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RESEARCH ARTICLE

Dielectric relaxation and dipole moment of binary mixture of 2, 3-dichloroaniline and 2-ethoxyethanol in 1, 4-dioxane solution using microwave absorption data

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ABSTRACT

Dielectric constant (ε′) and dielectric loss (ε″) of 2,3-Diachloroaniline (2,3-DCA),2-Ethoxyethanol (2-EE) and binary mixtures of 2,3-DCA+2-EE in 1,4-dioxane solutions were calculated by using standard standing microwave X-band technique were measured at microwave frequency 10.985 GHz at different temperatures 20^0C, 30^0C, 40^0C and 50^0C using Gopala Krishna's single frequency concentration variation method. The measured values of ε′ and ε″ have been used to evaluate dipole moment (μ) and relaxation time (τ). The dielectric relaxation process of binary mixtures containing 71% mole fraction of 2,3-DCA were calculated at the respective given temperatures. On the basis of observations, it is found that the dielectric relaxation process can be treated as the rate process like the viscous flow process. Non-linear variation of relaxation time with molar concentration of 2,3-DCA in the whole concentration range of the binary mixture indicates the existence of solute-solute and solute–solvent type of molecular association were predicted. Dipole moment (μ) of 2,3-DCA and 2-EE non linearly decreases with rise in temperature. Excess inverse relaxation time $(1/\tau)^e$ calculated at different temperatures are found to be positive.

Keywords: Dielectric relaxation, Binary mixture, 2,3-dichloroaniline, 2-Ethoxyethanol, Microwave absorption.

INTRODUCTION

Over the past few years, the use of microwave heating for promoting organic chemical transformations and in the development of microwave tomography images systems for medical applications increased [1]. The successful application of microwave frequency range is directly associated with the dielectric properties of the materials. The characterization of dielectric materials may include the measurement of complex permittivity as function of frequency at a given temperature or as a function of temperature at a given frequency. The measurement of dielectric properties in a wide frequency range can provide information on the conduction mechanism, interfacial polarization and molecular dynamics [2].

Dielectric relaxation phenomena of binary mixture of amides dissolved in non-polar solvent less than 10 GHz X-band electric field are of special interest to research workers [3-5] since long. The various molecular association as well as structure, shape and size of the polar molecules can be ascertained through the relaxation phenomenon which is one of the most unresolved problems of physics to day [6]. Schallamach [7] proposed that two polar liquid mixtures involving separate absorption centre exhibit sufficiently different resolutions. They may show molecular relaxation of sufficiently larger composition having indistinguishable, overall and average single absorption centre. 2,3-Dichloroaniline (2,3-DCA) is used starting material for synthesis of bioactive Schiff's bases, azetidinones, thiazolidinones, pyrazolines, acetohydrazides and in coupling reactions. It is used in preparation of poly (2,3-dichloroanline-Co-anline) which control conductivity in broad range from 10^{-9} to 10^{-2} S/cm. Also it is used in preparation in dyes, azo-dyes, isocynates and in plant protection agent [8]. 2-Ethoxyethanol (2-EE) [9] commercially known as "Cello solves" and widely used as complexes of solvents, co emulsifiers, stabilizers of emulsions, varnish removers, degreasing solutions, used in printing industries and an additive for jet fuel to prevent ice build-up.

The purpose of the present study is also to see the applicability of Debye-Smyth model in the case of the binary polar mixture 2,3-DCA+2-EE in 1,4-dioxane [10]. The polar-non polar mixture of 2, 3-DCA in 1, 4-dioxene exhibits relaxation time due to rotation of the whole and the flexible parts of molecules in their effective dispersion region [11] of 10.985GHz electric field. Thus it is worthwhile to study the temperature variation data of 2,3-DCA+2-EE in 1,4-dioxane under 10.985 GHz electric field to know the molecular dynamics of the system.

MATERIALS

2,3-Diochloroaniline (GC Grade) is obtained from Sigma-Aldrich. 2-Ethoxyethanol and 1,4-dioxane (AR Grade) are purchased from M/S Sd. Fine chemical, Mumbai, India. Without further purification the two liquids 2,3-DCA+2-EE according to their proportions by volume are mixed well and kept 6h in well stoppered bottles to ensure good thermal equilibrium. These liquids are used as solute and solvent.

