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Research Article

High Frequency Dielectric Relaxation and Structural Characterization of 1-bromopropane - ethanol Mixture using a TDR

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ABSTRACT

Complex permittivity spectra of 1-bromopropane -ethanol mixture for the entire concentration range and at different temperatures (10, 15, 20 and 25°C) have been measured using Time Domain Reflectometry (TDR) technique from 10 MHz to 50 GHz. The spectra have been fitted to single Debye model using non-linear least square fit method from which we get values of dielectric constant and relaxation time (in ps). The negative excess permittivity value indicates that the total number of dipoles gets reduced. The negative values of excess inverse relaxation time $(1/\tau)^E$ for all temperatures indicates that dipoles rotate slowly in the mixtures due to the formation of more multimers. The enthalpy (ΔH), entropy (ΔS) and Gibb's free energy (ΔG) have been calculated for various concentration and temperature. The values of the Kirkwood correlation factors indicate that the parallel alignment of dipoles tends to reduce with rise in temperature and with increase in volume fraction of 1-bromopropane in ethanol. Numbers of hydrogen bonds between BMP–BMP and BMP–Ethanol pairs are estimated by using the Luzar model. The hydrogen bonding energies for BMP–BMP and BMP–Ethanol are estimated and found to be –13.3 and –10 kJ mol^{-1}.

Key words: Time Domain Reflectometry, Complex permittivity, Luzar model, Thermodynamic parameter, Hydrogen Bonding

Introduction

Liquid mixtures exhibit various phenomena, which cannot be found in pure substances. Among all the phenomenon's polarization and inter and intra molecular hydrogen bonding which may arise from the extra degree of freedom introduced by possibility of varying the concentrations of the binary liquid mixtures (Mehrotra, 2017; Hasted, 1973). Hydrogen bond plays an important role in many biological and chemical processes (Finneran, 2015) .

Liquid 1-bromopropane (*n*-propylbromide) abbreviated as BMP is an organobromine compound with the chemical formula $CH_3CH_2CH_2Br$. Like many other liquid halocarbons, BMP is used as an adhesive in aerosol glues, in aviation industry for maintenance and for synthetic fiber production. It is also useful to remove the soldering residues from electronic circuit boards. Animal studies suggest that BMP exposure may be associated with reduced blood

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cell counts and immunosuppressant along with toxicity to the liver and to the reproductive and neurologic systems. Ethanol, also called ethyl alcohol which is volatile, flammable and colorless solvent. It is a straight chain alcohol, psychoactive agent, best known as the type of alcohol found in alcoholic beverages. As ethanol is rapidly bactericidal it has been widely used for disinfection of skin, oral and rectal thermometers (Chambers, 2006). In recent years, using time and frequency domain technique there has been considerable advancement in theoretical and experimental research on dielectric properties of binary liquid mixtures. A time domain dielectric study plays an important role in gaining the knowledge and understanding of the inter-molecular interactions of binary mixture. This technique is also useful to study the strength of relaxation processes in liquids, which can be discussed in terms of dielectric properties (Kirkwood, 1939; Murthy, 1996). Thus, the aim of this study is to shed light on the high frequency complex dielectric permittivity, Kirkwood correlation parameters, and hydrogen bonding interactions, excess dielectric and relaxation properties and thermodynamic parameters of binary liquids.

Materials and Methods

Materials

The reagents such as BMP ($CH_3CH_2CH_2Br$, purity 98%) and ethanol (C_2H_5OH, purity 99.9%) were procured from Merck Life Sciences Pvt. Ltd. Mumbai and used without purification. The solutions were prepared at eleven discrete volume percentage of BMP from 0 to 100% in steps of 10%.

Measurements

The Tektronix Digital Serial Analyzer model no. DSA8300 sampling mainframe along with the sampling module 80E10B has been used for TDR. A sampling module provides 12ps incident and 15ps reflected rise time pulse was fed through a coaxial line system having 50 ohm impedance. Sampling oscilloscope monitors change in step pulse after reflection from the end of line. The reflected pulse without sample $R_1(t)$ and with sample $R_x(t)$ were recorded in the time window of 5ns and digitized in 2000 points. The Fourier transform of the pulse and data analysis was performed to determine complex permittivity spectra $\varepsilon^*(\omega)$ using least square fit method (Kumbharkhane 1991).

