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INVESTIGATION OF INTERMOLECULAR INTERACTION BETWEEN MONOHYDRIC ALCOHOL AND 1, 4-DIOXANE MIXTURES USING EXCESS DIELECTRIC RALAXATION, BRUGGMAN FACTOR AND KIRKWOOD CORRELATION FACTOR

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Abstract: The study of dielectric properties in alcohols on addition of nonpolar solute like 1, 4-dioxane provides information about breaking of the molecular multimer structures in the systems. The values of complex permittivity for alcohol + 1, 4-dioxane using time domain reflectometry (TDR) method, the mixtures with various concentrations at 25° C have been determined in the frequency range 10 MHz to 20 GHz. The excess permittivity, excess inverse relaxation time, Bruggman factor and Kirkwood factor has been determined. The excess property for all the systems is negative.

Keywords: Dielectric relaxation, Kirkwood correlation factor, time domain reflectometry.

I. INTRODUCTION

1-Propanol and 2-Propanol are of the hydrogen bonded monohydric alcohols and are widely used as an industrial solvents. Monohydric alcohols are also classified into two groups according to the position of the hydroxyl group, i.e. primary and secondary alcohol. 1-Propanol is an ideal example of normal alcohol, where as 2-Propanol is secondary alcohol. Monohydric alcohols are typical hydrogen bonding substances. The hydroxyl group is a functional group of alcohols. The hydroxyl group can acts as proton donor as well as accepter, which results in the well known association in liquid alcohols via O---H-O bonds. The O-H bond in alcohol is highly polar, because oxygen is highly electronegative. The oxygen carries partial negative charge and the hydrogen carries partial positive charge. The polarity of O-H bond gives rise to attraction of partially positive hydrogen atom of other molecule. Over the past few year's much more experimental and theoretical studies concerning the hydrogen bonding behaviour of 1-Propanol (1P) and 2-Propanol (2P) have been reported [1-6]. The monohydric alcohol (1P & 2P) contains three carbon atoms and only one hydroxyl groups while it's position in the structure are different. It is interesting to note that static dielectric constant of 1-Propanol is large than that of 2-Propanol but relaxation time of 1-Propanol is smaller than that of 2-Propanol. The addition of 1-P and 2-P in dioxane forms three pair, Propanol-Propanol, Propanol-Dioxane and Dioxane-Dioxane. As dipole moment of Dioxane is very small (μ = 0.97D) [7], the interaction between third pair can be neglected. Therefore interaction between Propanol-Dioxane [8-15] can provide insight to understanding the exact nature of OH group through molecular interaction in the liquid. S. Czerkas et.al. [14]. In this paper we study the structure of polar liquids in terms of hydrogen bonding and modification of hydrogen bonding due to the presence of another polar or non-polar molecule. The study of dielectric properties of 1-Propanol and 2-Propanol on addition of non-polar solute like dioxane provides information about breaking of molecular multimer structure in the systems.

II. EXPERIMENTAL DETAILS

1-Propanol, 2-Propanol and 1, 4-dioxane (Aldrich chemicals) were obtained commercially used without further purification. The solution of alcohol + 1, 4-dioxane were prepared at eleven concentrations. The complex permittivity of the solutions was determined in the frequency range of 10 MHz to 20 GHz at 25° c using time domain reflectometry [16 - 19]. The Tektronix DSA8200 sampling oscilloscope with 30 GHz bandwidth and TDR module 80E08 with step generator unit was used. A fast rising step pulse with 18 ps incident pulse and 20 ps reflected pulse time was propagated through coaxial lines. All measurement is carried out in open load condition. Reflected pulse without sample R₁ (t) and with pulse R_X (t) were recorded in time window of 5ns and digitized in 2000 points. A temperature control system was used to maintain the temperature at 25° C to -5° C with accuracy limit of $\pm 1^{\circ}$ C. The temperature at the sample holder is checked by using electronic thermometer. A typical recorded pulses of R₁ (t) and R_X (t) as shown in fig 1.



Fig. 1 A typical recorded pulses without and with sample R_1 (t) and R_X (t) respectively.

III. Results and Discussion

The complex permittivity spectra, $\varepsilon^*(\omega) = \varepsilon'(\omega) - j\varepsilon''(\omega)$ for alcohol at 250C is shown in fig.2



Fig. 2. A Complex permittivity with Frequency (GHz) of alcohol.