EXPERIMENTAL

The X-band microwave bench is used to measure wavelength in the dielectric medium and voltage standing wave ratio (VSWR) using a short-circuiting plunger. The set up is tuned at microwave frequency 10.985 GHz. The experimental techniques of Heston *et.al* [12] for microwave measurements are used. All the measurements are carried out at temperatures 20^0C, 30^0C, 40^0C and 50^0C by circulating ethyleneglycol+water around the dielectric cell and temperature is thermostically controlled with $\pm$ 0.5^0C using Nevitech pvt. Ltd. Mumbai India. The whole of the equipment is standardized with the help of standard materials like methanol and ethyleneglycol+water (40:60). Microwave power measured by PM-437 (Attest) power meter, Chennai, India using source of Reflex klystron 2 K 25 (USSR). The densities and viscosities of the pure components and their binary mixtures are measured by using DMA 35 portable vibrating density meter. Anton paar Autria (Europe) having accuracy of density 0.001 gm/cm^3, repeatability 0.0005 gm/cm^3 and resolution 0.0001 gm/cm^3 [13] and viscosity by LVDL, V-pro II Brook field viscometer with an accuracy of $\pm$1% (USA) [14]. Rectangular wave guide working TE_{10} mode, 10 dB, Vidyut Yantra Udyog, India. To hold the liquid sample in the liquid cell, thin mica window whose VSWR and attenuation are neglected is introduced between the cell and rest of microwave bench. The X-band microwave bench is used to measure wavelengths in the dielectric and the voltage standing wave ratio (VSWR).

We determined the dielectric constant (ε'), dielectric loss (ε''), relaxation time (τ), dipole moment (μ) and excess inverse relaxation time $(1/\tau)^e$ of dilute solutions of binary mixture of 2,3-DCA +2-EE in 1,4-dioxane solution. All the measurements are carried out at temperatures 20^0C, 30^0C, 40^0C and 50^0C and the temperature is thermostatically controlled within $\pm 0.5^0$C at 20^0C, 30^0C, 40^0C and 50^0C for different mole fraction containing 0,0.2147,0.4507,0.7110,1 of 2,3-DCA+2-EE in 1, 4-dioxane solution. The microwave techniques have been used method suggested by Heston *et.al* [12].

To calculate dielectric constant (ε'), dielectric loss (ε'') by the using equations 1 and 2

$$\varepsilon' = \left(\frac{\lambda_0}{\lambda_c}\right)^2 + \left(\frac{\lambda_0}{\lambda_d}\right)^2 \qquad (1)$$

$$\varepsilon'' = \frac{2}{\pi}\left(\frac{\lambda_0}{\lambda_d}\right)^2 \cdot \frac{\lambda_g}{\lambda_d}\left(\frac{d\rho}{dn}\right) \qquad (2)$$

Where $\lambda_0, \lambda_c, \lambda_g$ and λ_d are the free space wavelengths, the cut-off wavelength, the waveguide wavelength and the wavelength in the waveguide filled with solution in centimeter respectively. ρ is the inverse of voltage standing wave ratio (VSWR) and $d\rho/dn$ is the slope of ρ versus n, where n=1,2,3….. Such that $(n\lambda_d/2)$ represents the length of the dielectric filled waveguide. The ε' and ε'' values are estimated to be reproducible within $\pm 0.5\%$ and $\pm 1\%$ respectively. The relaxation times (τ) and the dipole moment (μ) have been calculated by using equation 5 and 6 by the single frequency concentration variational method of Gopala Krishna [15].

$$X = \frac{\varepsilon'^2 + \varepsilon''^2 - 2}{(\varepsilon'+2)^2 + \varepsilon''^2} \qquad (3)$$

$$Y = \frac{3\varepsilon''}{(\varepsilon'+2)^2 + \varepsilon''^2} \qquad (4)$$

$$\tau = \frac{\lambda_0}{2\pi c}\left(\frac{dY}{dX}\right) \qquad (5)$$

$$\mu^2 = \frac{9kTM}{4\pi N d_0}\left[1 + \left(\frac{dY}{dX}\right)^2\right]\frac{dX}{dW} \qquad (6)$$

Where c is the velocity of electromagnetic waves in vacuum, K is the Boltzmann's constant, N is the Avogadro number, M is the molecular weight of polar substance in gm/mol, W is the weight fraction, T is absolute temperature and d_0 is the density of solution in gm/cm³. The slope of the line drawn between x and y used for determine the value of relaxation time (τ) and the slope of line x and w used for calculating the dipole moment (μ).