Results and Discussion

Complex permittivity

A frequency dependent complex permittivity spectrum of BMP-ethanol mixture with different concentration is shown in Fig. 1. It is observed that the values of dielectric permittivity (ε') decreases and dielectric loss peak (ε'') shifted towards higher frequency as concentration of BMP in ethanol increases. The shifting of loss peak towards higher frequency indicates the decrease of relaxation time in BMP rich region. It is observed that ethanol rich region shows maximum loss value (about 10) and BMP rich region (about 4).

Dielectric relaxation parameters of BMP-ethanol mixture are attained by using the Havriliak-Negami equation (Havriliak, 1966).

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_0 - \varepsilon_\infty}{[1+(j\omega\tau)^{1-\alpha}]^\beta} \qquad \ldots(1)$$

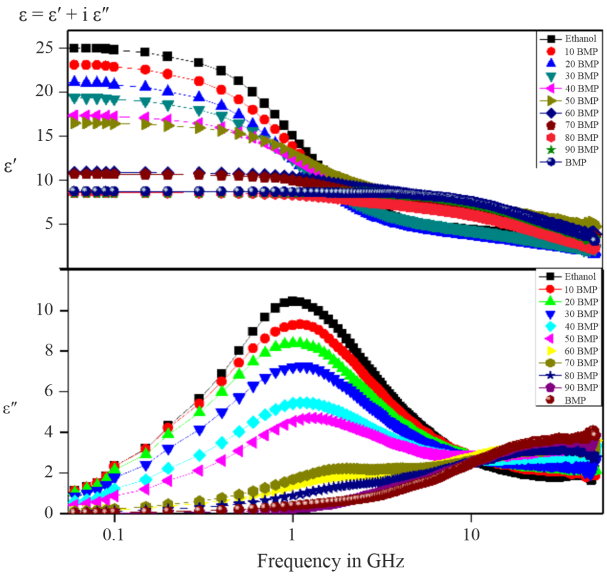

Fig. 1. Frequency dependent Complex dielectric permittivity Spectra of BMP-ethanol

where, $\varepsilon\varepsilon^*(\omega)$ is the complex permittivity having angular frequency ω, ε_0 is the static dielectric constant, ε_∞ is the permittivity at high frequency, τ is relaxation time, α and β are the empirical fitting parameters having the values in between 0 and 1. The complex permittivity spectra are fitted in Debye model (α =0 and β=1) to obtain the dielectric relaxation parameters.

The values of static permittivity (ε_0), relaxation time (τ) at various concentrations and temperatures are recorded and shown in Fig. 2 and Fig. 3, respectively. It is seen that, values of static dielectric constant are decreasing with increasing temperature and with the increasing volume fraction of BMP in ethanol. The decrease in the dielectric constant may be due to increase of thermal oscillation of the molecules and increase in degree of disorder of dipoles.

