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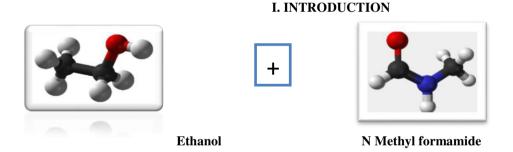
The Study of Inter Molecular Interaction between Ethanol- N Methyl Formamide in Microwave Region at 288, 298, 308 and 318 K

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ABSTRACT: Dielectric relaxation measurement for Ethanol (C₂H₅OH) N Methyl formamide (HCONHCH₃) binary mixture have been carried out for 11 different concentrations at 288,298,308 and 318 K in the frequency range of 10MHz to 10 GHz. The mixture exhibitsprincipal dispersion of Davison-Cole Model at microwave frequencies. Bilinear calibration method is used to obtain complex permittivity $\epsilon^*(\omega)$ from reflection coefficient $\rho^*(\omega)$. From complex permittivity static permittivity is obtain by fitting it to HaverliacNigami equation. The values of static permittivity are increases with increase in the concentration of NMF in ethanol, Relaxation Time, excess parameters, Brugman factors, Kirkwood correlation factor and thermodynamic parameters shows systematic interaction between solute solvent different temperature and concentrations.

KEYWORDS: Static permittivity are increases with increase in the concentration of NMF in ethanol, Relaxation Time, excess parameters, Brugman factors, Kirkwood correlation factor and thermodynamic parameters



Mainly alcohol play an important role in many chemical reactions on account of their ability to undergo selfassociation with manifold internal structure and widely used in industries, alternate fuel for petrol, or mixture in petrol, beverages, Medicines reagents, solvent, and attract great attraction as useful solvent in the green technology. Ethanol is obtained from food extract particularly from molasses of sugar in large quantity in sugar industry at the time of processing of sugar from sugar cane juice, molasses is the byproduct and which is west material so ethanol made from molasses is chipper.

The aim of present investigation is to report a systematic variation in dielectric parameter of binary mixture at 11 different concentrations at 288, 298, 308, and 318k.temperture by using time domain reflectometry in the frequency range 10MHzto 20 Ghz. The experimental data is fitted to different relaxation model [34,35,36.] by the nonlinear least square fit method. It is observed that the Debye model is adequate to describe major dispersion for present system. Static dielectric constant and dielectric relaxation time could be obtained by fitting the permittivity spectra with Debye equation [34]

The importance of measuring the dielectric constant of a liquid lies in the fact that it provides valuable information about intermolecular interaction and dynamics of molecules at molecular levels. In NMFand ETH molecules charge distribution is exposed and formation of hydrogen bond. When there is an exposed distribution of charges, there may be very strong intermolecular interaction, however, in shielded distribution relative contribution to neighbor molecules is absent and hence there may be weak intermolecular interaction between the molecules of the liquids. Because of the industrial, medical, research and biological importance we have chosen these liquids for present study. Here the picoseconds Time domain reflectometry[1-4] in reflection mode has been used to obtain dielectric parameters at temperatures of 288, 298, 308 and 318 K. From the dielectric parameters, excess

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permittivity, excess inverse relaxation time, Bj coefficient, Kirkwood correlation factor, and thermodynamic parameters are obtained. Based on above parameters, intermolecular interaction, and dynamics of molecules at molecular level are discussed.

- 1. ε ' and ε '' have been widely used in deriving information about intermolecular and intramolecular interaction in liquids.
- 2. Relaxation time (τ) gives the idea about molecular properties such as size shape, rigidity etc.
- 3. Excess Permittivity (ε^{E}) and Excess Inverse Relaxation time $(1/\tau)^{E}$ gives significant information about the formation of structures like monomers and multimers.
- 4. Kirkwood Correlation factor (g^{eff}) provides information about parallel or antiparallel alignment of dipoles.
- 5. Thermodynamic properties are helpful in accessing the states of dipoles under the influence of applied field.

The permittivity of a material (ε_s) is a property which describes the charge storing ability of that substance. The application of a static electric field to a solution containing polar molecules will have the effect of aligning the molecules in the direction of the external field. If the field is switched off the molecules do not immediately adopt random orientations. The relaxation time (τ) is a measure of the time taken to reorient its randomizing state. The relaxation time (τ) depends on the temperature and decreases as the temperature is increased, i.e. the larger translation motions of the molecules enable them to randomize their position more quickly when the electric field is switched off [5]. Alcohols (ethanol) have one -OH group attached to the carbon chain. These compounds are able to form hydrogen bond very extensively, which in turn correlate with a long relaxation time.