The excess inverse relaxation time $(1/\tau)^e$ may be evaluated for binary system in non-polar solvent using the relation [16-17].

$$(1/\tau)^E = (1/\tau)_m - \left[X_1(1/\tau_1) + X_2(1/\tau_2)\right] \qquad (7)$$

The symbol m, 1 and 2 are related to the mixture, liquid 1 and liquid 2 respectively. Information regarding the dynamics of a binary system interaction from this excess property is as follows.

Where $(1/\tau)^e$ 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 which is the inverse of the relaxation time from resonant spectroscopy [18]. The information regarding the dynamics of solute-solvent interaction from this excess property is as follows:

(i) $(1/\tau)^e = 0$: There is no change in the dynamics of solute-solvent interaction.
(ii) $(1/\tau)^e < 0$: The solute-solvent interaction produces a field such that the effective dipoles rotate slowly.
(iii) $(1/\tau)^e > 0$: The solute-solvent interaction produces a field such that the effective dipoles rotate quickly, i.e. the filed co-operate in the ratio of the dipoles.

The experimental techniques for the measurements of dielectric constant (ε'), dielectric loss (ε''), relaxation time (τ),dipole moment (μ) and excess inverse relaxation time $(1/\tau)^e$. All measurements are carried out at temperatures 20^0C, 30^0C, 40^0C and 50^0C in a dielectric cell by circulating thermo stated ethyleneglycol + water. The dielectric constant (ε'), dielectric loss (ε'') for 2,3-DCA+2-EE and their binary mixture containing mole fraction (X) 0,0.1818,0.4,0.6667,1 in 1,4-dioxane solution. The values of wavelength in dielectrics (λ_d) and ($d\rho/dn$) along with weight fraction of solute in 1,4-dioxane at different temperatures are listed in Tables I-IV.

RESULTS AND DISCUSSION

The values of ε' and ε'' for the binary mixture 2,3-DCA+2-EE in the 1,4-dioxane solution have been calculated using the short-circuited waveguide method of Heston [12]. This method is highly accurate for the measurement of ε' and ε'' of polar mixtures in dilute solutions of non-polar solvent at very low concentrations. The accuracy in measurements dielectric permittivity (ε') and dielectric loss (ε'') values is ±1% and ±2% respectively. From observations Table I-IV the variation of ε' and ε'' with weight fraction of solute in 1,4-dioxane for all binary mixtures is found to be linear. This shows that there is no change in the nature of the rotating molecular entities in the 1, 4-dioxane solution. This ensures the applicability of the Debye theory [19] and that of Gopala Krishna's method in the studied concentration range of the binary mixtures in the 1,4-dioxane solutions. The relaxation time depends upon the size and shape of the rotating molecular entities in the solution. This method determines the average value of the relaxation time for the participating molecular entities in the solution. The linear variation of the relaxation time from its value corresponding to one constituent to the value corresponding to the other constituent with the mole-fraction variation in the whole concentration range may be taken as the absence of any solute-solute association in the mixtures. On the other hand, non-linear variation of the relaxation time with the mole-fraction is interpreted as the possible solute-solute molecular association in the binary mixtures. So if the relaxation time of 2,3-DCA+2-EE binary mixture increase linearly with the mole fraction of 2,3-DCA ($X_{2,3\text{-DCA}}$), no molecular association can be inferred. However, if the relaxation time 2, 3-DCA+2-EE binary mixture increase non-linearly, a solute-solute type of molecular association between 2,3-DCA and 2-EE may be inferred. The variation of relaxation time (τ) versus mole fraction $X_{2,3\text{-DCA}}$ at different temperatures is shown in Fig. 1.