The general form of the relaxation model is given by the Havriliak-Negami equation [20-23].

$$\varepsilon^{*}(\omega) = \varepsilon_{\infty} + \frac{(\varepsilon_{0} - \varepsilon_{\infty})}{\left[1 + (j\omega\tau)^{(1-\alpha)}\right]^{\beta}}$$
(1)

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where $\varepsilon_0 \& \mathcal{E}_{\infty}$ is the static and high frequency permittivity, τ is the relaxation time, ω is the angular frequency and $\alpha \& \beta$ are the distribution parameters for the relaxation times with values between 0 and 1 obtained by non-linear least squares fit method. The Havriliak-Negami equation includes three relaxation models. The Debye model ($\alpha = 0$ and $\beta = 1$) implies a single relaxation time while the Cole-Cole model ($0 \le \alpha \le 1$ and $\beta = 1$) and Cole-Davidson ($\alpha = 0$ and $0 \le \beta \le 1$) both suggest a distribution of relaxation times. The magnitudes of α and β indicates the width of the distribution. The Cole-Cole plot of 1-Propanol and 2-Propanol as shown in fig 3.



Fig. 3. The Cole -Cole plot for alcohol

Variation of the static dielectric constant with volume fraction of Propanol in 1,4-dioxane is shown in fig.4.



Fig. 4. Static dielectric constant with volume fraction of alcohol

The static dielectric constant increases as the concentration of alcohol in 1,4-dioxane increases. Variation of relaxation time (τ) , as a volume fraction of P in Dx is as shown in fig. 5. The relaxation time (τ) increases as the concentration of P in Dx increases. The dielectric relaxation parameters of the primary process strongly depend on the alcohol concentration in the whole concentration range and this process is expected to be due to co-operative motion of alcohol-alcohol and alcohol-1,4-dioxane molecules through hydrogen bond.



Fig. 5. Relaxation time with volume fraction of propanol

IV. EXCESS PARAMETER

The excess parameter approach seems to be useful to provide some tred regarding the solute-solvent interaction which may provide the structural information of binary liquid. In liquid binary mixtures there is a wide range of possible interactions between the constituents such as hydrogen bonding, molecular associations, dipole-dipole and dipole-induced interactions occur from ideal behaviour of dielectric constant with concentration variation of the mixture constituents, which is defined as the excess dielectric constant \mathcal{E}_0^E [24].

The excess permittivity (ε_0^E) may also provide structural information [19, 20]. This is determined for the Propanol-Dioxane solution as follows. $(\varepsilon_0^E) = (\varepsilon_0)_m - [(\varepsilon_0)_D X_D + (\varepsilon_0)_P (1 - X_D)]$ (2)

as follows. $(\varepsilon_0^{E}) = (\varepsilon_0)_m - [(\varepsilon_0)_D X_D + (\varepsilon_0)_P (1 - X_D)]$ (2) Where $(\varepsilon_0)_m$, $(\varepsilon_0)_D$ and $(\varepsilon_0)_P$ represent values static dielectric constant corresponding to mixture, 1,4-dioxane and Propanol respectively and 'X_D' is the volume fraction of 1,4-dioxane. The information extracted from the values on the binary mixtures is calculated value of excess permittivity $\varepsilon_0^E = 0$ means there is no interaction between solute and solvent or there is no change in net dipole alignments. If the value of $\varepsilon_0^E < 0$ means the mixture constituents act as 'Structure-breaker' for the other constituent H-bonded structure with orientation of some of the neighboring dipoles in opposite direction (antiparallel), and hence there is decrease in total number of parallel aligned effective dipoles that contributed to the mixture dielectric polarization due to which effective dipolar polarization gets reduced. If the $\varepsilon_0^E > 0$ means the mixture constituents of the binary mixture interact in such a way that they act as H-bonded 'Structure-makers' with parallel dipolar alignments in the same direction, which results in increase of effective number of aligned dipoles contributing to the mixture dielectric polarization. The higher value is stringer H-bond molecular connectivity and vice-versa. The negative values of excess permittivity confirm that the polar associate forms with lower dipole due to orientation of sum of the neighboring dipoles in the total permittivity. The hydrogen bonded model gives quantitative agreement with the experimental data of excess dielectric constants of the mixture. The shape of the theoretical curve of the excess dielectric constant matches with the experimental ones over the whole concentration of the alcohol in Dioxane.

