

# Dielectric Relaxation Study of Aqueous Amides in Non-Polar Solvent Using Picoseconds Time Domain Technique

G. R. Mahajan and A. C. Kumbarkhane

**Abstract**—The dielectric permittivity of amide - 1, 4-dioxane (DX) for various concentrations has been studied in the frequency range from 10 MHz to 30 GHz using the time domain reflectometry (TDR) method. A hydrogen bonded theory suggested by Luzar is applied to compute correlation terms for the mixtures. Static permittivity for the mixtures can be explained using the Luzar model by assuming the formation of two types of hydrogen bonded dimmers, one between amide-amide (pair 1) and other between amide-1, 4-dioxane (pair 2). The numbers of these pairs are also estimated as a function of concentration.

**Index Terms**— Dielectric permittivity, relaxation time, time domain reflectometry (TDR), bruggeman factor

## I. INTRODUCTION

Amide represents an important class of bio-organic solvent. The hydrogen bond formation ability of these solvents is useful in understanding the peptide linkage in complex bio-systems [1]-[3]. The study of hydrogen bonding molecular interactions of binary mixtures is currently a significant challenge in the research field of science and technology. Hydrogen bond constitutes a very interesting class of intermolecular interactions, which are of extreme importance in many fields of chemistry and molecular biology. "Hydrogen Bonding in Biological Structures" can be used in many ways by biophysicists [4]. The study of the H-bonds of the type O=H --- O=C occupies a position of considerable importance as it relates to the study of biopolymers [5]. In the case of amides, N, N-dimethylformamide (DMF) is able to form the H-bonded network structures with dipolar aprotic and protic solvents [6]. *Malathi M. et al.* have studied mutual interaction of amides with non polar solvents to understand the conformational stability of protein molecules [7]. Thenappan and Sankar were studied hydrogen-bonded complexes of alcohol with N, N-dimethylformamide (DMF) [8]. DMF is a widely used solvent for many recently developed synthetic procedures because of its powerful solvating properties [9]. N-methylacetamide (NMA) is the most frequently used analog compound for the peptide unit in

proteins. Paul Firman and S. Sahoo were studied the mixture of N-methylacetamide (NMA) with non-polar solvent [10], [11]. The non polar solvent 1, 4-dioxane (DX) is used as a laboratory reagent and as a solvent for the production of cellulose acetate, ethyl cellulose, benzyl cellulose, dyes, oils, fats and polyvinyl polymers. The intermolecular interaction plays an important part in intermolecular recognition processes essential to most of biological systems. It also found that Physical and chemical phenomenon of binary systems are closely related to the intermolecular interactions.

In the present work, we report a comparative dielectric relaxation study of amide (DMF and NMA) with non polar solvent (DX) at various concentrations at 25°C employing Time Domain Reflectometry (TDR) in the frequency range of 10 MHz-30 GHz. The complex permittivity spectra of DMF-DX and NMA-DX have been fitted to Debye model and Davidson – Cole model respectively. The static permittivity, dielectric relaxation time and Kirkwood correlation factor has been obtained. The experimental results of static dielectric constant have been used to estimate the average number of hydrogen bonds between solvent – solvent and solute – solvent molecules.

## II. EXPERIMENTAL

### A. Materials

N, N-dimethylformamide (DMF) and N-methylacetamide (NMA) was obtained commercially from s d fine-chem Ltd. (AR Grade 99%). The solutions were prepared by mixing the 1, 4-dioxane (DX) and amides in volume.

### B. Measurements

The densities of solutions were measured using specific gravity bottle and weighting were carried out by using electronic balance. The uncertainties of the measured values of densities were  $\pm 10^{-4}$  gram per  $\text{cm}^3$ . The dielectric complex permittivity of the mixtures was measured by TDR [12]. The Tektronix model no. DSA8200 Digital Serial Analyzer sampling mainframe along with the sampling module 80E08 has been used. The details of the apparatus and data analysis procedures of the TDR have been reported previously [13].

## III. RESULTS AND DISCUSSION

Fig.1 (a, b) shows frequency dependent complex permittivity spectra for DMF–DX and NMA–DX at 25°C. It can be seen from Fig. 1 (b) that the position of the peak is

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shifted towards lower frequency for NMA system.

