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Section B: Dielectric Properties

Hydrogen Bonding Interaction between Amide and Alcohols: Dielectric Relaxation and FTIR Study

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Abstract

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The dielectric relaxation study for the hydrogen bonded binary system of polar liquid N,N-dimethylacetamide in butanol and pentanol, has been carried out at 11 concentrations over the frequency range of 10 MHz–20 GHz at 303 K using TDR. The least squares fit method has been used to obtain the static dielectric constant (ϵ_0) and relaxation time (τ). By using dielectric parameters the excess permittivity (ϵ^E), excess inverses relaxation time $(1/\tau)^E$, Kirkwood Correlation factor (g) are also obtained to explore the hydrogen-bonded hetero-molecular interactions. Conformational analysis of the hydrogen bond between the two systems is supported by the FTIR spectra.

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Q Keywords: Dielectric relaxation excess parameter Kirkwood correlation factor Time Domain Reflectometry FTIR

1. Introduction

Dielectric relaxation studies have been undertaken of binary mixtures of polar molecules under varying conditions of compositions to help in formulating adequate models of liquid relaxation and also in obtaining information about the relaxation process in mixtures. Hydrogen bonding is complex in the liquid state because of the uncertainty in identifying the particular bonds and the number of molecules involved. The presence of hydrogen bonds brings a considerable change in the dielectric properties of liquid mixtures such as dipole moment, dielectric constant, relaxation time, Kirkwood correlation factor, excess dielectric constant, and excess inverse relaxation time. The hydrogen bonding interaction between -OH with -OH [1], -OH with -NH [2-11], -OH with -C = O [12-20], -OH with -S = O [21] and FTIR study [22-28] of various molecules investigated earlier. The present paper reports the dielectric relaxation study of N,N-dimethylacetamide with primary alcohols viz. butanol (BU) and pentanol (PE) mixture using Time Domain Reflectometry technique in reflection mode at 303 K.

The aim of this study is to provide the precision dielectric constant and relaxation time values of these mixed solvents and to confirm the nature of H-bonded unlike molecular structures and the effect of the extent of substitution in amide and the number of alcohol molecules -OH groups on the hetero-molecular H-bonding interactions. Conformational analysis of the formation of hydrogen bonds is carried out from FTIR study.

2. Experimental

AR grade 1-butanol (BU), 1-pentanol (PE) and N,N-dimethylacetamide (DMA) were purchased from Aldrich with purity > 99% and used as received. The solutions were prepared at eleven different volume percentage of DMA in alcohols from 0% to 100% in steps of 10%, at 303 K.

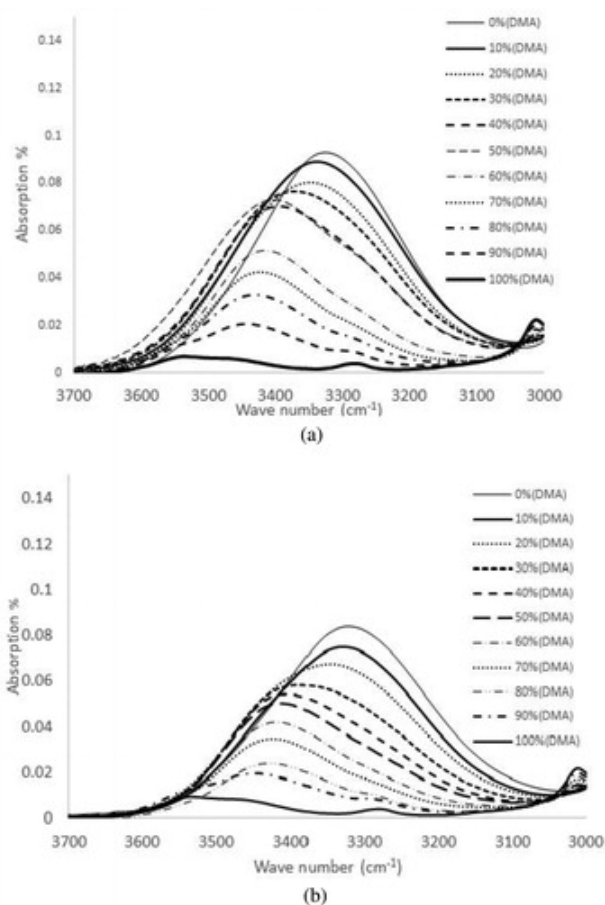
In the present study the FTIR spectra of all the samples were recorded using a Bruker Alpha-T