II. EXPERIMENTAL

The chemicals used in the present work are Ethanol and N methyl formamide are of AR grade and are used without further purification. The solutions are prepared at eleven different volume fractions of ETH from 0 to 1 in step of 0.1. These volume fractions are converted to mole fractions for further calculations. The time domain spectroscopy in reflection mode has been used to obtain dielectric parameters

N methyl Formamide is studied due to its industrial importance as a solvent for some organic liquids and similar physical properties to the universal solvent water. In particular it has a high static permittivity in order of 170, at room temperature which falls with high temperature and frequency. Its freezing point is near to water and its density is $1.011 \times 103 \text{ Kg/m}^3$. However, its boiling point and viscosity are higher than pure water. Its dielectric relaxation time is ten times greater than water. [38,39]

III. RESULT AND DISCUSSION

Material	ρ	M P	ВР	n _D (RI)	μ_{D}	UV λ_{max}
Ethanol	0.789	$-114.14^{\circ}c$	78.23 [°] c	1.3611	1.69D	280nm
NMF	1.011	$-4^{0}c$	$182.6^{\circ}c$	1.432	3.83D	357nm

Permittivity and relaxation time

The frequency dependent complex permittivity $\varepsilon^* = \varepsilon' - j \varepsilon''$ data was obtained by using calibration process. The static dielectric constant (ε_s) and relaxation time (τ) for the mixture obtained by fitting experimental data with the Debye equation at four different temperatures have been given in table 1 and are shown in figures 1 and 2 respectively [22,38,39,40,41&42].

$$\varepsilon^{*}(\omega) = \varepsilon_{\infty} + \frac{\varepsilon_{s} - \varepsilon_{\infty}}{1 + j\omega\tau}$$

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Table: 1.: Temperature dependent dielectric parameters for binary mixture of Ethanol + N methyl
Formamide

	ε ₀			τ				
Conc of NMF	288	298	308	318	288	298	308	318
0	26.5	24.4	23.66	20.52	146.12	126.25	115.65	105.11
0.0993	32.7	31.89	30.55	29.12	136.41	114.54	96.54	78.54
0.1988	41.96	41.9	38.58	36.76	125.28	102.12	85.55	72.18
0.2984	49.82	49.03	47.35	45.19	114.15	97.41	82.32	70.83
0.3982	62.02	59.4	56.59	55.11	111.65	95.12	81.21	69.9
0.4981	77.28	73.54	71.4	67.43	116.55	98.41	83.56	72.35
0.5982	92.46	88.07	84.51	79.44	121.83	104	88.28	75.65
0.6984	111.99	105	100.64	96.26	126.33	109.8	93.56	81.24
0.7988	134.64	125.65	119.25	112.21	135.55	117.45	99.45	87.17
0.8993	157.98	149.79	142.15	133.64	140.25	124.32	106.15	93.45
1	181	170.41	161.9	150.95	155.61	132.78	118.55	102.25

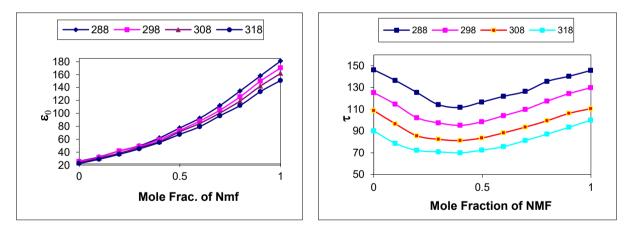


Figure 1: Variation of static dielectric constant (ϵ_s) and relaxation time (τ) as a function of mole fraction of N methyl formamide at temperatures 288, 298, 308 and 318K.

Table1 reports the values of dielectric parameters obtained by fitting experimental data to Debye equation. It can be seen that by increasing the concentration of NMF in ETH the static permittivity of values increases towards NMF rich region. Increase of dielectric constant is due to the transition of spherical molecular aggregate in to elongated aggregate giving rise to parallel orientation of the dipoles. Similar conclusion was drawn by shirke et al. [37] for alkyl acetate alcohol system. It is found that in the NMF rich region the dielectric constant increases whereas, when the concentration of ETH dominates in the mixture, the static permittivity decreases. It is also found that the ε_0 and τ values of mixture lie between the individual component values which indicates the solute solvent interaction between the –OH group of ETH and (-C=O) –NH group of amide.