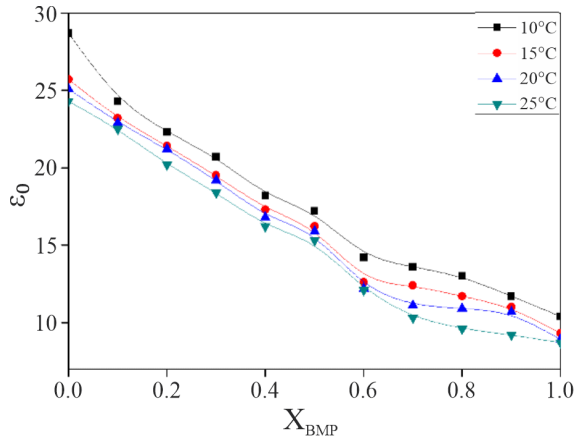

Fig. 2. Static Dielectric constant of BMP-ethanol

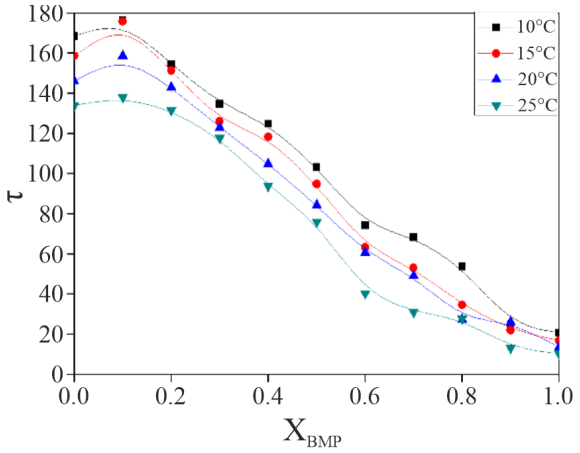

Fig. 3. Relaxation time of BMP-ethanol

The variation of relaxation time with increase in volume fraction of BMP at different temperatures presents an interesting behavior. When 0.1 volume fraction of BMP is added into ethanol, the relaxation time of solution suddenly increases. This indicates bulky structure formation at this concentration of BMP in the solution (Undre, 2007). The higher relaxation time may be due to more association through H- bonding of –OH group in ethanol (Kamble, 2008). On further increase of BMP in the solution, the relaxation time values decrease linearly towards pure BMP value.

Excess parameters

The excess inverse relaxation $\left(\frac{1}{\tau}\right)^E$ may provide the structural information. In present study it is determined for the BMP – Ethanol solution as follows (Fakhar 2015):

$$\left(\frac{1}{\tau}\right)^E = \left(\frac{1}{\tau}\right)_M - \left[\left(\frac{1}{\tau}\right)_E X_E + \left(\frac{1}{\tau}\right)_B (1 - X_E)\right] \quad \ldots(2)$$

where, $\left(\frac{1}{\tau}\right)^E$ is the excess inverse relaxation time, which represents the average broadening of dielectric spectra. The inverse relaxation time analogy is taken from spectral line broadening in the resonant spectroscopy (Mehrotra, 1977). The variation of excess relaxation time $\left(\frac{1}{\tau}\right)^E$ with volume fraction BMP at 25p C is shown in Fig. 4. The inverse relaxation time values are negative for all concentrations for both systems studied indicates slower rotation of the dipoles which produces a field in such a way that the effective dipole rotation is hindered due to the solute–solvent interaction forming the H bonded complex.

The contribution of hydrogen bonds to the dielectric properties of the associating mixtures is studied in terms of excess permittivity. The excess permittivity ε_0^E is defined as (Ingole, 2018).

$$\varepsilon_0^E = (\varepsilon_0)_M - \left[(\varepsilon_0)_E X_E + (\varepsilon_0)_E (1 - X_E)\right] \quad \ldots(3)$$

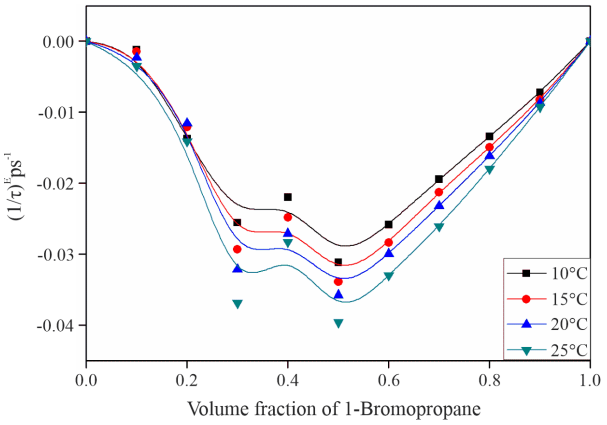

Fig. 4. Excess inverse relaxation time $(1/\tau)^E$ Vs. volume fraction of BMP in ethanol at different temperatures

where, the subscript M, E and B represent mixture, ethanol, BMP respectively and X_E represent the volume fraction of ethanol in mixture. Mehrotra *et al*. mentioned that the change in value of dielectric permittivity with concentration is because of interaction between dissimilar molecules, which may produce structural changes. In present study, excess permittivity has negative value at all concentration of BMP-ethanol, suggests strong intermolecular interaction due to hyper conjugation in these mixture which leads to decrease in total number of dipoles (Carey, 1990; Pawar, 2002; Zeberg-Mikkelsen, 2005). Also, from Fig. 5 it is observed that the excess dielectric permittivity is more negative at $X_E \approx 0.3$ volume