V. EXCESS RELAXATION TIME

The information related to solute-solvent interaction may be obtained by excess relaxation time in mixtures [21-23]. The excess inverse relaxation time $(1/\tau)^{E}$ is defined as

$$\left(\frac{1}{\tau}\right)^{E} = \left(\frac{1}{\tau}\right)_{m} - \left[\left(\frac{1}{\tau}\right)_{D} X_{D} + \left(\frac{1}{\tau}\right)_{P} (1 - X_{D})\right]$$
(3)

Where m, D and P corresponding to mixture, dioxane and Propanol, respectively and X_D is the volume fraction of Dx. While $(1/\tau)^E$ is the excess inverse relaxation time which represents the average broading of dielectric spectra. If the $(1/\tau)^E$ is equal to zero, there is no change in the dynamics of Dioxane and Propanol interaction. If the $(1/\tau)^E$ is less than zero, Dioxane and Propanol interaction produces a field such that the effective dipoles rotate slowly and if $(1/\tau)^E$ is greater than zero, the Dioxane and Propanol interaction produces a field such that the effective dipoles rotate faster i.e. the field will cooperate in rotation of dipoles. Excess inverse relaxation time was plotted against volume fraction of alcohol is shown in fig. 6. From the figure it can be seen that the excess inverse relaxation times is negative in the entire concentration region. This suggested that solute-solvent interaction provides a field such that the effective dipoles rotation of dipoles.



VI. BRUGGMAN FACTOR

A useful model known as Bruggeman mixture formula can find out the static permittivity of mixture of two polar dielectric. The static permittivity of mixture with volume fraction of solute is given by [27-29].

$$f_B \left[\frac{\left(\varepsilon_{0m} - \varepsilon_{02} \right)}{\left(\varepsilon_{01} - \varepsilon_{02} \right)} \right] \left(\frac{\varepsilon_{01}}{\varepsilon_{0m}} \right)^{\frac{1}{3}} = 1 - V_2$$

$$\tag{4}$$

Where (f_B) is the Bruggeman dielectric factor. The ε_{0m} , ε_{01} and ε_{02} are the static dielectric constant corresponding to mixture Propanol and Dioxane respectively; V_2 is the volume fraction of Dioxane. The Bruggman equation may be modified for binary liquids as [27-29].

$$f_B = \left[\frac{\left(\varepsilon_{0m} - \varepsilon_{02}\right)}{\left(\varepsilon_{01} - \varepsilon_{02}\right)}\right] \left(\frac{\varepsilon_{01}}{\varepsilon_{0m}}\right)^{\frac{1}{3}} = 1 - \left[a - (a - 1)V_2\right]V_2$$
(5)

In this equation, volume fraction (V_2) is changed by a factor 'a' (a-1) V_2 of the mixture, a=1corresponds to Bruggeman's equation and the values as shown in table 1.

Fable 1 Bruggeman factors :	for DX-P	mixture
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Propanol	ʻa'
1- Propanol	1.28
2- Propanol	1.23

The value of 'a' can be determined by least square fit method and deviation of 'a' from unity indicates the molecular interaction in the mixture. Plot of f_B with volume fraction of alcohol as shown in fig. 8. From figure it can be seen that f_B is not a linear function of volume fraction of alcohol. The non linearity of the curves indicates hetero interaction which may be due to hydrogen bonding of the OH group of alcohol.



Fig. 8. Bruggeman factor with volume fraction of alcohol

VII. KIRKWOOD CORRELATION FACTOR

The Kirkwood correlations determine from the dielectric constant gives information about collective correlation between molecules [15, 16, 25]. The calculation of Kirkwood correlation factor 'g' provides information regarding the hindrance of molecular orientation by neighbouring molecules. The deviation of 'g' from unity is a measure of degree of hindered relative molecular motion arising from short-range intermolecular forces. When the molecules tend to direct themselves with parallel dipole moments, 'g' will be greater than unity. If the molecules prefer an ordering with ant parallel dipoles, 'g' will be smaller than unity. The Kirkwood correlations factor 'g' for a mixture, can be expressed as [15] follows:

$$\left[\frac{(\varepsilon_{0i} - \varepsilon_{\infty i})(2\varepsilon_{0i} + \varepsilon_{\infty i})}{9\varepsilon_{0i}}\right] = \frac{4\pi}{9kT} \sum_{i=1}^{2} g_{i} \rho_{i} \mu_{i}^{2}$$
(6)

