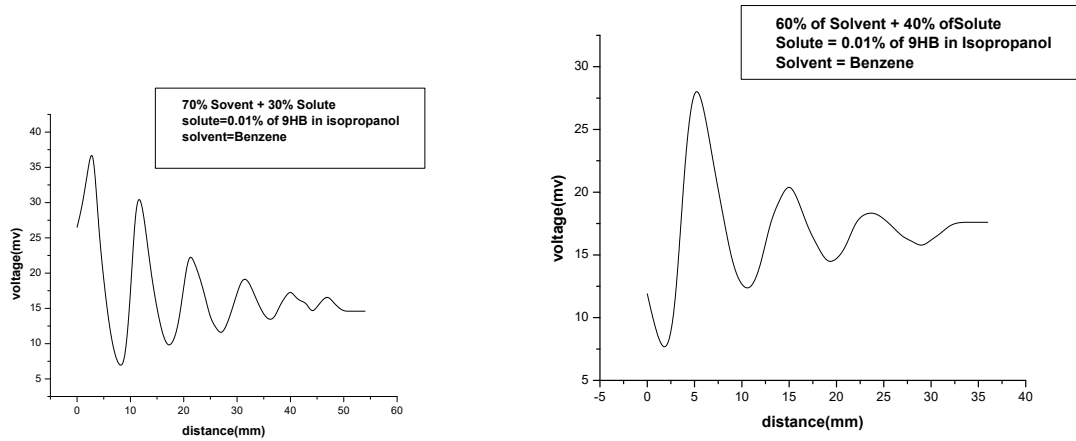


Fig 1. Length of the liquid in dielectric cell Vs Voltage

**Table 2:** Comprehensive data on dielectric constant, relaxation time, dipole moment for various concentrations of (9HB + Isopropanol) in Benzene

% of 9HB in isopropanol	Volume fraction of solvent + solute	$\epsilon_{static}$	$\epsilon_{microwave}$ Plunger Method	$n^2$ (Optical frequency)	Relaxation Time $\tau$ (pico sec)	Dipole moment $\mu$ (Debye)
0.01%	90+10	2.76	2.68-j0.29	2.229	5.83	5.39
	80+20	3.07	2.98-j0.62	2.190	6.85	
	70+30	4.30	3.12-j0.87	2.150	20.51	
	60+40	5.35	2.61-j1.07	2.114	44.11	
0.02%	90+10	2.64	2.61-j0.17	2.229	3.09	5.19
	80+20	3.42	3.09-j0.39	2.193	9.56	
	70+30	4.92	2.95-j0.43	2.157	48.07	
	60+40	5.00	2.74-j0.62	2.122	83.80	
0.03%	90+10	2.71	2.68-j0.21	2.232	4.15	5.10
	80+20	3.42	2.82-j0.49	2.190	17.80	
	70+30	4.91	2.89-j0.59	2.175	67.96	
	60+40	5.28	2.78-j0.48	2.128	159.06	
0.04%	90+10	2.77	2.69-j0.12	2.229	5.14	5.08
	80+20	3.23	2.83-j0.43	2.187	13.31	
	70+30	4.23	2.99-j0.48	2.166	32.61	
	60+40	5.30	3.12-j0.87	2.134	55.11	
0.05%	90+10	2.84	2.73-j0.20	2.226	7.81	4.97
	80+20	3.28	2.90-j0.35	2.199	9.89	
	70+30	4.53	3.36-j0.58	2.172	19.07	
	60+40	5.23	3.25-j0.77	2.140	37.83	

All these data were used to fit in an Argand diagram ( $\epsilon' Vs \epsilon''$ ) to calculate the relaxation times. It is observed that a distribution of relaxation times as witnessed from Cole-Cole arc plot instead of Debye arc plot. Cole-Cole equations signify the distribution of relaxation times. Eventually a macroscopic relaxation time (predominant relaxation time) is computed using Cole-Cole plots.



The corresponding parameters obtained from Cole-Cole arcs such as distribution parameter ‘ $\alpha$ ’ and the corresponding angle  $\theta = \pi\alpha/2$  and also the corresponding u, v parameters are given in Table 3.