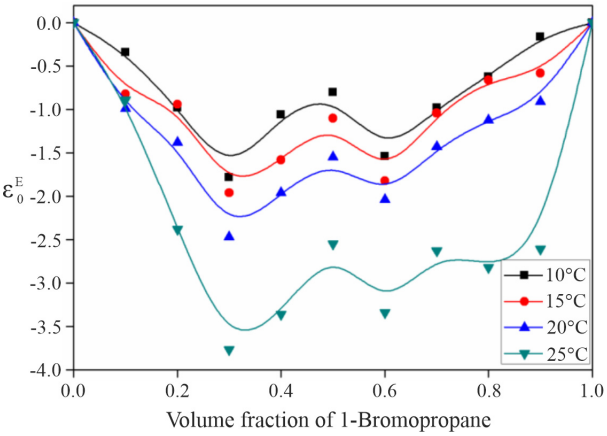

Fig. 5. Excess dielectric permittivity (ε_0^E) vs. volume fraction of BMP in ethanol at different temperatures

fraction of BMP at all temperatures. This indicates that at this concentration there is an aggregation of multiple molecules held together through hydrogen bonding.

Thermodynamic parameters

Eyring was the first one who correlates the dielectric relaxation to the chemical rate theory. According to this theory the following relation was carried out (Eyring, 1936):

$$\tau = \frac{h}{KT} \exp \frac{\Delta G}{RT} \qquad \ldots(4)$$

where, ΔG is the free energy of activation for dipole relaxation, K is the Boltzmann's constant, and h is Planck's constant.

The Gibb's free energy (ΔG) is related to enthalpy of activation (ΔH) and the entropy of activation (ΔS) by following relation:

$$\Delta G = \Delta H - T^* \Delta S \qquad \ldots(5)$$

Equation (4) and (5) indicate that the plot of log(τT) versus (1000/T) which is represented in Fig. 6 should give approximately a linear relationship with a slope ((ΔH/R), from which ΔH can be calculated (Glasstone 1941).

From Table 1, it can be noticed that enthalpy of activation (ΔH) for ethanol and BMP is found to be 8.35 kJ/mole and 9.37 kJ/mole respectively. All values of enthalpy of activation are positive suggesting the endothermic interaction (Hashem, 2005). All the entropy value of ethanol, BMP and their binary mixture seems to be positive signifies

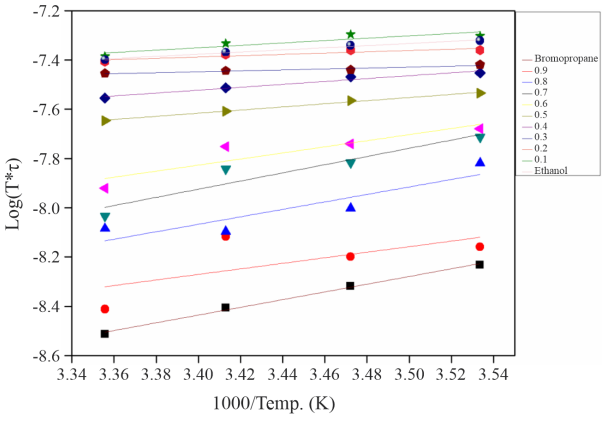

Fig. 6. Arrhenius behaviour of BMP-ethanol

Table 1. Enthalpy, Entropy of activation and Gibb's free energy for BMP-ethanol

Volume fraction of BMP	Enthalpy of activation ΔH (kJ mol^{-1})	Entropy of activation ΔS (J mol^{-1} K^{-1})	Gibb's free energyat 25°C ΔG (kJ mol^{-1})
0.0	8.35 (7)	0.201 (2)	8.29
0.1	9.24 (3)	0.204 (1)	9.18
0.2	5.08 (1)	0.190 (5)	5.00
0.3	3.62 (7)	0.187 (2)	3.55
0.4	11.25 (1)	0.214 (5)	11.14
0.5	12.18(6)	0.219 (5)	12.10
0.6	9.27 (2)	0.217 (8)	9.17
0.7	13.18 (1)	0.232 (1)	13.11
0.8	12.10 (1)	0.232 (4)	12.02
0.9	9.13 (1)	0.224 (5)	9.05
1.0	9.37(8)	0.226 (3)	9.30