permittivity spectra were obtained by using a time domain reflectometry method. All details are already described elsewhere [29].

3. Result and Discussion

The [0:1] complex formation (DMA): the spectra of pure DMA can be assigned to the vibration of C = O, C-H, and N-H bands. The carbonyl stretching fundamental occurs in a range that is reasonably narrow, $1750 \pm 150 \text{ cm}^{-1}$ [30]. N-H stretching vibration is broad and appears at 3536.89 cm^{-1} . C-H stretching vibration band absorption is appearing at 2932.17 cm^{-1} . The C = O stretching vibration absorption band is narrow and appears at 1634.76 cm^{-1} . The bending vibrations of N-H are appearing in the range $1548.18\text{--}1497.11 \text{ cm}^{-1}$. The absorption band (Figure 1a) of C-O stretching vibration is observed in the range $1058.60\text{--}1010.44 \text{ cm}^{-1}$.

Figure 1. FTIR Absorption spectra of O-H (a) DMA with BU and (b) DMA with PE.



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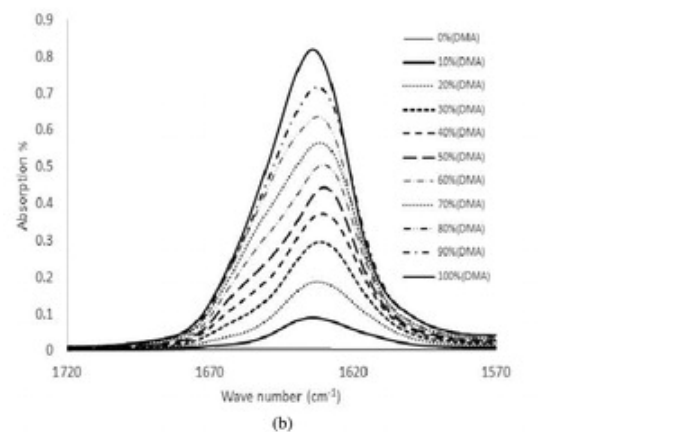
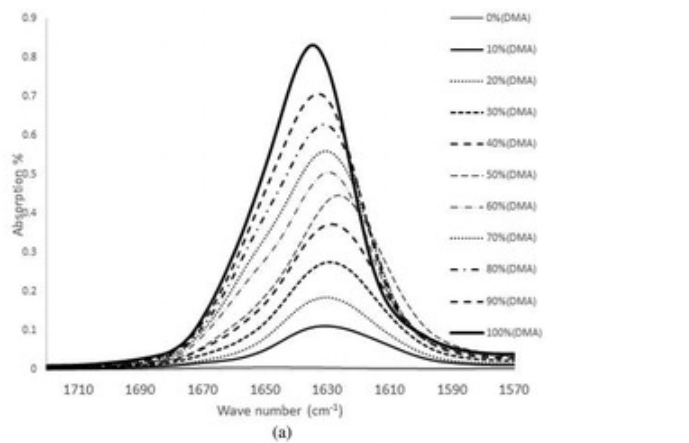
In [1:1] complex formation it is observed that N–H stretching vibration shifted towards high wave number (135.11 cm^{-1}). This suggests that the alcohols interact with amide through the hydroxyl group of alcohol and carbonyl group of amide. The alkyl chain-length of alcohols plays an important role in the determination of the strength of hydrogen bond (O–H: C = O) [31].