The relaxation time shows continuous decrease with increase in concentration of NMF in ETH up to equal concentration region and increases in the NMF rich region dominating molecule offers hindrance to the rotation of the molecule. The increase in relaxation time with increase in concentration of NMF is to be expected in view of the fact that hydroxyl group reorientation depends to some extent on the no of molecules of NMF to be added in

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mixture, due to viscosity of mixture get increases. The relaxation time decreases with addition of NMF up to equal concentration region, which indicate that the degree of cooperativity for reorientation of the molecules decreases with increasing length and bulk of the cluster decreases. Whilein NMF rich region relaxation time increases, which indicate that the degree of cooperativity for reorientation of the molecules increases with increasing length and bulk of the cluster decreases. Whilein NMF rich region relaxation time increases, which indicate that the degree of cooperativity for reorientation of the molecules increases with increasing length and bulk of the cluster increases. It means that it shows mixed behavior. From this calculated data dielectric constant (static permittivity) & relaxation time we can obtain excess parameter & thermodynamic parameter which gives information about the interaction of solute solvent among the system.

Excess Permittivity and Excess Inverse Relaxation Time

The variation of Excess permittivity (ε^{E}) and Excess inverse relaxation time ($1/\tau$)^E with change in mole fraction of NMF at different temperatures is shown in fig 2. The experimental values of both the excess parameters were fitted to the Redlich - Kister equation [15] to obtain Bj coefficients at different temperatures. Using these B_j values excess parameters at various concentrations were calculated and used to draw the smooth curves. The estimated values of B_j are given in table 2

The excess parameters [20, 23.] related to $\varepsilon_0 \& \tau$ provides valuable information regarding solute solvent interaction these properties are also useful for the detection of cooperative domain in the mixture and may give evidence for the formation of multimers in the mixture due to intermolecular interaction.

The excess permittivity is defined as

$$\varepsilon^{E} = (\varepsilon_{0} - \varepsilon_{\infty})_{m} - [(\varepsilon_{0} - \varepsilon_{\infty})_{A} x_{A} + (\varepsilon_{0} - \varepsilon_{\infty})_{B} x_{B})]$$

Where X is the weight fraction and suffices M, A, B represent mixture, liquid A and liquid B respectively the excess permittivity provides qualitative information about structure formation in the mixture as follows $\varepsilon^{E} = 0$ indicates the solute and solvent do not interact at all

 $\varepsilon^{E} < 0$ indicates the solute and solvent do not interact in such a way that the total effective dipoles are reduced. The solute and solvent may be form multimers leading to the less effective dipoles

 $\varepsilon^{E} > 0$ indicates the solute and solvent interact in such a way that the total effective dipole moment increases. There is a formation of monomers and dimmers

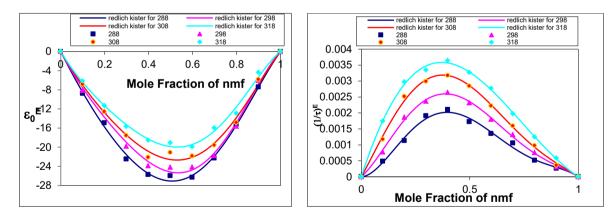


Fig.2 Variation of excess permittivity (ϵ^{E}) and Excess Relaxation Time(1/ τ)^E as a function of mole fraction (x2) of N Methyl Formamide at 288, 298, 308 and 318K.

Fig shows the plot of excess permittivity against mole fraction of NMF for all four temperatures. In the present study excess permittivity values are found to be negative for all temperatures and concentrations, which indicate that the total effective dipoles are decreases in the mixture. This is due to antiparallel or opposite alignment of the dipoles of the two interacting solvent molecules. The curves are more deviated from zero at equal concentration region indicates strong intermolecular interaction in this region.

The excess inverse relaxation time is defined as $(1/\tau)^E = (1/\tau)_m - [(1/\tau)_A X_A + (1/\tau)_B X_B]$

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Where $(1/\tau)^E$ is the excess inverse relaxation time, which represent the average broadening of dielectric spectra. The inverse relaxation time analogy is taken from spectral line broadening (which is the inverse of relaxation time) from resonant spectroscopy (Mehrotra and Boggs, 1977)

The information regarding the dynamics of solute solvent interaction gives from this excess property is as follows $(1/\tau)^{E} = 0$ there is no change in the dynamics of solute solvent interaction

 $(1/\tau)^{E} < 0$ the solute solvent interaction produces a field such that the effective dipoles rotate slowly.

 $(1/\tau)^{E}$ 0 the solute solvent interaction produces a field such that the effective dipoles rotate faster. ie the field facilitates rotation of dipoles.