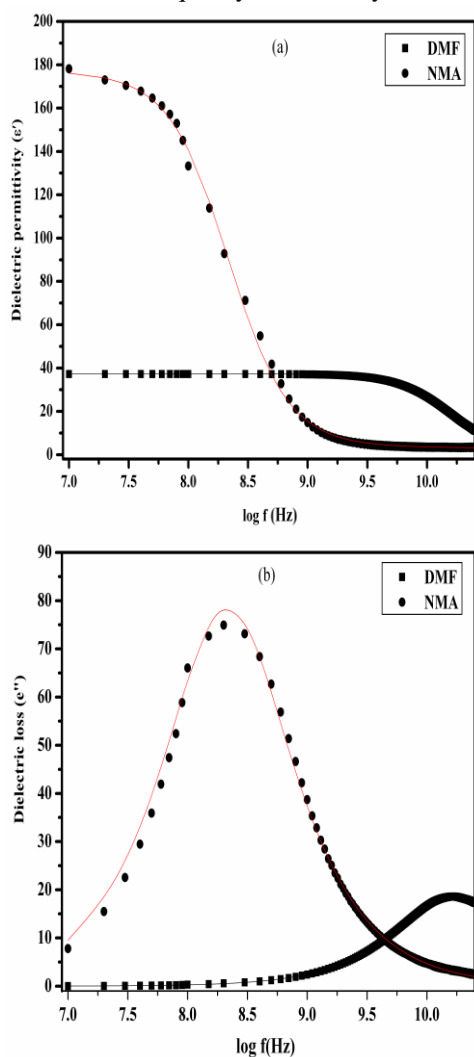


Fig. 1. Frequency dependent (a) dielectric permittivity ( $\epsilon'$ ) and (b) dielectric loss ( $\epsilon''$ ) for DMF and NMA at 250C.

The complex permittivity spectra measured using TDR is fitted by the non-linear least squares fit method to the Havriliak - Negami expression [14], [15]:

$$\epsilon^*(\omega) = \epsilon_\infty + \frac{\epsilon_0 - \epsilon_\infty}{[1 + (j\omega\tau)^{1-\alpha}]^\beta} \quad (1)$$

where  $\epsilon_0$  is the static dielectric constant,  $\epsilon_\infty$  is the permittivity at high frequency,  $\tau$  is relaxation time and  $\alpha$  and  $\beta$  are the distribution parameters. Equation (1) includes the Debye ( $\alpha = 0, \beta = 1$ ), Cole-Cole ( $0 \leq \alpha \leq 1$  and  $\beta = 1$ ) and Davidson - Cole ( $\alpha = 0$  and  $0 \leq \beta \leq 1$ ). For the system DMF-DX, the values of  $\alpha$  and  $\beta$  are found to be 0 and 1, respectively; whereas NMA-DX system,  $\beta$  is less than 1.

The contribution of hydrogen bonds to dielectric properties of the mixture can also be studied by using the hydrogen bonding model as suggested by Luzar [16]. We have used the Luzar model to explain static permittivity of the mixture. Considering only the hydrogen bond contribution to dipole-dipole correlation, the dielectric constant for binary mixtures can be determined using Kirkwood-Frohlich equation [17]

$$\frac{(\epsilon_{0i} - \epsilon_{\infty i})(2\epsilon_{0i} + \epsilon_{\infty i})}{9\epsilon_{0i}} = \frac{4\pi}{9kT} \sum_{i=1}^2 g_i \rho_i \mu_i^2 \quad (2)$$

where  $i=1$  and 2 represent solute and solvent, respectively. Here  $\mu_i$  is the corresponding dipole moment in the gas phase,  $\rho_i$  is the density,  $k$  is Boltzmann constant,  $T$  is the temperature and  $g_i$  is the Kirkwood correlation factor for the  $i^{\text{th}}$  liquid component. It is also impossible to separate the average correlation factor  $g_1$  and  $g_2$  from single value of the static dielectric constant without any assumptions. Luzar suggested theoretical model based on mean field approximation for hydrogen bonded mixture [16]. The correlation factor  $g_1$  and  $g_2$  are calculated by following equations:

$$g_1 = 1 + Z_{11} \cos \phi_{11} + Z_{12} \cos \phi_{12} (\mu_2 / \mu_1) \quad (3)$$