Table 3: Data on u, v,  $\theta$ ,  $\alpha$  and relaxation time

% of 9HB in isopropanol	Volume fraction of solvent + solute	U	v	$\theta = \pi\alpha/2$	Distribution parameter( $\alpha$ )	Relaxation time $\tau$ (pico sec)
0.01%	90+10	2.4	1	6	0.06	5.83
	80+20	2.6	1	11	0.12	6.85
	70+30	6.5	7.2	11	0.12	20.51.
	60+40	6.3	13.8	10	0.11	44.11
0.02%	90+10	3.8	0.8	11	0.12	3.09
	80+20	4.5	2.8	24	0.26	9.56
	70+30	2.7	5.2	29	0.32	48.07
	60+40	2.8	6.1	10	0.11	83.80
0.03%	90+10	4.6	1.1	3	0.03	4.15
	80+20	4.0	3.9	14	0.15	17.80
	70+30	2.9	5.9	18	0.2	67.96
	60+40	3.1	5.6	36	0.4	159.06
0.04%	90+10	2.3	0.8	15	0.16	5.14
	80+20	3.8	2.9	13	0.14	13.31
	70+30	4.8	6.8	36	0.4	32.61
	60+40	6.0	11.5	37	0.41	55.11
0.05%	90+10	2.7	1.2	4	0.04	7.81
	80+20	4.0	2.5	21	0.23	9.89
	70+30	6.5	6.67	37	0.41	19.07
	60+40	6.8	10.7	34	0.37	37.83

**Calculation of relaxation time**

The dielectric data (0.01% of 9HB) in isopropanol taken as solute and benzene as solvent for concentration of 60% benzene and 40% solute is represented in complex plane ( $\epsilon''$  Vs  $\epsilon'$ ) resulting Cole-Cole plot and a typical one is shown in Fig. 2.

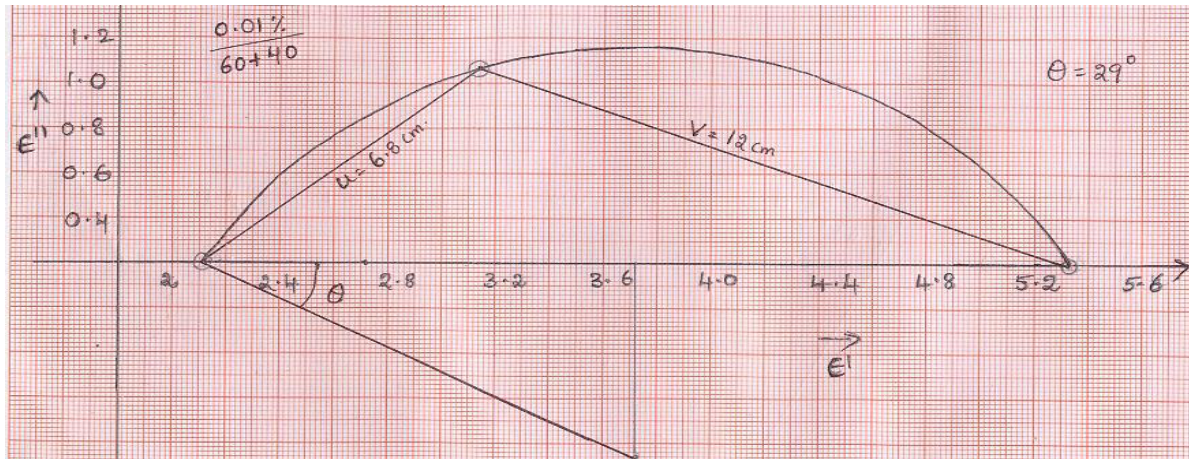


Fig.2: Cole-Cole plot for Solute = 0.01MF of 9HB in Isopropanol, Solvent= Benzene, 60% Solvent & 40% Solute



The parameters obtained from Cole-Cole arcs such as distribution parameter ‘h’ and the corresponding angle  $\theta = \pi\alpha/2$  and also the corresponding u, v parameters are given in Table 3. The relaxation times calculated from these Cole-Cole plots are also given in Table 2 and 3.