The absorption spectra of BU and PE are represented in [Figure 1a and b](#), respectively. The [1:0] complex formation (BU): the absorbed spectra of hydrogen bonded (O–H stretching) group observed at 3325.88 cm^{-1} . The C–H stretching observed at 2930.65 cm^{-1} . The bending vibration of C–H absorbed at 1377.28 cm^{-1} . The [0:1] complex formation (PE): the absorbed band of (O–H stretching) group observed at (3500 cm^{-1} – 3300 cm^{-1}), which is attributed to the stretching frequency of O–H bond in alcohol. The absorbed spectra of hydrogen bonded (O–H stretching) was observed at 3323.13 cm^{-1} . The band at 2935.74 cm^{-1} referred to the stretching frequency (wave number) of C–H bond. C–O stretching in alcohols produces strong band at 1051.74 cm^{-1} . Alcohol produces characteristic infrared bands due to O–H stretching, C–H stretching and C–O stretching. The absorption band of O–H bond is broad (due to hydrogen bonding) and very strong absorption (due to the highly polar nature) which derives from the stretching vibration of the hydroxyl group. The characteristic bands observed in the spectra of alcohols result from O–H stretching and C–O stretching.

In [1:1] complex formation it is observed that the N–H stretching vibration and O–H stretching vibrations shifted to high wave number when concentrations of DMA increase in DMA-PE mixture. This suggests that there is strong intermolecular interaction like hydrogen bonding between the oxygen in the carbonyl group of DMA and hydrogen in hydroxyl group of PE.

The C = O stretching vibration bond in DMA-BU and DMA-PE mixtures reported in [Figure 2a and b](#), respectively. From these figure it is observed that the C = O stretching absorption band shifted towards higher wave number and it is more in case of DMA-BU (8.34 cm^{-1}) than DMA-PE (4.13 cm^{-1}). This indicates the hydrogen bonding between O–H and C = O group [32].

Figure 2. FTIR Absorption spectra of C = O **(a)** DMA with BU and **(b)** DMA with PE.



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The complex permittivity spectra $\epsilon^*(\omega)$ [33] were obtained from reflection coefficient spectra $\rho^*(\omega)$ by using calibration method [34]

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

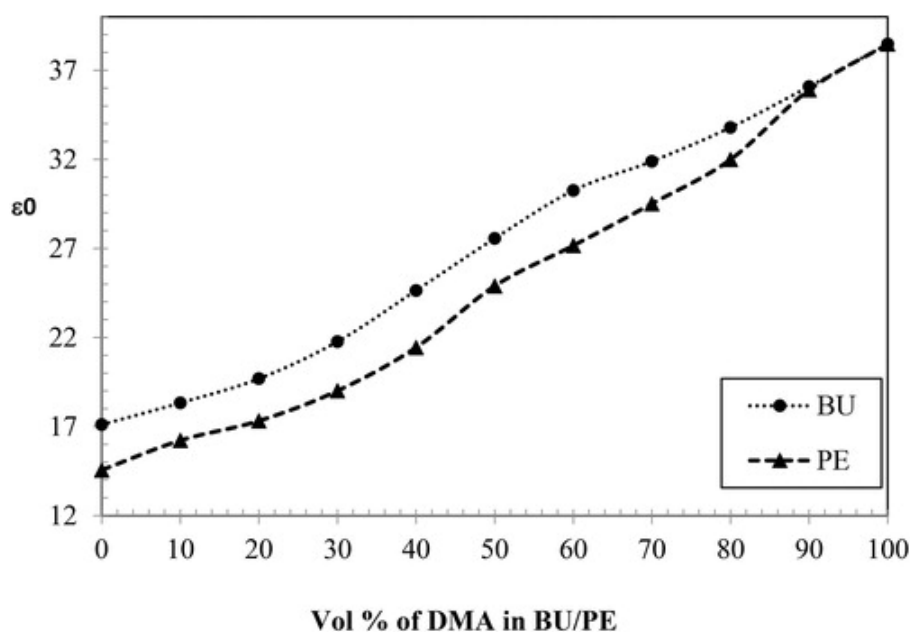
(1) where ϵ_0 , ϵ_∞ and τ are fitting parameters. In Eq. (1), ϵ_0 is the static dielectric constant; ϵ_∞ is dielectric constant at high frequency, ω is the angular frequency and τ is the relaxation time of the system. Using these values the excess dielectric constant (ϵ^E) [2, 3], excess inverse relaxation time $(1/\tau)^E$ [2, 3], Kirkwood correlation factor (g^{eff}) [35] and corrective Kirkwood correlation factor (g_f), were determined.