Note: Numbers given in parenthesis represents uncertainties in the least significant digits obtained by least square fit method. e.g. ΔH 8.35(7) means 8.35±0.07

that the arrangement of molecules in the activated state becomes relatively less ordered (Garbarczyk, 2003). According to the principle of thermodynamics, the equilibrium state of a thermodynamic system is decided by Gibbs free energy of activation (ΔG). Gibbs free energy of activation for ethanol and BMP are found to be 8.29 kJ/mol and 9.30 kJ/mol respectively. So, it can be asserted that dipoles of BMP require more energy than dipoles of ethanol in order to attain the equilibrium. The free energy of activation values are less than the minimum activation energy needed for breaking of the hydrogen bonds (Sarode, 2011; Sengwa, 2003).

Kirkwood factor

The Kirkwood-Froehlich equation for pure liquid gives the useful information regarding orientation of electric dipole. It can be represented as (Sengwa, 2009).

$$\frac{(\varepsilon_0-\varepsilon_\infty)(2\varepsilon_0+\varepsilon_\infty)}{\varepsilon_0(\varepsilon_\infty+2)^2}=g\mu^2\frac{4\pi N\rho}{9kTM} \qquad \ldots(6)$$

where, μ is the dipole moment, ρ is the density, M is the molecular weight, k is the Boltzmann constant, N is Avogadro's number, ε_0 and ε_∞ are the static dielectric constant and dielectric constant at high frequency respectively and g is the Kirkwood correlation factor which is measure of degree of short range dipole ordering due to hydrogen bond interactions.

For the binary mixtures, the effective Kirkwood correlation factor (g^{eff}) gives information of dipole-dipole correlation in associating binary mixture of polar liquids by the modified effective Kirkwood equation (Kirkwood 1939).

$$\frac{4\pi N}{9KT}\left[\frac{\mu_E^2\rho_E V_E}{M_E}+\frac{\mu_B^2\rho_B(1-V_E)}{M_B}\right]\times g^{eff}=\frac{(\Xi\epsilon_{om}-\epsilon\Xi_{\infty m})(2\epsilon\Xi_{om}+\epsilon\Xi_{\infty m})}{\epsilon\Xi_{om}(\Xi_{\infty m}+2)^2} \qquad \ldots(7)$$

where, g^{eff} is the effective Kirkwood correlation factor, μ_E and μ_B are the dipole moments of ethanol and BMP respectively, N is Avogadro's number, ρ_E and ρ_B are the densities of ethanol and BMP respectively, ε_0 is static dielectric constant, ε_∞ is permittivity at high frequency, K is Boltzmann's constant, M_E and M_B are the molecular weights of ethanol and BMP respectively and T is the temperature.

The values of the effective Kirkwood correlation factor g^{eff} given in Table 2 indicate that the parallel alignment of dipoles tends to reduce with rise in temperature and with increase in volume fraction of BMP in ethanol. At 25°C estimated values of effective Kirkwood correlation factor (g^{eff}) for ethanol and BMP are