$$g_2 = 1 + Z_{21} \cos \phi_{21} (\mu_1 / \mu_2) \quad (4)$$

where  $Z_{11} = 2\langle n_{\text{HB}}^{11} \rangle$ ,  $Z_{12} = \langle n_{\text{HB}}^{12} \rangle$  and  $Z_{21} = \langle n_{\text{HB}}^{12} \rangle X_{\text{DX}} / (1 - X_{\text{DX}})$  are the average number of hydrogen bond with solvent - solvent and solvent - solute pairs respectively.  $\phi_{11}$  and  $\phi_{21}$  are the average angles between neighboring dipoles of solvent and solute molecule. Since 'g' is measure of the molecular association between a reference molecule and its nearest neighbors, the departure of 'g' from unity can be indicative of molecular association. The values of  $g_1$  and  $g_2$  for different DMF-DX and NMA-DX mixtures are computed by using the parameters given in Table 1 and are shown in Figure 2. The values of  $g_1$  and  $g_2$  depend on the concentration of DX in DMF-DX and NMA-DX mixtures. This model gives a good qualitative account of the static dielectric constant of the binary mixtures (DMF-DX and NMA-DX) along with the experimental values determined from Time Domain Reflectometry technique (TDR) in our laboratory, and are shown in Figure 3 respectively. The value of static dielectric constant and relaxation time at 25°C for DMF and NMA are reported in Table 2. In case of secondary amine (NMA) the single bond character is decreases and double bond character is increased. Due to this the rotation of molecules are slower and relaxation time is more as compared to DMF.

TABLE I: MOLECULAR PARAMETERS USED IN COMPUTATION OF THE STATIC DIELECTRIC CONSTANT ( $\epsilon_0$ ).

Molecular parameters	
Dipole moment of DMF in Debye	3.22
Dipole moment of NMA in Debye	6.05
Dipole moment of DX in Debye	0.75
Polarizability of DMF in $\text{\AA}^3$	4.08
Polarizability of NMA in $\text{\AA}^3$	7.82
Polarizability of DX in $\text{\AA}^3$	5.68
Binding energy of Amide - Amide in kJ/mol	-14.39
Binding energy of Amide - DX in kJ/mol	-18.25
Statistical volume ratio for Amide - Amide	28
Statistical volume ratio for DX-DX	40

TABLE II: STATIC DIELECTRIC CONSTANT AND RELAXATION TIME AT 25°C.

	DMF	NMA
Static dielectric constant ( $\epsilon_0$ )	37.22	179.54
Relaxation time ( $\tau$ )	10.43	736.40

The average number of hydrogen bonds  $\langle n_{\text{HB}}^{11} \rangle$ ,  $\langle n_{\text{HB}}^{12} \rangle$  and  $\langle n_{\text{HB}}^{21} \rangle$  per DX molecule for  $1i$  pairs ( $i = 1, 2$ ) has been determined using following relation [16]:

$$\langle n_{\text{HB}}^{1i} \rangle = n_{1i} \omega^{1i} / n_1 \quad (5)$$

where  $\omega^{1i} = 1/[1 + \alpha^{1i} \exp(\beta E^{1i})]$  is the probability of bond formation between solvent and solute.  $n_1$  is the number density of solvent molecules,  $\beta = 1/kT$ , and  $\alpha^{1i}$  are the statistical volume ratios of the two sub volumes 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 11 and 12 pair formed bonds, respectively. The values of  $\langle n_{\text{HB}}^{11} \rangle$  and  $\langle n_{\text{HB}}^{12} \rangle$  depend on the number densities of the hydrogen-bonded pairs between solvent - solvent and solvent - solute molecules respectively. Fig.4. shows a plot of the average number of hydrogen bonds between amide-amide and amide-1, 4-dioxane against the mole fraction of 1, 4-dioxane.