Variation in ϵ_0 , τ , ϵ^E , $(1/\tau)^E$, g^{eff} and g_f as a function of volume fraction of DMA in BU/PE at 303 K represented in Figures 3–8, respectively. The small difference in ϵ_0 values of BU and PE confirms that the increase in molecular size of alcohols decreases the ϵ_0 values. The g values greater than unity confirm that these alcohol molecules in their pure liquid state exist in intermolecular H-

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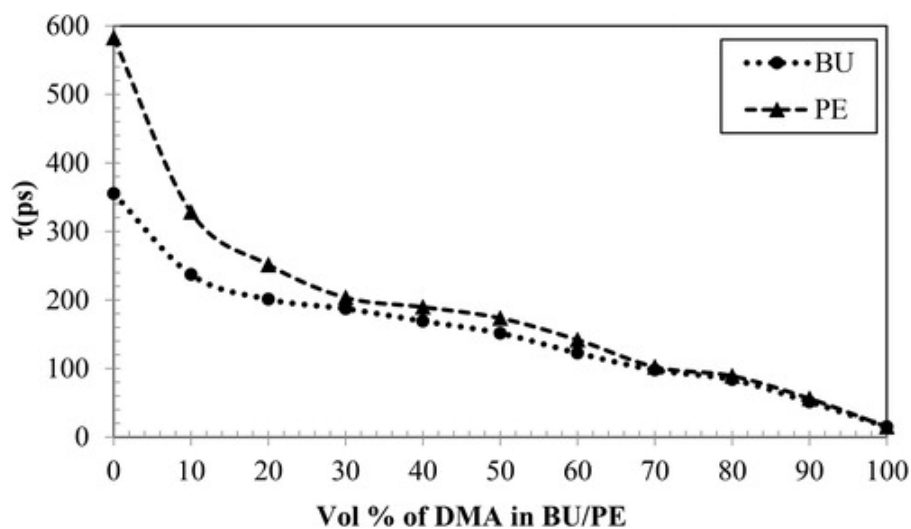
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Figure 3. Variation of dielectric constant (ϵ_0) as a function of volume percentage of DMA in DMA-BU and DMA-PE at 303 K.



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Figure 4. Variation of relaxation time (τ) as a function of volume percentage of DMA in DMA-BU and DMA-PE at 303 K.