The variation of relaxation time (τ) versus mole fraction $X_{2,3\text{-DCA}}$ at different temperatures is shown in Fig. 1.
From Fig. 1 relaxation time (τ) first decreases with increase in mole fraction of 2,3-DCA in 2,3-DCA+2-EE binary mixture and attain a minimum value at $X_{2,3\text{-DCA}}$=0.21 of 2,3-DCA+2-EE binary mixture. Then with further increase in the mole fraction of 2,3-DCA in binary mixture, there is gradual increase in relaxation time and reaches maximum value at $X_{2,3\text{-DCA}}$=0.71 towards the value of pure 2,3-DCA in 1,4-dioxane solution. This behavior indicates solute-solute type of molecular association between 2,3-DCA and 2-EE.In its whole concentration range, the relaxation time of 2,3-DCA+2-EE binary mixture remains longer that of pure 2,3-DCA. Therefore, the solute-solute type molecular association between 2,3-DCA and 2-EE indicates in its entire concentration range. In view of above results, it is proposed that in the binary mixtures of 2,3-DCA and 2-EE, 2,3-DCA exists in the dimer structure resulting because of H-bonding and dimer structure of 2,3-DCA interacts with the 2-EE molecules so as to give the maximum values of relaxation time at 71 mol% 2,3-DCA binary mixture. This type of molecular associations have been proposed in Fig 2. The value of dipole moment 2,3-DCA binary mixture with 100% mole fraction of 2,3-DCA in the binary mixture depends on the temperature. This indicates the presences of solute-solvent molecular association of pure 2,3-DCA in 1, 4-dioxane solution. The value of dipole moment of 2-EE binary mixture with 0.00 mole fraction of 2,3-DCA in binary mixture Table I-IV is found to be slightly change with temperature. This could be explained on the basis of the solvent effects [20]. The change in dipole moment with temperature may be due to the stretching of the bond moment and due to the change in the bond angle. The dipole moment value of 2,3-DCA slightly non-linearly increases with the rise in temperature in 1, 4-dioxane solution [21-22].

This predicts the solute-solvent type of molecular association for 2,3-DCA in the 1,4-dioxane solution. Solute-solute association can be interpreted because of the molecule association arising due to hydrogen bonding between 2-ethoxyethanol and 2,3-dichloroaniline. The hydrogen bonding δ^+ on hydrogen of hydroxyl group of 2-ethoxyethanol that form hydrogen bonding with nitrogen of 2-chloroaniline as shown in Fig. 2. It may be explained on the basis that the dielectric relaxation process involves the rotation of molecular entities.

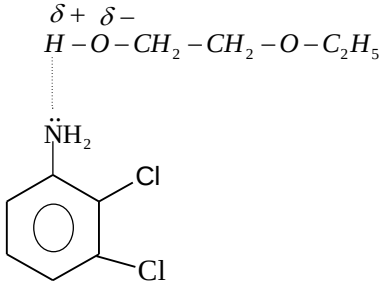

Fig.2. Solute-solute molecular association between 2,3-DCA and 2-EE

Solute- solvent association can be interpreted because of the molecule association arising due fractional positive charge on hydrogen of 2-ethoxyethanol and lone pair electron present on oxygen of 1,4-dioxane is shown in Fig. 3.

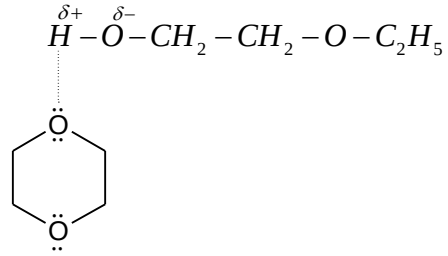

Fig.3. Solute-solvent molecular association of 2-EE in 1,4-dioxane solution

The excess inverse relaxation time $(1/\tau)^{e}$ for the binary mixture of system 2, 3-DCA+2-EE for different mole fraction of 2, 3-DCA and different temperatures are calculated using equation 7 are listed in Table I-IV.From Fig.4 the excess inverse relaxation time $(1/\tau)^{e}$ is found to be positive for all temperature through the entire range of concentration of mole fraction of 2, 3-DCA. The positive peak is observed at around X=0.2147 mole fraction of 2, 3-DCA concentration in 2, 3-DCA+2-EE binary liquid mixture This suggests that the solute-solute interaction provides a field such that effective dipoles rotates faster i.e. the field facilitates rotation of dipoles. Similar results have been already predicated by Krishna *et. al.* [23].