The variation of $(1/\tau)^{\tilde{E}}$ with mole fraction of NMF at 15, 25, 35 and $45^{\circ}c$ and at 11 concentrations as shown in above fig. From fig it can be seen that, for all the four-temperature excess inverse relaxation values are positive for all temperatures which indicates solute solvent interaction produces field such that the effective dipoles rotate faster.ie. field facilitate rotation of dipoles or cooperate to the dipole.

Table .2: The estimated values of Bj coefficients in Redlich-Kister equation for ethylene glycol N Methyl Formamide mixture at different temperatures.

Donomotor	D apofficient	Temperature					
Parameter	B _j coefficient	288 K	298 K	308 K	318 K		
	B ₀	-108.25	-100.75	-90.333	-79.523		
$(\boldsymbol{\epsilon}_{s})^{E}$	B ₁	-3.9073	-18.616	-13.099	-12.419		
(ε _s)	B ₂	30.446	31.693	20.172	22.254		
	B ₃	18.264	37.975	22.689	32.124		
	B ₀	0.0073	0.0094	0.0114	0.0131		
$(1/\tau)^{\rm E}$	B ₁	-0.0069	-0.0082	-0.0099	-0.0089		
$(1/\tau)^{-1}$	B ₂	-0.0049	-0.0042	-0.0029	-0.0003		
	B ₃	0.009	0.0074	0.0061	0.0007		

The Kirkwood correlation factor

The values of $g^{eff} g_{f}$ are given in table .no 3 and shown in fig. 3

The amount of solute-solvent interaction can be accessed using these parameters.

Vol. frac of	288	298	308	318
0	1.00	0.98	0.90	0.86
0.1	0.81	0.79	0.75	0.72
0.2	0.76	0.76	0.70	0.67
0.3	0.71	0.70	0.68	0.65
0.4	0.73	0.70	0.67	0.65
0.5	0.77	0.73	0.71	0.67
0.6	0.80	0.76	0.73	0.69
0.7	0.85	0.80	0.76	0.73
0.8	0.91	0.85	0.81	0.76
0.9	0.96	0.91	0.87	0.81
1	1.00	0.94	0.89	0.83

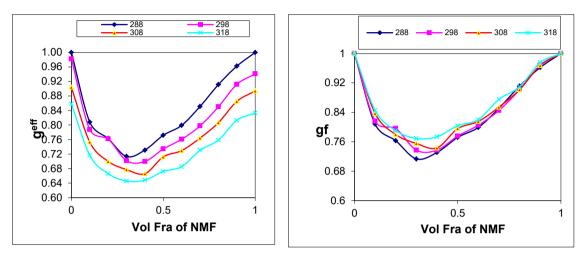
Vol. frac of NMF	288	298	308	318
0	1	1	1	1
0.1	0.81	0.82	0.84	0.85
0.2	0.76	0.80	0.78	0.79
0.3	0.71	0.74	0.76	0.77
0.4	0.73	0.74	0.74	0.77
0.5	0.77	0.78	0.80	0.80
0.6	0.80	0.80	0.82	0.82
0.7	0.85	0.85	0.85	0.88
0.8	0.91	0.90	0.90	0.91
0.9	0.96	0.97	0.97	0.98
1	1	1	1	1

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Fig No.3 Variation of Kirkwood correlation factor g^{eff} and g_f with variation of volume fraction of NMF at various temperatures.



Thermodynamic parameters

r

The values of molar enthalpy of activation (Δ H) and molar entropy of activation (Δ S) at different concentrations determined using Eyring rate equation [18] are listed in table 4. The variation of molar enthalpy of activation and molar entropy of activation with increase in volume fraction of NMF in the mixture are shown in fig 4. The Arrhenius plot for ETH+NMF system is shown in fig 5.

Table No 5. The activation enthalpy (Δ H) entropy (Δ S) of ETH + NMF binary mixture for various	
concentrations.	