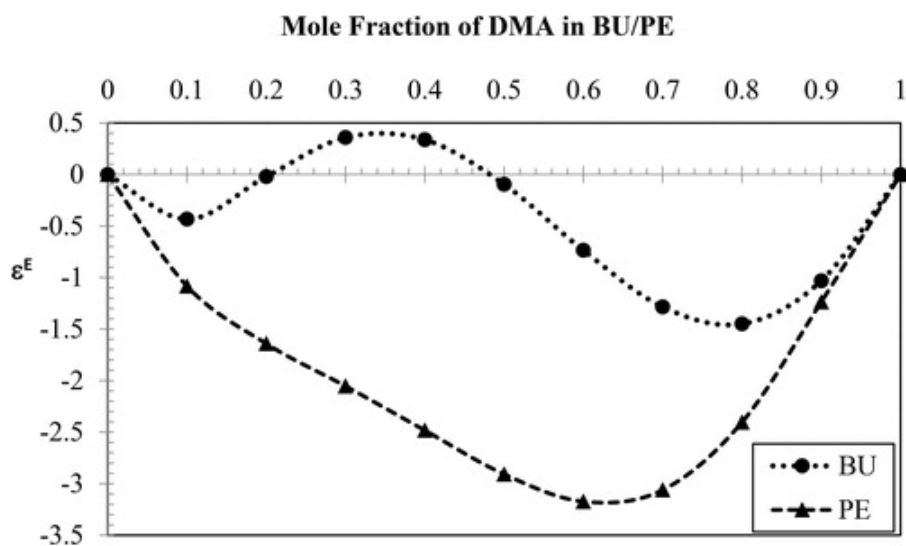


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Figure 5. Variation of excess permittivity (ϵ^E), as a function of mole fraction of DMA in DMA-BU and DMA-PE at 303 K.

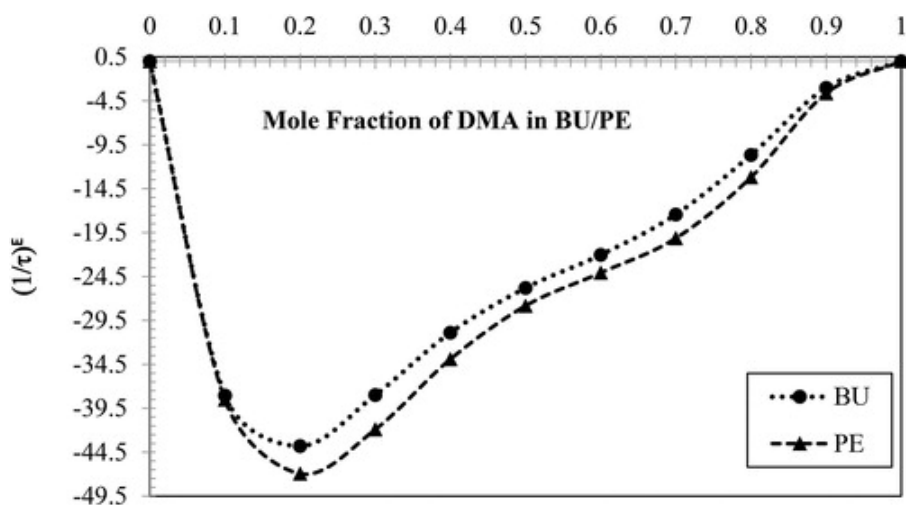
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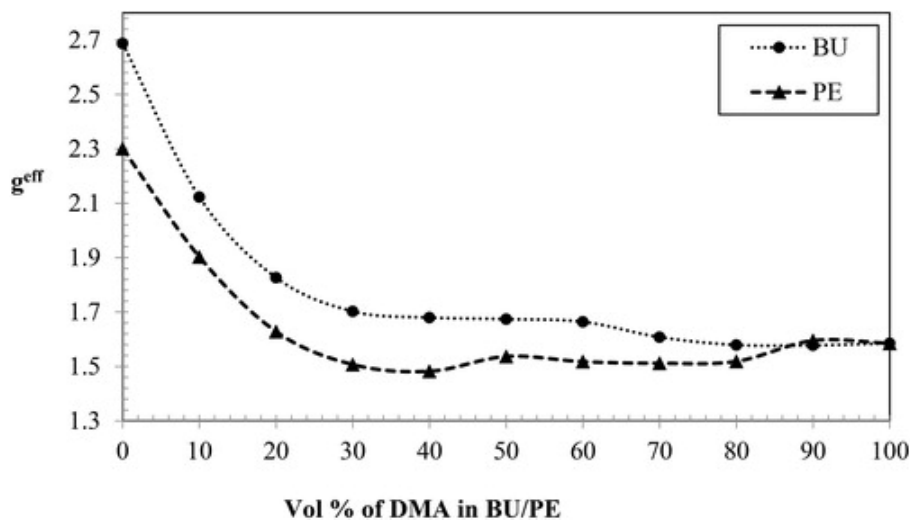
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Figure 6. Variation of excess inverse relaxation time $(1/\tau)^E$, as a function of mole fraction of DMA in DMA-BU and DMA-PE at 303 K.



