

Dielectric Investigations on Pentyl *P*-Hydroxy Benzoate

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Abstract— Cavity perturbation technique and plunger method in microwave frequency region to obtain dielectric constant and further to compare the data on both techniques, for static dielectric constant by LCR bridge and for measurement of high frequency dielectric constant through Abbe's refractometer were used in order to calculate relaxations times. Higasi method was used for dipole moments in the dielectric study for different concentrations of the binary system of nonyl *p*-hydroxy benzoate (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--Cavity perturbation technique, Plunger technique, Dielectric constant, Relaxation time, Dipole moment,

1. INTRODUCTION

Pentyl-*p*-Hydroxy benzoate has been used in various fields' viz. food preservatives, cosmetic preservatives [1-3] where as alcohols have found various applications [4] including commercial use in medical and other fields, for example a very effective against a broad spectrum of microorganisms including bacteria, fungi and viruses such as HIV, hepatitis-B, and respirator syncytial viruses. 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. The dielectric constants and losses of binary mixtures of polar components as function of temperature 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 Schallamach [511]. 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 been reported in literature [3,5-9]. The dielectric absorption of some benzene derivatives and their binary mixtures has been studied by Boruah *et al* [10] 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* [11] 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 some hydrogen bonded polar binary mixtures of alkyl *p* -Hydroxy Benzoates and Isopropanol by Sreehari Sastry *et al.* [12].

A resonant cavity method intermediate between the Hartshorn-wartd method and the microwave resonance methods suited for measurement on low loss materials in the frequency range has been described by Works *et al*[13]. They measured the dielectric properties of a variety of low loss materials at 200MHz. Birnbaum *et al* [14] observed that the microwave resonance method is used to determine the dielectric constant and loss of liquids, solids and very lossy gases provided the cavity volume is only partly filled with absorbing dielectric. A microwave resonance technique suited for measurement on low loss liquids has been described by Pitt and Smyth [15]. Cavity perturbation method is widely used in the characterization of dielectric [16], magnetic [17, 18] and super conducting materials [19]. The different microwave cavity perturbation techniques are discussed by Raman [20]. The application of microwave cavity perturbation technique for the study of transients in semiconductors is becoming popular due to its simplicity in measurement procedure and high sensitivity. Effects of quality factor, sample size, and coupling factor of the sensitivity of the measurement are discussed. [21]. The complex dielectric constant for different liquids are measured using waveguide transmission-line techniques at four frequency ranges. The results are discussed and compared with the literature and the calculated data using Debye's method [22].

A comparison is achieved between the four positions of the cavity from which the optimum cavity position is selected alongwith the limitations of this method are represented in [23].

In continuation of our dielectric work in binary systems Cavity perturbation technique and plunger method in microwave frequency region to obtain dielectric constant and further to compare the data on both techniques. Further, for static dielectric constant by LCR bridge and for measurement of high frequency dielectric constant through Abbe's refractometer were carried out in order to calculate relaxations times. Higasi method was used for dipole moments in the dielectric study for different concentrations of the binary system of pentyl *p*-hydroxy benzoate (5HB) and Isopropanol and benzene as solvent.

II. MATERIALS AND EXPERIMENTS

a. Materials

Pentyl *p*-hydroxy benzoate (5HB) of extra pure quality with 99.5% purity was supplied by M/s Frinton Laboratories Inc, USA. Both Isopropanol and Benzene were supplied by Qualigens. Both of them were Excelsior grade and were double distilled before use. Benzene is used as solvent, the binary system of pentyl *p*-hydroxy benzoate (5HB) and Isopropanol is used as solute in the preparation of solution. The structure of 5HB is shown in Fig. 1,

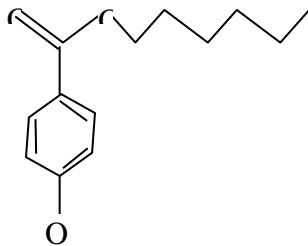


Fig. 1.: Structure of 5HB

b. Measurements

5HB is dissolved in Isopropanol in various concentrations. The various weights of 5HB for each concentration is given for 26.5 ml of Isopropanol, the mole fraction of 5HB in isopropanol from 0.02, 0.03, 0.04 and 0.05 %, the weight of 5HB of 1.62949, 2.46943, 3.32688, and 4.20237 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 technique and cavity perturbation technique [4-7,24] method (in X-band). The Resonant frequencies f_0 , f_1 and f_2 correspond to empty cavity, cavity with empty holder alone and lastly cavity containing holder with the sample. The dielectric constant is obtained through the equation

$$\epsilon^1 = 1 + \frac{V_c}{4V_s} \left[f_0^2 \left(\frac{1}{f_1^2} - \frac{1}{f_2^2} \right) \right]$$

where V_c and V_s = Volume of the cavity and sample respectively. The error in the real part of the dielectric constant is 2% . The cavity signals are shown in Plate 1 and 2.