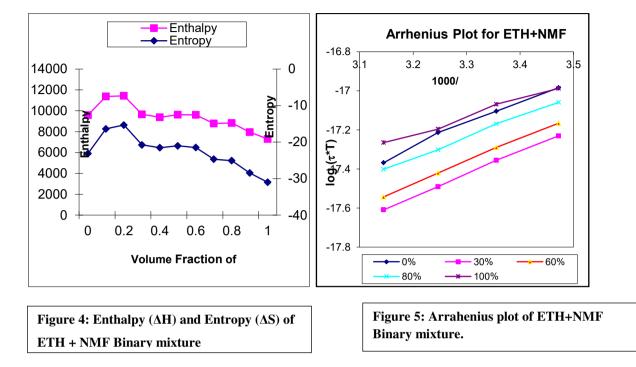
Vol Fr. NMF	Enthalpy	Entropy
0	9586.3	-23.13
0.1	11373	-16.37
0.2	11437	-15.31
0.3	9664.7	-20.76
0.4	9384.3	-21.54
0.5	9627.5	-21.02
0.6	9612.3	-21.49
0.7	8781.3	-24.69
0.8	8835.2	-25.07
0.9	7952.4	-28.46
1	7302	-31

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The arrhenius plot of $\ln(1/t)$ vs (1000/T) are plotted in fig (5) for various concentrations. The ln (1/T) values increase with increase in concentration of NMF all plots having similar slope, which indicates that at all concentrations the activation energy remain almost same.

IV. DISCUSSION

Static Permittivity and Relaxation Time

From fig 1 It can be seen that there is an linear relationship between the values of static dielectric constant (ε s) and concentration of NMF in the mixtures static dielectric constant increases with increase in concentration of NMF. It can also be seen that relationship for the relaxation time is nonlinear. It means that near to equal concentration molecule get lighter so reorient easily so at this region relaxation time is smaller than pure liquids. This suggests weak intermolecular interaction due to exposed charge distribution in ETH and NMF molecules.

Excess Parameters

From figure 2, it can be seen that $(\varepsilon_s)^{E}$ is negative for all concentration of NMF in the mixture for all temperature. This indicates that the molecules of mixture may form multimers structures in such a way that the effective dipoles get reduced.

The values of $(1/\tau)^{E}$ positive for all concentration and temperature this suggest that the molecular interaction produces a cooperative field and the effective dipoles have more freedom of rotation and total effective dipoles are rotate faster. from fig 2, it is also seen that at equal concentration field is highly cooperative so curve is more positive.

Kirkwood correlation factor

The value of g_{eff} is 1 for ETH at 288 K which decreases to 0.86 at 318 K. The values confirm the formation of hydrogen bonding in pure ETH system. These values are near to unity or less than unity at all temperatures suggesting anti parallel orientation of electric dipoles. The corresponding values for NMF are 1.00 and 0.83 respectively indicating weak dipole-dipole interaction resulting formation of antiparallel dipoles in the pure system of NMF. From table 4 the value of g_f is unity for an ideal mixture and deviation from unity may indicate interaction between two components of the mixture. The g_f value less than one indicates that the dipoles of mixture will be oriented in such a way that the effective dipole will be less than the corresponding values of pure liquid.

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Thermodynamic Parameters

From table5 it observe that the molar enthalpy of activation (Δ H) having nonlinear nature on addition of NMF in ethanol first it increases and then decreases with increase in concentration of NMF in ETH, it shows mixed nature. This means that the required for group dipole reorientation is more or less with increase in volume fraction of NMF in the mixture. All negative value of molar entropy of activation (Δ S) with volume fraction of NMF indicates relatively high ordered arrangement of molecules in the activated state at equal concentration.

Arrhenius plot for the system is shown in fig 5 The Arrhenius plot is almost linear for this system. The linear nature of Arrhenius plot shows that equivalent incremental change in temperature causes equivalent changes in values of activation enthalpy (Δ H) in temperature range under consideration. The slope of Arrhenius plot changes with concentration, which shows the change in activation energy of the system. The temperature dependence of relaxation time follows Arrhenius behavior.

V. CONCLUSION

It can be observed from above discussion that static permittivity (ε_s) of pure Ethanol increases with addition of NMF. This increase in permittivity is due to increase in effective dipole moment per unit volume in mixture. Relaxation time (τ) is very sensitive parameter related to molecular size as well as mobility of molecules in liquid. Excess permittivity (ε^E) negative for all concentrations of NMF in Ethanol indicates the solute and solvent interact in such a way that the total effective dipoles are reduced. The solute and solvent may be form multimers leading to the less effective dipoles. ($1/\tau$)^E is positive for all concentrations Which indicates the formation of spherical structures, solute solvent interaction produces field such that the total effective dipoles rotate faster. intermolecular interaction is confirmed by Kirkwood correlation factor and Bj coefficient.

The value of activation enthalpy (ΔH) gives an idea about nature of compactness in molecules of liquid. The variations in (ΔH) with change in solute concentration provide us information about relative change in amount of hydrogen bonding in mixture. The peak value of (ΔH) for systems under study show very strong hydrogen bonding in molecules of mixture.

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