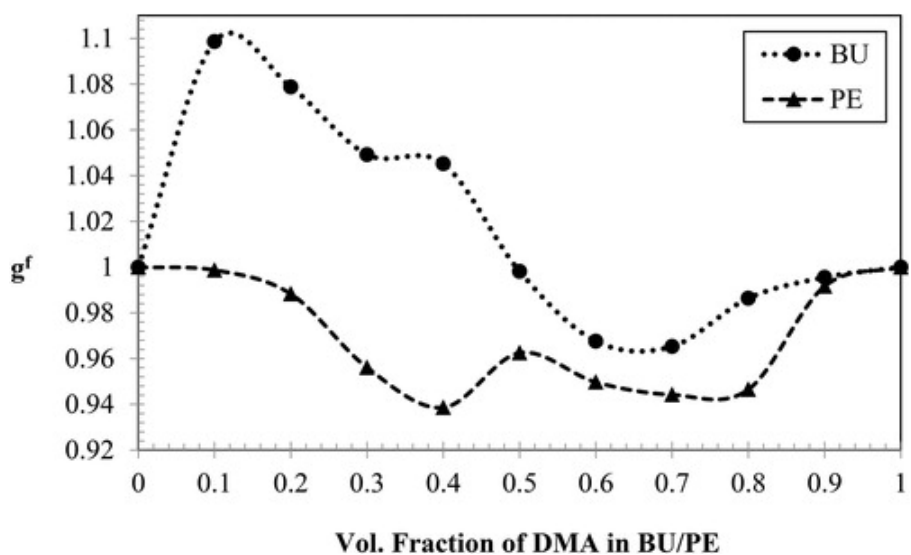
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Figure 7. Variation of g^{eff} , as a function of volume percentage of DMA in DMA-BU and DMA-PE at 303 K.



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Figure 8. Variation of g^f , as a function of volume fraction of DMA in DMA-BU and DMA-PE at 303 K.



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The values of ϵ_0 increase and τ , and g^{eff} decrease as the percentage of amide in alcohol increases for both the systems. This suggests that the intermolecular association is taking place in all these systems. Similar results were reported by Khirade et al. [36] for DMF-alcohol mixtures.

From Figure 3, it is found that the values of ϵ_0 for BU, PE, and their binary mixtures with DMA are in the order: BU > PE for concentrations studied. This trend can be ascribed to the decrease

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linear behavior of concentration dependent ϵ_0 values of DMA-BU and DMA-PE binary mixtures is the experimental evidence of unlike molecular interactions such as H-bonding, dipole-dipole and dipole-induced dipole interactions in these mixed solvents. The deviation in ϵ_0 values from ideal behavior in mixed solvents is mainly due to H-bond hetero molecular interactions with dipolar orientation [2-6, 9-22].

It is observed from the Figure 4 that, the values of τ for BU, PE and their binary mixture with DMA increase in the order: $\tau_{BU} < \tau_{PE}$. This indicates that the higher alkyl chain length of alcohols hinders the rotation of the molecule which leads to increasing relaxation time are noticed. This may be linearly correlated with the variations of viscosity and molecular size of the alcohols. From this result, it may be concluded that the strength of hydrogen bonding is linearly increased with chain length of alcohols [38]. The dielectric constant for the mixtures increases towards the corresponding values of DMA. Decrease of dielectric constant is due to the transition of elongated aggregates into spherical molecular aggregates.