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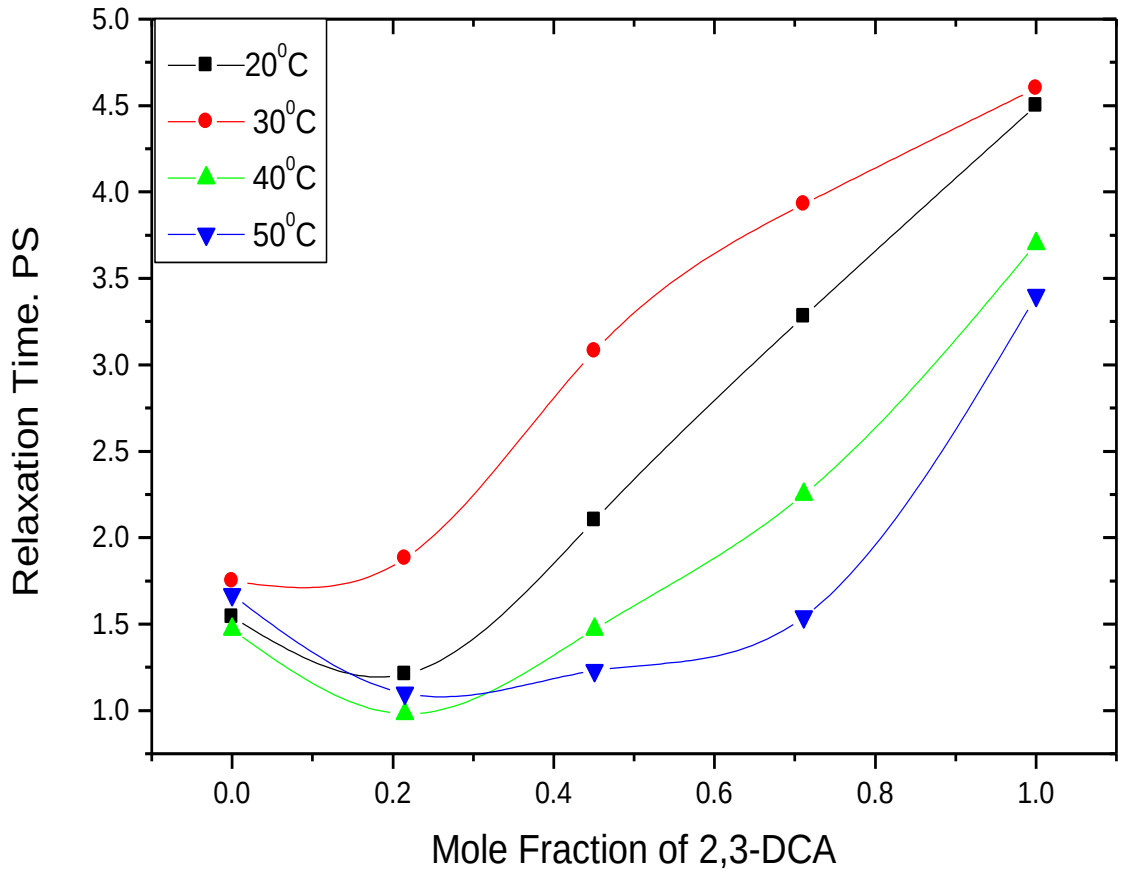

Figure 1. Relaxation time (τ) versus mole fraction of 2,3-DCA in 2,3-DCA+2-EE binary mixture in 1,4-dioxane solution at different temperatures.