**Calculation of Dipole moment**

The dipole moments are calculated from Higasi method [25].

$$\mu^2 = \frac{27 kTM_2}{4\pi Nd_1} \cdot \frac{(a_0 - a_\infty)}{(\epsilon_1 + 2)^2}$$

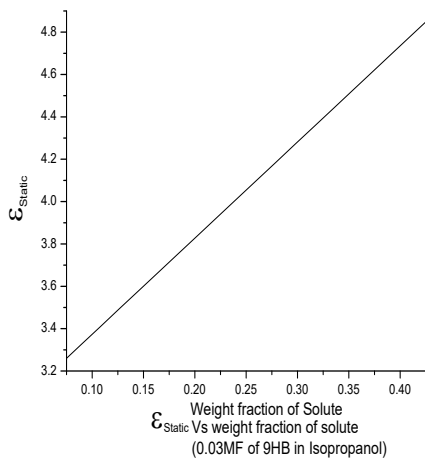
where  $M_2$  is molecular weight of solute,  $\epsilon_1$  is the dielectric constant of benzene, N is the Avogadro’s number,  $d_1$  is density of solvent,  $\epsilon_0$  and  $\epsilon_\infty$  are respectively the slopes of  $a_0$  and  $a_\infty$  with respect to the concentration .

The data required for calculating dipole moment is given along with the dipole moment for the above concentration in Table 3.

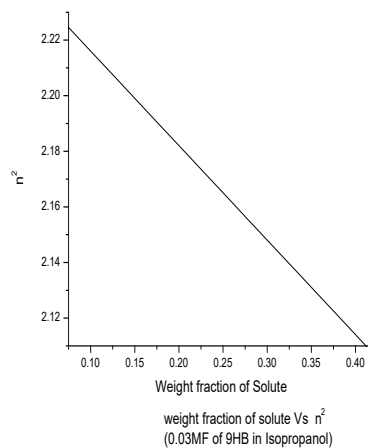
The values of  $a_0$  and  $a_\infty$  are found from the graphs of  $\epsilon_0$  (static permittivity) and  $\epsilon_\infty$  versus concentration respectively which are shown in Fig. 3 and 4 respectively for 0.03 mole fraction of 9HB in isopropanol. A representative dipole moment in 0.01% of 9HB in isopropanol is given in Table 4.(this is also seen in Table 2)

Table 4: Values of parameters for dipole moment in 0.01% of 9HB in isopropanol

% of 9HB in isopropanol	Volume fraction of solvent + solute	$\epsilon_{static}$	$\epsilon_{microwave}$ Plunger Method	$n^2$ (Optical frequency)	Relaxation Time $\tau$ (pico sec)	Dipole moment $\mu$ (Debye)
0.01%	90+10	2.76	2.68-j0.29	2.229	5.83	5.39
	80+20	3.07	2.98-j0.62	2.190	6.85	
	70+30	4.30	3.12-j0.87	2.150	20.51.	
	60+40	5.35	2.61-j1.07	2.114	44.11	



**Fig.3**



**Fig.4**



It is evident from the data given in the Tables 2 and 3, the dielectric constant increases with the increase of frequency. The microwave data show that these compounds are polar in nature and correspondingly the dielectric constant is a complex quantity. The relaxation time is found to increase in magnitude for a given concentration of 9BA in isopropyl alcohol with the increase of dilution in benzene which shows that the molecules are free to rotate as benzene concentration decreases. The dipole moment values decrease with increase of 9BA in Isopropyl alcohol which is probably due to the hydrogen bonding in the system.

## V. CONCLUSIONS

The low frequency static dielectric constant and microwave frequency dielectric constant increased with the increase of concentration of the solute binary system (benzoates and alcohols) in non-polar solvent benzene at room temperature. The optical refractive index value decreased with increase in the concentration of the solute binary system (benzoates and alcohols) in non-polar solvent benzene at room temperature. There is an increase in the relaxation time as the concentration of the benzoate is increased in solute system in different concentrations of non-polar solvent benzene and it is due to the formation of hydrogen bond between the solute systems. There is a decrease in the dipole moment as the concentration of the benzoate is increased in solute system in non-polar solvent benzene and it is due to the formation of hydrogen bond between the solute systems.

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