The negative ϵ^E values also confirm that the DMA and alcohol molecules (except at 0.3 and 0.4 vol. fraction of DMA in BU) are apt to coalesce and form hetero-molecular structures by breaking some of the self-associated structures. It is observed that the magnitude of ϵ^E values of DMA-PE mixtures is higher than the DMA-BU mixtures, which is evidence of the formation of comparatively stronger interactions between DMA and PE molecules. Increasing concentrations of DMA, a negative (ϵ^E) deviation is noticed. This indicates the formation of multimer through hydrogen bonding, which leads to a decrease in total number of dipoles in the systems studied.

The relaxation time (Figure 4) shows continuous increase with chain length of alcohols offers hindrance to the rotation of the molecule. The increase in relaxation time with chain length (τ , of BU = 355.27 ps and PE = 582.27 ps) is to be expected in view of the fact that the hydroxyl group reorientation depends to some extent on the length of the alkyl group, and the viscosity of the liquid. The relaxation time increases with increasing alkyl chain length, which indicates that the degree of cooperativity for reorientation of the molecules increases with increasing length and the bulk of the cluster increases. The relaxation time increases with increasing chain length in agreement with the fact that the relaxation time is directly related to the size of the molecules [39]. From Figure 4, it is observed that the relaxation time values gradually decrease with increase in mole fraction of DMA in BU and PE. Increase in mole fraction of DMA in BU/PE, decrease in relaxation time is a manifestation of the dielectric orientation of the clusters containing both DMA

the heterogeneous mixture.

From [Figure 6](#), it is found that the $(1/\tau)^E$ values of DMA-BU and DMA-PE binary mixtures are negative over the entire concentration range. The negative values of $(1/\tau)^E$, which also confirms the enhance in breaking of DMA structure and form the hydrogen bonded structures which produces a field in such a way that the effective dipole rotation is hindered [[9](#)].

The evaluated value of Kirkwood correlation factor for BU and PE $g = 2.68$ and 2.30 , respectively also confirms the existence of H-bonded molecular multimers with parallel dipolar ordering alignments in their pure state. The g^{eff} values ([Figure 7](#)) of the DMA-BU/PE mixed solvents decrease from the g value of one solvent to the g value of another pure solvent over entire concentration range. It is found that the g^{eff} values have negative deviation from ideality, confirms the net decrease in dipolar ordering due to H-bond complexation.

The corrective Kirkwood correlation factor g_f of the investigated mixtures deviate from unity ([Figure 8](#)) confirming the change in net dipolar ordering. The plots of g_f values of DMA-BU and DMA-PE mixtures have good resemblance with their corresponding ϵ^E plots i.e. the mixtures of which have g_f values less than unity, their ϵ^E values are negative and vice-versa ([Figure 5](#)). The g_f values are less than unity, which suggest that the addition of small amount of DMA breaks large amount of H-bonded long-range ordered BU and PE structures.

4. Conclusions

The hydrogen bonding interaction in the binary mixture of butanol and pentanol in N,N-dimethylacetamide were studied using FTIR and dielectric relaxation technique. The dielectric constants, the relaxation times, the Kirkwood correlation factor, excess permittivity and excess inverse relaxation times for binary mixtures of DMA-BU and DMA-PE at various concentrations at 303 K have been reported. The dielectric parameters show systematic change with concentration DMA in BU and PE. The excess static permittivity and excess inverse relaxation time values are negative for both systems indicating that solute-solvent interaction hinders the dipole rotation in the system. From this study, it is confirm that the strength of hydrogen bonding is depending on the alkyl chain length of alcohol. With stronger intermolecular bonding, broad and very intense bands are observed. In general the bands become broader

increasing polarity of the OH bond which might therefore be expected to show a greater rate of change of dipole during the vibration.

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