Table 2. Kirkwood correlation factor for BMP in ethanol

Volume fraction of BMP	25°C			20°C			15°C			10°C		
	g^{eff}	g_1	g_2	g^{eff}	g_1	g_2	g^{eff}	g_1	g_2	g^{eff}	g_1	g_2
0	3.16		1.38	3.21	-	1.42	3.23	-	1.48	3.57	-	1.52
0.1	2.88	2.06	1.38	2.89	2.06	1.42	2.88	2.06	1.48	2.97	2.06	1.52
0.2	2.55	1.99	1.26	2.63	2.00	1.27	2.62	2.01	1.28	2.68	2.02	1.29
0.3	2.28	1.93	1.20	2.35	1.94	1.21	2.35	1.96	1.20	2.46	1.98	1.21
0.4	1.97	1.85	1.17	2.01	1.87	1.17	2.04	1.89	1.17	2.12	1.90	1.17
0.5	1.83	1.78	1.14	1.88	1.80	1.14	1.88	1.81	1.14	1.98	1.83	1.14
0.6	1.40	1.70	1.11	1.39	1.72	1.11	1.42	1.73	1.11	1.59	1.76	1.11
0.7	1.15	1.62	1.08	1.24	1.64	1.08	1.38	1.66	1.08	1.50	1.68	1.08
0.8	1.05	1.53	1.05	1.20	1.55	1.05	1.28	1.56	1.06	1.41	1.58	1.06
0.9	0.99	1.43	1.03	1.16	1.44	1.03	1.18	1.46	1.03	1.24	1.47	1.03
1	0.82	1.30		0.84	1.31	-	0.90	1.32	-	1.12	1.33	-

3.16 and 0.82 respectively. Value of BMP is close to unity shows the insufficient interaction due to anti-parallel orientation of electric dipoles between pure BMP molecules.

For a mixture of associated compounds, the interpretation of dielectric phenomena in terms of the Kirkwood correlation factor is very difficult. It is impossible to separate average correlation factors g_1 and g_2 from a single value of static dielectric constant without any assumptions. So, Kirkwood–Froehlich theory has been applied to the media containing two species of molecules and the cross-correlation terms must be taken into consideration to separate g_1 and g_2. This theory is also useful to consider the H-bond contribution to dipole-dipole correlation. In the present method, the Kirkwood correlation factors g_1 and g_2 are modified, and these new correlations are described as follows:

$$g_1 = 1 + Z_{11}COS\Phi_{11} + Z_{12}COS\Phi_{12}(\mu_2/\mu_1) \quad \ldots(8)$$

$$g_1 = 1 + Z_{21}COS\Phi_{21}\,(\mu_1/\mu_2) \quad \ldots(9)$$

where, Φ_{12} and Φ_{21} are the angles between the neighboring dipoles of BMP and ethanol molecules. $Z_{11} = 2<n\frac{11}{HB}>$, $Z_{12} = 2<n\frac{12}{HB}>$ and $Z_{21} = 2<n\frac{21}{HB}>\,(1\text{-}X_{BMP})/X_E$ are the average number of H-bonds with BMP-BMP and BMP-ethanol pairs respectively. X_E and X_{BMP} are the volume fraction of Ethanol and BMP respectively. The temperature effect on the values of g_1 and g_2 at different concentrations is shown in Table 2. The values of g_1 and g_2 for different BMP- ethanol mixtures are calculated using the parameters given in Table 3. A good qualitative account of

Table 3. Molecular parameters used in computation of the Static Dielectric Constant ($å_0$)

Molecular parameters	10°C	15°C	20°C	25°C
Effective Dipole moment[a] of 1-BMP	2.03	2.09	2.13	2.18
Effective Dipole moment[a] of ethanol	1.64	1.68	1.71	1.73
Polarizability[b] of 1-BMP	13.6	12.5	12.3	12.1
Polarizability[b] of ethanol	7.3	5.8	5.5	5.4
Bonding energy[c] of 1-BMP-1-BMP	-13.3	-13.3	-13.3	-13.3
Bonding energy[c] of 1-BMP-ethanol	-10	-10	-10	-10
Enthalpy[c] of 1-BMP-1-BMP	28	28	28	28
Enthalpy[c] of ethanol-ethanol	41	41	41	41
Number of H-Bond	2	2	2	2