We calculated the average number of hydrogen-bonded solvent - solvent pairs  $[n_{\text{HB}}^{11}]_V$  and solvent - solute pairs  $[n_{\text{HB}}^{12}]_V$  per unit volume (per  $\text{cm}^3$ ) using the following equations [18]:

$$[n_{\text{HB}}^{11}]_V = \frac{C_{\text{SOLVENT}} \rho_{\text{mix}} N_A}{M_{\text{SOLVENT}}} n_{\text{HB}}^{11} (/ \text{cm}^3)$$

and (6)

$$[n_{\text{HB}}^{12}]_V = \frac{C_{\text{SOLVENT}} \rho_{\text{mix}} N_A}{M_{\text{SOLVENT}}} n_{\text{HB}}^{12} (/ \text{cm}^3)$$

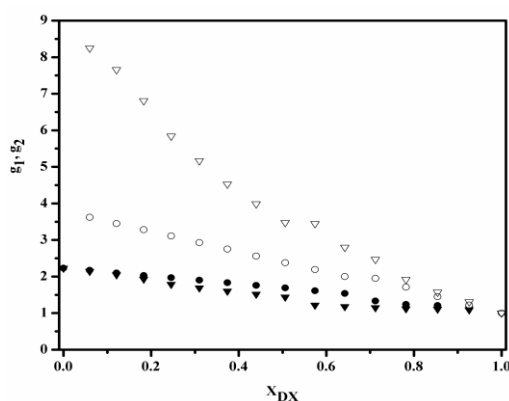


Fig. 2. Mole fraction of 1, 4-dioxane dependence of the  $g_1$  (closed symbols) and  $g_2$  (open symbols)  $\blacktriangledown$  - N-methylacetamide  $\bullet$  - N-dimethylformamide at  $25^\circ\text{C}$ .

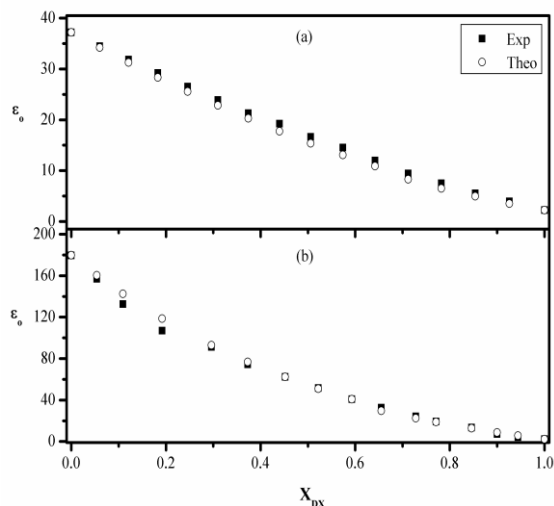


Fig. 3. Comparison of experimental and theoretical static dielectric constant for (a) DMF-DX and (b) NMA-DX at  $25^\circ\text{C}$ .

where  $C_{\text{SOLVENT}}$  is the weight fraction of solvent,  $\rho_{\text{mix}}$  (gram per  $\text{cm}^3$ ) is the density of mixture,  $N_A$  is the Avogadro number, as  $6.02 \times 10^{23}$  (per mol) and  $M_{\text{SOLVENT}}$  is the molecular weight of solvent.

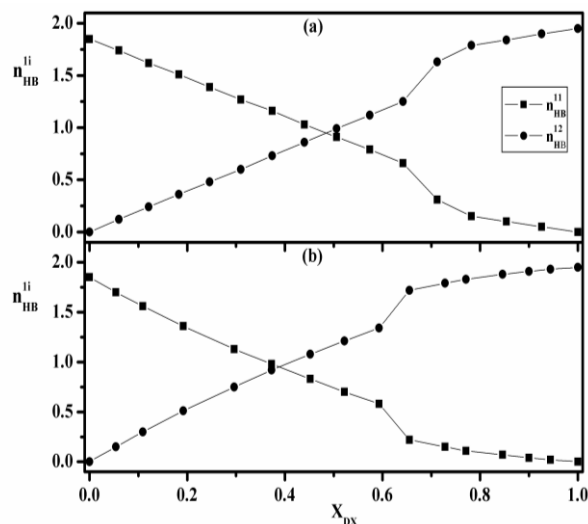


Fig. 4. Plots of the average number of hydrogen bonds  $[n_{11}$  pair and  $n_{12}$  pair] for (a) DMF-DX and (b) NMA-DX vs. mole fraction of 1, 4-dioxane ( $X_{\text{DX}}$ ) at  $25^\circ\text{C}$ .