Where i = 1,2 represent Propanol and Dioxane, respectively; μ_i -is corresponding dipole moment in gas phase, ρ_i -is the density, k-is the Boltzmann constant, T-is the temperature, ' ε_{0i} ' and ' $\varepsilon_{\infty i}$ ' are the static dielectric constant and dielectric constant at high frequency. ' g_i ' is the Kirkwood correlation factor for the ith liquid system. The interpretation of the dielectric phenomena in terms of the Kirkwood correlation factor is very difficult for mixture of associating compound.

(i) In the first model, we have assumed that the mixture can be represented by one correlation factor g^{eff} as follows:

$$\left\lfloor \frac{\left(\varepsilon_{0i} - \varepsilon_{\infty i}\right)\left(2\varepsilon_{0i} + \varepsilon_{\infty i}\right)}{9\varepsilon_{0i}} \right\rfloor = \left[\frac{4\pi}{9kT}\right]g^{eff}\left[\rho_{m}\mu_{m}^{2}\right]$$
(7)

To calculate g^{eff} values, we have taken dipole moment of 1P, 2P and DX as 2.26D, 2.2D and 0.97D respectively. The value ($\mathcal{E}_{\infty i}$) is taken as the square of the refractive index (R.I. =1.526 for 1P and 1.375for 2P). We believe that g^{eff} defined by eq. 7 is a reasonable measure of the molecular correlation. The observed value of $g^{\text{eff}} > 1$ in Propanol-Dioxane mixture, leads to the conclusion that the molecules associate to form multimer with parallel dipole moment. The high values of g^{eff} indicate average parallel orientation of electric dipoles in molecule. The plot of g^{eff} against the volume fraction of alcohol is as shown in fig. 9. Form fig. 9 it can be seen that as Propanol is added in Dioxane at regular steps in the mixture, the values of g^{eff} increase. The increase of correlation in P-P and P-DX mixtures may be due to the association effect. The correlation factor is essentially connected to the total orientation polarization and the dominating contribution to it, is embodied in $(\mathcal{E}_{0i} - \mathcal{E}_{\infty i})$. To calculate Kirkwood correlation factor (g^{eff}) from eq. 7 is not fully legitimate since in the investigating binary system, the P-P, P-Dx and Dx-Dx correlation exists. Figure 9 shows the variation of g^{eff} against volume fraction of Propanol, which indicates that as volume fraction of Propanol increases g^{eff} also increases. This indicates that the parallel of orientation of electric dipoles in molecule increases as volume fraction of alcohol increases.



Fig. 9. Plot of geff against volume fraction of Propanol

(ii) It is impossible to determine g_1 and g_2 form a single value of the static dielectric constant without some assumption. In the second model, the Kirkwood correlation factors for individual species i =1,2 are modified by assumed for P-Dx mixture that two kinds of intermolecular hydrogen bonds exist. One is the hydrogen bond between the P-P molecules; another is the hydrogen bond between the P-Dx molecules. These new correlations (g_1 and g_2) are described by the relation as follows [26]:

(8)

$$g_{1} = 1 + Z_{11} \cos \varphi_{11} + Z_{12} \cos \varphi_{12} \left(\frac{\mu_{2}}{\mu_{1}}\right)$$

$$g_{2} = 1 + Z_{21} \cos \varphi_{21} \left(\frac{\mu_{1}}{\mu_{2}}\right)$$
(9)

Where $Z_{11} = 2\langle n_{HB}^{11} \rangle$; $Z_{12} = 2\langle n_{HB}^{12} \rangle$; and $Z_{21} = 2\langle n_{HB}^{21} \rangle V_2/1 - V_2$ are the average number of particles forming the hydrogen bond with Propanol-Propanol and Propanol-dioxane pairs, respectively. V_2 ' is the mole fraction of alcohol. φ_{11} and φ_{21} are the angles between the neighboring dipoles of Propanol and dioxane molecules. The values of g_1 ' and g_2 ' for different Propanol -DX mixture are shown in fig. 10. The values of g_1 ' and g_2 ' increases as concentration of P in P-Dx mixture increases.