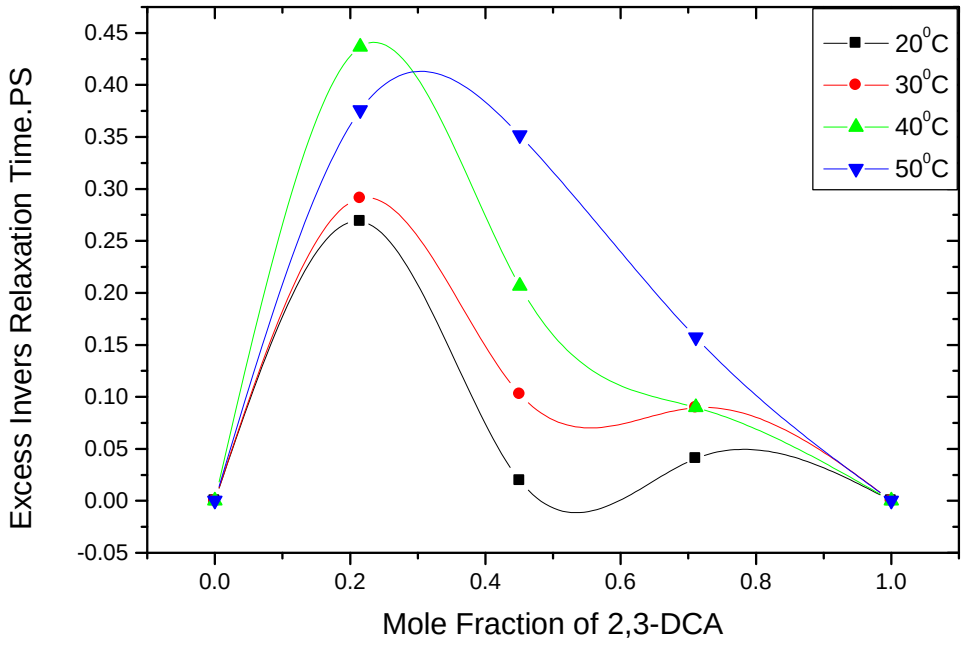

Figure 2. Excess inverse relaxation time $(1/\tau)^e$ versus mole fraction(X) of 2,3-DCA in 2,3-DCA+2-EE binary mixture in 1,4-dioxane solution at different temperatures.

Table 1. Relaxation properties and dipole moment of 2,3-DCA in 2,3-DCA+2-EE binary mixture in 1,4-dioxane at 20^0C.

MF[a]	WF[b] (W)	ε′ [c] (±0.5%)	ε″ [d] (± 1%)	τ [e] (P.Sec)	μ (D) [f] (Debye)	g $(1/\tau)^e$
	0.0528	2.145	0.098			
0	0.1004	2.375	0.128	1.54	1.45	0
	0.1434	2.504	0.150			
	0.1825	2.505	0.158			
	0.0590	2.064	0.100			
	0.1115	2.382	0.129	1.21	1.57	0.2689
0.2147	0.1584	2.422	0.153			
	0.2006	2.498	0.163			
	0.0841	2.114	0.112			
	0.1553	2.298	0.151	2.10	1.35	0.0194
0.4507	0.2161	2.444	0.181			
	0.2688	2.512	0.199			
	0.0707	2.173	0.097			
	0.1322	2.347	0.128			
0.7110	0.1860	2.420	0.152	3.28	1.27	0.0407
	0.2335	2.455	0.180			
	0.0769	2.245	0.124			
	0.1428	2.380	0.188	4.50	1.02	0
1	0.1999	2.420	0.193			
	0.2499	2.422	0.195			

a Mole Fraction.; b Weight Fraction (W).; c Dielectric Permittivity (ε'); d Dielectric Loss (ε''); e Relaxation Time (τ).
f Dipole Moment μ (D); g Excess inverse relaxation time $(1/\tau)^e$

Table 2. Relaxation properties and dipole moment of 2,3-DCA in 2,3-DCA+2-EE binary mixture in 1,4-dioxane at 30^0C.

MF[a]	WF[b] (W)	ε′ [c] (±0.5%)	ε″ [d] (± 1%)	τ [e] (P.Sec)	μ (D) [f] (Debye)	g $(1/\tau)^e$
	0.0528	2.097	0.105			
	0.1004	2.335	0.134	1.75	1.57	0
0	0.1434	2.470	0.155			
	0.1825	2.480	0.161			
	0.0590	2.131	0.116			
	0.1115	2.237	0.141	1.88	1.54	0.291
0.2147	0.1584	2.478	0.170			
	0.2006	2.516	0.172			
	0.0841	2.192	0.098			
	0.1553	2.368	0.148	3.08	1.34	0.1027
0.4507	0.2161	2.500	0.158			
	0.2688	2.564	0.159			
	0.0707	2.048	0.071			
	0.1322	2.318	0.134			
0.7110	0.1860	2.444	0.170	3.93	1.78	0.0897
	0.2335	2.559	0.190			
	0.0769	2.148	0.106			
	0.1428	2.298	0.164	3.97	1.49	0
1	0.1999	2.480	0.185			
	0.2499	2.512	0.194			

a Mole Fraction.; b Weight Fraction (W).; c Dielectric Permittivity (ε'); d Dielectric Loss (ε''); e Relaxation Time (τ).
f Dipole Moment μ (D); g Excess inverse relaxation time $(1/\tau)^e$

Table. 3 Relaxation properties and dipole moment of 2,3-DCA in 2,3-DCA+2-EE binary mixture in 1,4-dioxane at 40^0C.