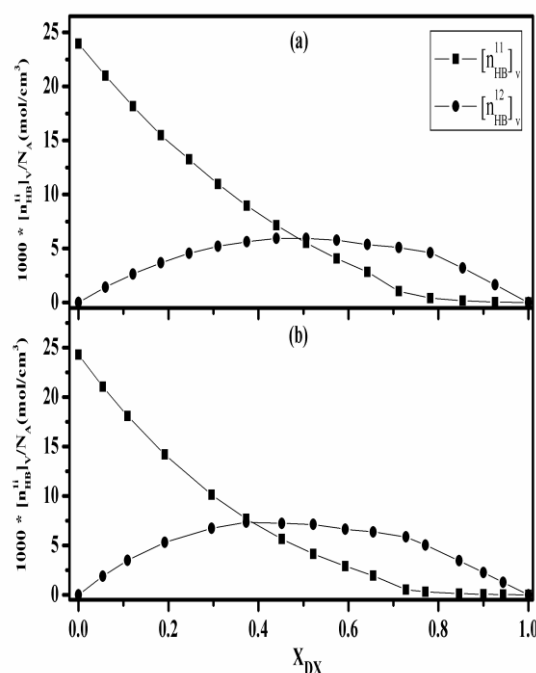


Fig. 5. Plots of the number of hydrogen bonds per unit volume against  $X_{\text{DX}}$  for (a) DMF-DX and (b) NMA-DX at  $25^\circ\text{C}$ .

Fig. 5 shows the plots of  $[n_{\text{HB}}^{11}]_V$  and  $[n_{\text{HB}}^{12}]_V$  against mole fraction of 1, 4-dioxane ( $X_{\text{DX}}$ ). The value of  $[n_{\text{HB}}^{11}]_V$  increases with increasing  $X_{\text{DX}}$  and that of  $[n_{\text{HB}}^{12}]_V$  has a maxima at concentration  $X_{\text{DX}} \approx 0.50$  for DMF-DX and  $X_{\text{DX}} \approx 0.37$  for NMA-DX. These results provide information regarding the intermolecular interaction of solvent - solvent and solvent - solute molecules.

#### A. Excess Dielectric Constant

The contribution of hydrogen bonds to the dielectric properties of the mixture is also studied in terms of the excess static dielectric permittivity. The excess permittivity ( $\epsilon_0$ )<sup>E</sup> can be written as [19]:

$$(\epsilon_0)^E = (\epsilon_0)_M - [(\epsilon_0)_{DX} X_{DX} + (\epsilon_0)_{SOLVENT}(1 - X_{DX})] \quad (7)$$

where the subscripts M, DX and SOLVENT represents mixture, 1, 4-dioxane and DMF/NMA respectively. Also  $X_{DX}$  represents mole fraction of DX in solvent. The plot of excess permittivity vs. the mole fraction of DX is shown in Figure 6. The values of excess permittivity for amide-DX binary mixtures are negative over the entire concentration range which indicates that apolar co-solvent DX acts as a structure breaker for self associated amides. Thus the amides dipoles are oriented in opposite direction which reduce the total number of parallel aligned effective dipoles. The negative peak value of excess permittivity is observed at  $X_{DX} \approx 0.50$  for DMF-DX and  $X_{DX} \approx 0.37$  for NMA-DX which indicates that the formation of maximum complexes through H-bonding at this concentration.

The magnitude of  $\epsilon_0^E$  value is the verification of the strength of unlike molecules H-bond interactions. It means a higher value represents stronger and long-range H-bond molecular interactions between unlike molecules and vice-versa. Thus the interactions in NMA-DX mixture are comparatively stronger than DMF-DX mixture.

Also the dependence of maximum magnitude of excess permittivity on the value of dielectric constant ( $\epsilon_0$ ) and Kirkwood correlation factor ( $g$ ) of the pure amides. As the  $\epsilon_0$  and  $g$  values of the pure amide are increases then the strength of H-bond interaction in mixture also increases.

#### B. Bruggeman Factor

The static permittivity of binary mixtures can be obtained by using the Bruggeman mixture formula [20]:

$$f_B = \left[ \frac{(\epsilon_{0m} - \epsilon_{02})}{(\epsilon_{01} - \epsilon_{02})} \right] \left( \frac{\epsilon_{01}}{\epsilon_{0m}} \right)^{1/3} = 1 - V_{DX} \quad (8)$$

where ( $f_B$ ) is the Bruggeman dielectric factor. The  $\epsilon_{0m}$ ,  $\epsilon_{01}$  and  $\epsilon_{02}$  are the static dielectric constants corresponding to mixture, solvent (DMF/NMA) and solute (DX) respectively. ( $V_{DX}$ ) is the volume fraction of DX. Bruggeman expression predicts a linear relationship between  $f_B$  and the volume fraction of DX but the experimental values of  $f_B$  shows a non linear behavior (Figure 7). To explain the non linear relationship the eq. (8) is modified as follows [21]:

$$f_B = \left[ \frac{(\epsilon_{0m} - \epsilon_{02})}{(\epsilon_{01} - \epsilon_{02})} \right] \left( \frac{\epsilon_{01}}{\epsilon_{0m}} \right)^{1/3} = 1 - [a - (a-1)V_{DX}]V_{DX} \quad (9)$$

where 'a' is arbitrary parameter, the value of  $a=1$  implies the ideal mixture with no interaction between solute and solvent. Experimental values represent the deviation from ideality of mixture which indicates the existence of hydrogen bonding interaction between solute – solvent mixtures. The values of 'a' for DMF-DX and NMA-DX mixtures are determined to be

1.00 and 1.77 respectively, under experimental errors.

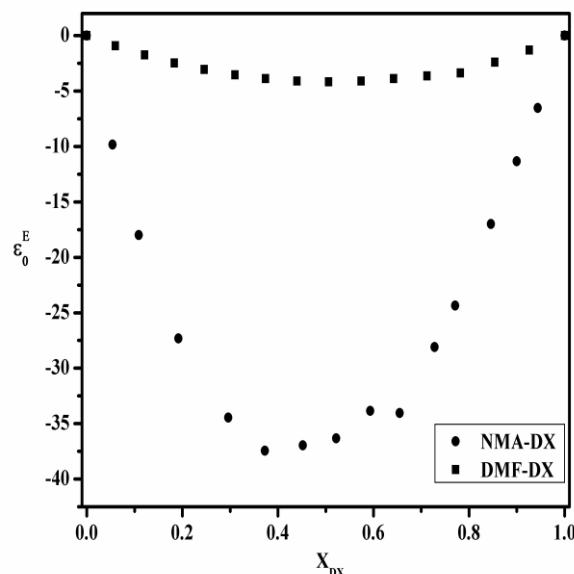


Fig. 6. Excess permittivity vs. mole fraction of 1, 4-dioxane ( $X_{DX}$ ) for DMF – DX and NMA – DX mixture at 25°C.

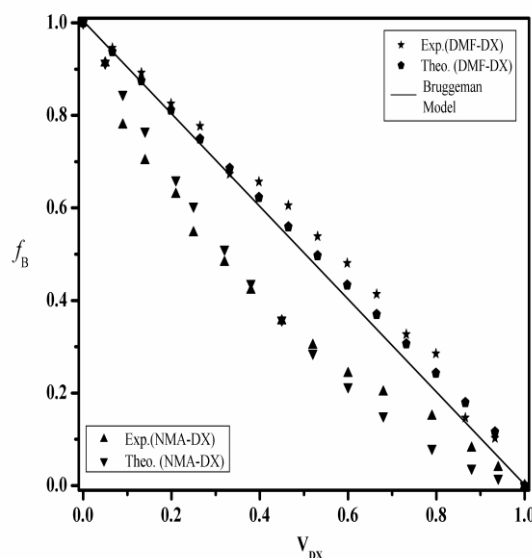


Fig. 7. Bruggeman dielectric factor ( $f_B$ ) Vs. mole fraction of 1, 4-dioxane ( $V_{DX}$ ) for DMF – DX and NMA – DX mixture. Solid line represents according to Bruggeman model.

#### IV. CONCLUSIONS

The temperature dependent complex permittivity spectra of amide - 1, 4-dioxane mixtures have been studied using time domain reflectometry technique in the frequency range 10 MHz to 30 GHz. The deviation from ideality in Kirkwood correlation factor, excess dielectric properties and Bruggeman dielectric factor provides information regarding hydrogen bonding interactions in mixtures. The number of hydrogen bonds of DMF-DX and NMA-DX molecules increases with increasing DX concentration and maximum at  $X_{DX} \approx 0.50$  and  $X_{DX} \approx 0.37$ , respectively. The Kirkwood correlation factors and relaxation times of NMA-DX system are found to be larger than the corresponding values of DMF-DX system, indicating stronger hydrogen bonding in NMA-DX system. By using recently developed Luzar theory

one may get quantitative information about solute-solvent interactions.

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