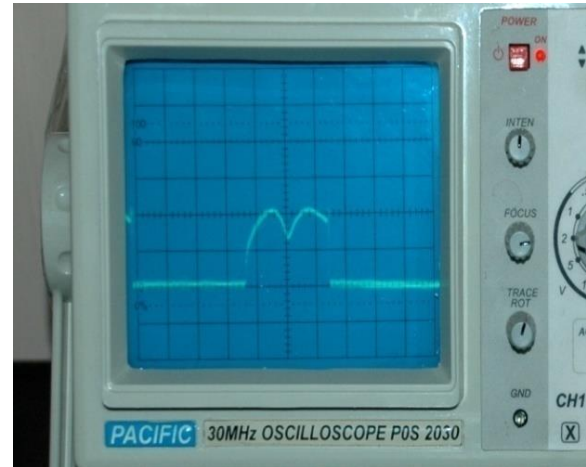


Plate 1: Signal due to cavity without sample

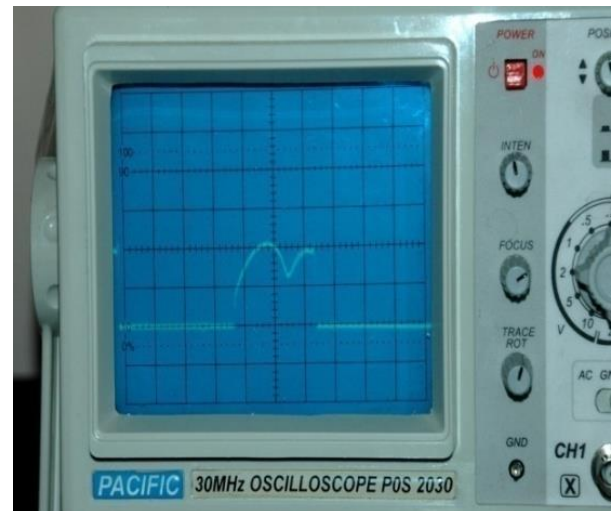


Plate 2 : Signal due to cavity with sample

Abbe's refractometer used for refractive index (high frequency-sodium light, dielectric constant ϵ_∞) at room temperature (300 °K). The error in the estimation of ϵ_∞ by this method is +0.04%.

III. RESULTS AND DISCUSSION

In this section the experimental results obtained from the optical, static and microwave frequencies for different concentrations of 5HB in isopropyl alcohol, and it is diluted in a non polar solvent medium benzene. Since the solute (5HB+ Isopropanol) is a highly polar compound, 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 5HB 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 5HB and Isopropanol. From the standing wave pattern in the microwave frequency region, dilution studies of different concentrations of solute (5HB 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).

Dielectric constant:

The different concentrations of solute system (5HB + Isopropanol) are dissolved in non-polar solvent benzene at various volume concentrations. The dielectric constant of each concentration is measured at optical frequency, low frequency 1 kHz and microwave frequency 8.83GHz at room temperature. The complex dielectric constant ($\epsilon^* = \epsilon' - j\epsilon''$) in the X band (8-12 GHz) microwave frequency region is measured by using the cavity perturbation and waveguide plunger techniques.

In cavity perturbation technique the signal obtained for without and with sample is fed to CRO from the tuned detector, which is shown in Plates 3.1. and 3.2 respectively. The measurement of dielectric constant and dielectric is made.

Standardization of the cavity technique:

The data was obtained from cavity perturbation technique with the samples Ethyl *p*- hydroxyl benzoate (EHB), Propyl *p*- hydroxyl benzoate (PHB) and Butyl *p*- hydroxyl benzoate (BHB) at room temperature for different concentrations. It is observed that the values are in good agreement with in the accuracy of the techniques involved.