Fig. 10. Pot of g1 & g2 against volume fraction of Propanol

The average number of hydrogen bonds $\langle n_{HB}^{11} \rangle$, $\langle n_{HB}^{12} \rangle$ and $\langle n_{HB}^{21} \rangle$ per Propanol molecule for 1*i* pairs (*i*=1,2) have determined according to the following relation [26].

$$\left\langle n_{HB}^{1i} \right\rangle = \frac{n_{1i}\omega_{1i}}{n_1} \tag{10}$$

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Where, $\omega^{1i} = \frac{1}{1 + \alpha^{1i} e^{-\beta E^{1i}}}$ is the probability of bond formation between Propanol and dioxane. n_1 is the number density of dioxane molecules. The value of $\beta = \frac{1}{kT}$ and α^{1i} is the ratio of the two sub volume of the phase space, related to the non-hydrogen bonded and hydrogen bonded pairs. These hydrogen-bonded pairs have only two energy levels, E_{11} and E_{12} , for Propanol-Propanol and Propanol -Dioxane pair formed bonds, respectively. The values of $\langle n_{HB}^{11} \rangle$ and $\langle n_{HB}^{12} \rangle$ depend on the number of densities of hydrogen bonding pairs between Propanol-dioxane n_{12} and those between Propanol - Propanol molecule i.e. $n_{11}=2n_1$ - n_{12} . This can be calculated during which Propanol-Propanol (pair 1) and Propanol -dioxane (pair 2) are formed [26]. Fig. 11 shows plot of the average number of hydrogen bonds between Propanol - Propanol molecules (11 pairs) and Propanol -dioxane (12 pairs) against mole fraction of Propanol. It can be seen from the values of $\langle n_{HB}^{11} \rangle$ and $\langle n_{HB}^{12} \rangle$ depend on the concentration of Propanol in alcohol-dioxane mixtures increase which indicates solute solvent interaction increases.



Fig. 11. Plot of average number of hydrogen bonds between Propanol-Propanol molecules (n₁₁ pair) and Propanol-Dx (n₁₂ pair) against volume fraction of Propanol.

The different parameters required in the Luzar model are dipole moments, polarizability, possible number of hydrogen bonds and angles between dipoles $\cos \varphi_{11}$ and $\cos \varphi_{12}$ for the Propanol and Dioxane. The best possible values of molecular parameters in our analysis for which static dielectric constant values are in reasonable agreement with the experimental values are given in table 2.

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Molecular parameters	1-Propanol	2-Propanol	1,4-Dioxane
Dipole moment (μ_1 , μ_2)	2.26 D	2.2 D	0.97 D
Polarizability (α_1, α_2) (A ⁰³)	4.94	4.94	2.79
Bonding energy (E ₁₁ , E ₁₂) (kg/mol)	-13.98	-13.98	-16.25
Molecular weight (gm/mol)	60.11	60.11	88.11
Density (gm/cm ³)	0.7995	0.7811	1.028

Table 2 Molecular parameters used in computation of the static dielectric constant.

(11-P-P pair, 12-P-DX pair)

The Luzar model [26] gives a good qualitative account of the dielectric constant for P-Dx mixture at 25° C. The values of static dielectric constant obtained from the Luzar model and obtained from TDR technique are in good agreement. It should be noted that the Luzar applied the model to the water-dimethyl sulfoxide (DMSO) system and found that the experimental data can be explained by using large values of dipole moments in the liquids phase. We have also used the model to explain the static dielectric constant of mixture. We found that our experimental data can explained by the theory, provided values of the dipole moments of Propanol and Dioxane are large than the corresponding values of dipole moments in the gas phase. It can be seen that the theoretical values are in general, greater than the corresponding experimental values. This is due to the fact that the Luzar theory neglected formation of hydrogen bond interaction Dioxane-Dioxane (22 pair).

VIII. CONCLUSION

The complex permittivity for alcohol-dioxane are determined using TDR method. The dielectric constant for the mixtures can be explained using hydrogen-bonded model by assuming the formation of hydrogen bonds between alcohol-alcohol and alcohol-dioxane pairs. Thy orientation correlations between neighbouring molecules due to hydrogen bonding interaction are determined in terms of Kirkwood factors. The numbers of hydrogen bonds of alcohol-alcohol and alcohol-dioxane molecules are also computed.

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