[a]Unit : Debye; [b]Unit : A^{03}; [c]Unit: kJ/mol

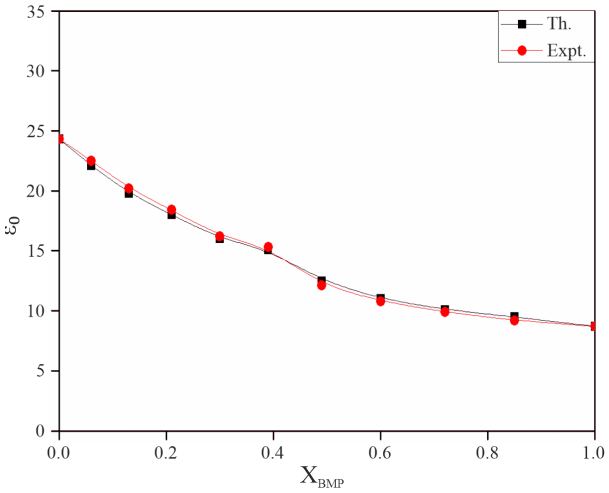

Fig. 7. Theoretical and experimental ε_0 vs. volume fraction of BMP at 25°C

theoretical static dielectric constant with experimental dielectric constant (Luzar, 1990) for BMP-ethanol mixtures at 25°C is shown in Fig. 7.

The values of $<n\frac{11}{HB}>$, $<n\frac{12}{HB}>$ depend on the number of densities of H-bonding pair between pure BMP molecules and between BMP and ethanol respectively. This can be calculated during which BMP-BMP (11 pairs) and BMP-ethanol (12 pairs) are formed. As shown in Fig. 8, it is seen that at around $X_{BMP} \approx 0.25$, the average size of cooperative domain (CD) and the average number of H-bonds between BMP and

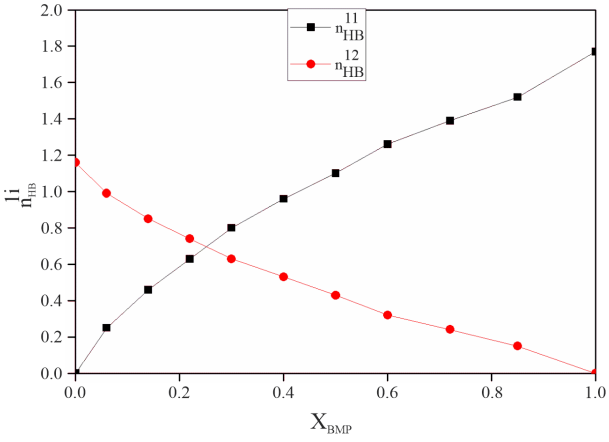

Fig. 8. Average number of H-bonds between BMP-BMP (n_{11} pair) and BMP-ethanol (n_{12} pair) vs. volume fraction of BMP at 25°C

ethanol molecules are nearly equal. At $0<X_{BMP}<0.25$ BMP molecules interact with surrounding ethanol molecules by H-bonding and these molecules form CD_{12}. For $X_{BMP} > 0.25$, the decrease in the number of ethanol molecules with increasing X_{BMP} leads to a decrease in the average number of hydrogen bonds between ethanol and BMP molecules, which reflected in the decrease in the fraction of CD_{12}. The value of $n\frac{11}{HB}$ is 1.77 at $X_{BMP} = 1$ which suggests that one BMP molecule interacts with nearly ≈ 2 BMP molecules by hydrogen bonding and approximately two hydrogen bonds have been predicted.

Conclusions

The complex dielectric relaxation spectra of BMP in ethanol solution has been studied using time domain technique in the frequency range 10 MHz to 50 GHz. The intermolecular interactions among BMP-ethanol molecules are confirmed by the Kirkwood correlation factor, excess inverse relaxation, excess dielectric permittivity and thermodynamic parameters. The negative values of excess dielectric properties suggest that the hydrogen bonding interaction among BMP-ethanol mixtures is such that there is slower rotation of dipoles and effective dipoles get reduced. The positive values of enthalpy ensure the endothermic reaction. The positive entropy of activation indicates that the activated state is more disordered. Gibb's free energy value of BMP is more than ethanol which specifies that the dipoles of BMP require more energy in order to attain equilibrium. The Kirkwood correlation factor indicates that the parallel alignment of dipoles tends to reduce with the rise in temperature and increase in volume fraction of BMP in ethanol.

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