On the compound 5HB the experiment is repeated using cavity perturbation and plunger techniques for different concentrations at room temperature and the data is shown in Table 1

Table 1. Dielectric data for various concentrations of (5HB+Isopropanol) in Benzene using Cavity and Plunger techniques

% of 5HB in isopropanol	Volume fraction of solvent + solute	$\epsilon_{\text{microwave (cavity)}}^* = \epsilon' - j\epsilon''$	$\epsilon_{\text{microwave (plunger)}}^* = \epsilon' - j\epsilon''$
0.02%	90+10	2.81-j0.37	2.78-j0.39
	80+20	3.52-j0.52	3.53-j0.51
	70+30	3.90-j0.78	3.91-j0.75
	60+40	4.39-j0.87	4.40-j0.86
0.03%	90+10	3.22-j0.47	3.21-j0.46
	80+20	3.46-j0.50	3.44-j0.51
	70+30	3.53-j0.66	3.53-j0.67
	60+40	4.43-j0.84	4.41-j0.86
0.04%	90+10	3.19-j0.36	3.21-j0.34
	80+20	3.64-j0.98	3.62-j1.11
	70+30	4.41-j0.85	4.40-j0.86
	60+40	4.96-j0.99	4.98-j0.98

0.05%	90+10	3.17-j0.60	3.19-j0.58
	80+20	3.44-j0.50	3.44-j0.51
	70+30	4.42-j0.85	4.41-j0.86
	60+40	5.11-j0.94	4.99-j0.98

Further, the frequency variation of dielectric investigations for different concentrations at room temperature is carried out. The data is presented in Table 2

Table 2: Comprehensive dielectric data at different frequencies for various concentrations of (5HB+ Isopropanol) in Benzene

% of 5HB in isopropanol	Volume fraction of solvent + solute	ϵ_{static}	$\epsilon_{\text{microwave (cavity)}}^* = \epsilon' - j\epsilon''$	n^2 (Optical frequency)
0.02%	90+10	3.03	2.81-j0.37	2.23
	80+20	3.73	3.52-j0.52	2.19
	70+30	4.43	3.90-j0.78	2.15
	60+40	5.24	4.39-j0.87	2.12
0.03%	90+10	3.40	3.22-j0.47	2.22
	80+20	3.81	3.46-j0.50	2.18
	70+30	4.24	3.53-j0.66	2.14
	60+40	4.77	4.43-j0.84	2.12
0.04%	90+10	3.37	3.19-j0.36	2.23
	80+20	4.05	3.64-j0.98	2.19
	70+30	4.77	4.41-j0.85	2.15
	60+40	5.26	4.96-j0.99	2.10
0.05%	90+10	3.39	3.17-j0.60	2.24
	80+20	4.11	3.44-j0.50	2.18
	70+30	4.96	4.42-j0.85	2.15
	60+40	5.33	5.11-j0.94	2.10

From the above data, the obtained values from both cavity perturbation and plunger techniques of dielectric constant and dielectric loss are same within the experimental accuracy at microwave frequency. Hence the data from any technique can be used to calculate the relaxation times. The data on dielectric constant, relaxation time and dipole moment is given in Table 3.

Table 3 Comprehensive data on dielectric constant, relaxation time, dipole movement for various concentrations of (5HB + Isopropanol) in Benzene

% of 5HB in isopropanol	Volume fraction of solvent + solute	Relaxation Time τ (pico sec)	Dipole moment μ (Debye)
0.02%	90+10	11.23	4.42
	80+20	4.41	
	70+30	6.68	
	60+40	6.83	
0.03%	90+10	6.54	3.55
	80+20	7.21	
	70+30	8.96	
	60+40	5.95	
0.04%	90+10	5.57	4.14
	80+20	11.12	

	70+30	5.95	
	60+40	5.15	
0.05%	90+10	9.40	4.17
	80+20	7.75	
	70+30	6.09	
	60+40	5.26	

A representative readings due to plunger method is give in Table 4.