MF[a]	WF[b] (W)	ε′ [c] (±0.5%)	ε″ [d] (± 1%)	τ [e] (P.Sec)	μ (D) [f] (Debye)	g $(1/\tau)^e$
	0.0528	2.210	0.094			
	0.1004	2.466	0.126			
0	0.1434	2.524	0.141	1.47	1.56	0
	0.1825	2.604	0.157			
	0.0590	2.075	0.100			
	0.1115	2.318	0.149	0.982	1.83	0.4365
0.2147	0.1584	2.422	0.156			
	0.2006	2.633	0.173			
	0.0841	2.210	0.094			
	0.1553	2.466	0.126	1.47	1.37	0.2065
0.4507	0.2161	2.524	0.141			
	0.2688	2.604	0.157			
	0.0707	2.166	0.116			
	0.1322	2.359	0.164			
0.7110	0.1860	2.512	0.192	2.25	1.46	0.0899
	0.2335	2.521	0.197			
	0.0769	2.166	0.129			
	0.1428	2.278	0.178	3.70	1.15	0
1	0.1999	2.369	0.194			
	0.2499	2.422	0.197			

a Mole Fraction.; b Weight Fraction (W).; c Dielectric Permittivity (ε'); d Dielectric Loss (ε''); e Relaxation Time (τ).
f Dipole Moment μ (D); g Excess inverse relaxation time $(1/\tau)^e$

Table. 4 Relaxation properties and dipole moment of 2,3-DCA in 2,3-DCA+2-EE binary mixture in 1,4-dioxane at 50^0C.

MF[a]	WF[b] (W)	ε′ [c] (±0.5%)	ε′′ [d] (± 1%)	τ [e] (P.Sec)	μ (D) [f] (Debye)	g $(1/\tau)^e$
	0.0528	2.069	0.095			
	0.1004	2.380	0.140	1.67	1.81	0
0	0.1434	2.359	0.154			
	0.1825	2.559	0.184			
	0.0590	2.064	0.096			
	0.1115	2.380	0.147	1.10	1.83	0.3758
0.2147	0.1584	2.514	0.156			
	0.2006	2.516	0.169			
	0.0841	2.104	0.123			
	0.1553	2.401	0.165	1.23	1.43	0.3516
0.4507	0.2161	2.498	0.185			
	0.2688	2.536	0.189			
	0.0707	2.221	0.140			
	0.1322	2.359	0.176			
0.7110	0.1860	2.536	0.195	1.54	1.37	0.1572
	0.2335	2.540	0.199			
	0.0769	2.131	0.117			
	0.1428	2.318	0.164	3.40	1.18	0
1	0.1999	2.338	0.176			
	0.2499	2.870	0.181			

a Mole Fraction.; b Weight Fraction (W).; c Dielectric Permittivity (ε'); d Dielectric Loss (ε''); e Relaxation Time (τ).
f Dipole Moment μ (D); g Excess inverse relaxation time $(1/\tau)^e$

CONCLUSION

The molecular association between 2,3-DCA+2-EE is maximum nearly at a 29:71 mol% ratio respectively and then increases at higher mol% of 2,3-DCA in binary mixtures of 2,3-DCA+2-EE exists in the dimer structure resulting because of H-bonding and dimer structure of 2,3-DCA interact with the 2-EE molecules so as to give the maximum values of relaxation time at 71 mol%, of 2,3-DCA binary mixture respectively. Relaxation time increases due to addition of -CH_2 group in the binary mixtures. This confirms that the relaxation time (τ) increases due to increase in carbon atoms in binary mixtures. The change in dipole moment with temperature may be due to the stretching of bond moment and due to the change in bond angle. The excess inverse relaxation time $(1/\tau)^e$ are found to positive.

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