Table 4: Microwave bench readings obtained by moving plunger in 0.02% of pentyl p-hydroxy benzoate (5HB) in Isopropanol taken as solute and benzene as solvent for concentration of 90% benzene and 10% solute

Distanc e (mm)	Volta ge (mv)	Distanc e (mm)	Volta ge (mv)	Distanc e (mm)	Voltag e (mv)
1	0.8	21	1.4	41	1.35
2	1.4	22	1.5	42	1.3
3	1.6	23	1.4	43	1.2
4	1.7	24	1.3	44	1.1
5	1.6	25	1.1	45	1.0
6	1.5	26	1.0	46	1.0
7	1.3	27	0.9	47	1.1
8	1.5	28	1.0	48	1.2
9	1.7	29	1.2	49	1.3
10	1.1	30	1.3	50	1.25
11	1.3	31	1.4	51	1.2
12	1.5	32	1.35	52	1.15
13	1.6	33	1.3	53	1.1
14	1.4	34	1.2	54	1.0
15	1.3	35	1.1	55	1.0
16	1.1	36	1.9	56	1.1
17	0.9	37	1.0	57	1.2
18	0.8	38	1.1	58	1.2
19	1.0	39	1.3	59	1.2
20	1.3	40	1.4	60	1.2

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

The above data is also shown in fig 2 showing attenuated (damped) waves when the distance is varied with respect to mica film.

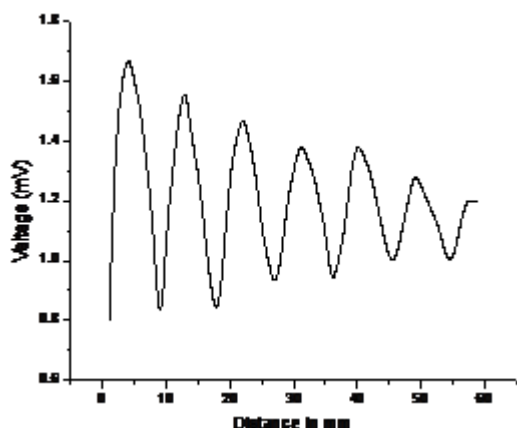


Fig 2: Voltage Vs Length of the liquid in cell

Cole – Cole plots and Relaxation time:

All these data were used to fit in an Argand diagram (ϵ'' versus ϵ') 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 which signifies 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 ' α ' and the corresponding angle $\theta = \pi\alpha/2$ and also the corresponding u, v parameters are given in Table 5. The corresponding relaxation times calculated from these Cole-Cole plots (Table 5) . The typical Cole-Cole plot is shown in figure 3.

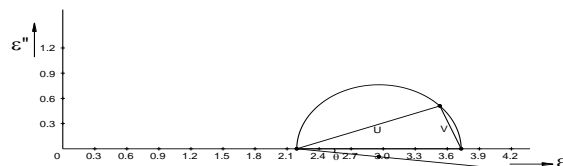


Fig 3: Cole – Cole plot for Solute=0.02MF of 5HB in Isopropanol Solvent = Benzene, 80% Solvent 20% Solute

Table 5 : Values of parameters for dipole moment in different concentrations of 5HB in isopropanol

% of 5HB in isopropanol	Volume fraction of solvent + solute	u	v	$\theta = \pi\alpha/2$	Distributi on paramete r(α)	Relaxati on time τ (pico sec)
0.02%	90+10	3.2	2.2	2	0.02	11.23
	80+20	5.9	1.8	7	0.07	4.41
	70+30	4.9	2.3	12	0.13	6.68
	60+40	6.2	3.2	20	0.22	6.83
0.03%	90+10	5.4	2.3	4	0.04	6.54
	80+20	4.5	2.2	9	0.10	7.21
	70+30	4.0	2.5	17	0.18	8.96
	60+40	6.1	2.4	5	0.05	5.95
0.04%	90+10	5.2	1.9	4	0.04	5.57
	80+20	5.8	3.8	15	0.16	11.12
	70+30	6.1	2.4	5	0.05	5.95
	60+40	5.9	2.0	4	0.04	5.15
0.05%	90+10	3.4	2.0	0	0	9.40
	80+20	4.5	2.8	31	0.34	7.75
	70+30	4.9	2.1	11	0.12	6.90
	60+40	6.0	2.1	5	0.05	5.26

Dipole moment:

The dipole moments are calculated from Higasi method

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

where M_2 is molecular weight of solute, d_1 is density of solvent, ϵ_1 is the dielectric constant of solvent (benzene), a_0 and a_∞ are respectively the slopes of ϵ_0 and ϵ_∞ with respect

to the concentration. The other constants have the usual meaning.

The data required for calculating dipole moment are given along with the dipole moment at each concentration in Table 3.

The values of a_0 and a_∞ are found from the graphs of ϵ_0 (static dielectric constant) and ϵ_∞ Vs concentration respectively which are shown in Figs. 4 and 5.

It is evident from the data given in Table 2, the dielectric constant increases with the increased 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 5HB in Isopropanol with the increase of dilution in benzene which shows that the molecules are free to rotate as benzene concentration increases.

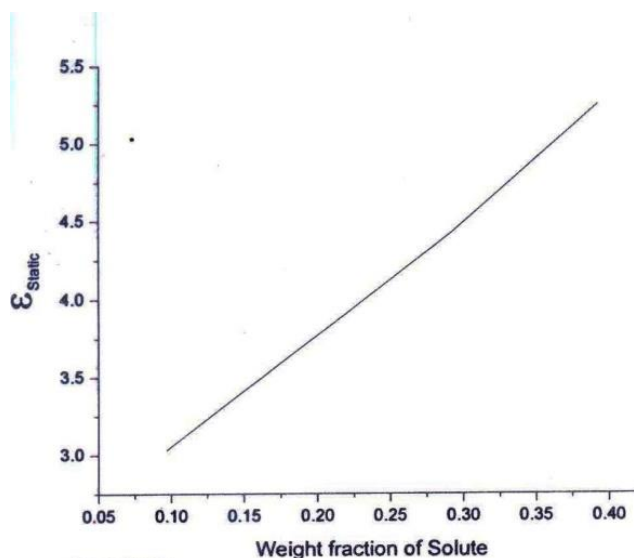


Fig. 4 ϵ_{static} Vs weight fraction of solute (0.02MF of 5HB in isopropanol)

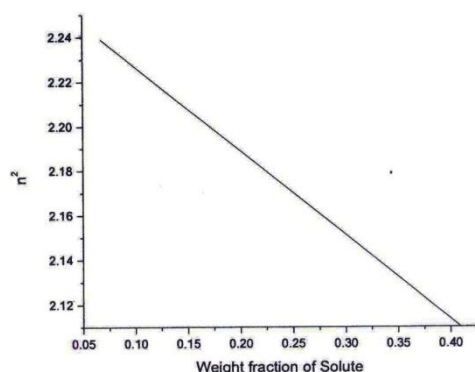


Fig.5 n^2 Vs weight fraction of solute (0.02MF of 5HB in isopropanol)

The same tendency is also seen for different concentrations of 5HB in Isopropanol. The dipole moment values decrease with increase of 5HB in Isopropanol which

is probably due to the hydrogen bonding in the system. This can be concluded from *ab-initio* calculation which gives molecular conformations, FTIR studies, etc

However, from the present study the dipole moment is in between 3.55 to 4.42 for various concentrations of 5HB in Isopropanol.

From the Table 2 it can be said that, the static dielectric constant (ϵ_{static}) increases with the increase of concentration of solute. Comparing the static dielectric constant of EHB [25], PHB [26] and BHB [27], it can be concluded that when concentration of solute increases static dielectric constant also increases.

In the microwave region also the dielectric constant increases with the increase of concentration of 5HB in binary system.

But at optical frequency region the dielectric constant decreases when the concentration of solute increases in the binary system. It is due to dipolar polarization contribution decreases at high frequency. At high frequency the dipolar polarization disappears and only atomic polarization or electronic polarization is present.

The dielectric constant decreases with the increase of frequency (from static (low frequency) to optical (high) frequency). It means frequency increases the dipolar polarization contribution decreases and vanishes. At static frequency all polarizations are present.

The relaxation time increases with the increase of concentration of solute. This behaviour may be attributed to the formation of hydrogen bond between isopropyl alcohol and 5HB.

The phenomenon of dielectric relaxation of the binary mixture of polar molecules in non-polar solvents at microwave frequencies has been attempted by many research workers. Raman Kumar and V.S Rangra [28] also calculated the binary mixture of N- Methylacetamide (NMA) and acetonitrile in benzene. They concluded that some molecular interactions exist in the binary mixture. The same may attributed to the present case that is, forming of hydrogen bond between 5HB and isopropanol. The strength of hydrogen bond reflects in the corresponding relaxation time and dipole moment value.

IV. CONCLUSIONS

The low frequency static dielectric constant and microwave frequency electric constant increased with increase in the concentration of the solute binary system (benzoate and alcohol) in non-polar solvent benzene at room temperature. The optical refractive index value decreased with increase in the concentration of the solute binary system (benzoate and alcohols) in non polar solvent benzene at room temperature. Dipole moment and relaxation times are calculated. 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. The data obtained through the plunger and cavity perturbation techniques is similar but the advantage is less quantity of sample is required in the cavity technique.

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