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Study of Intermolecular Interaction of Ethanol -N Methyl Acetamide Binary Mixture at High Frequency

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Abstract - Dielectric Behavior of N-methyl Acetamide [C_3H_7NO] – Ethanol [C_2H_5OH] binary mixture at different temperatures 15, 25, 35 and 45^{0}c have been determined over frequency range of 10 MHz to 20 GHz. Using the Time Domain Reflectometry Technique in reflection mode for 11 different concentrations of given binary system has been used to measure reflection coefficient. Further Fourier transform and least square fit method have been used to obtain dielectric parameters such as dielectric constant (ε_0) and relaxation time (τ). The dielectric constant decreased with an increase in temperature where as it increased with increase in concentration of NMA in Ethanol. Relaxation time decreased with an increase in temperature and increases with the concentration of NMA in Ethanol. By using dielectric parameters excess permittivity (ε^E) has negative values for all concentrations and temperature indicates that total effective dipoles are decreases in the mixture. excess inverse relaxation time $(1/\tau)^E$ has positive values in ethanol reach region which indicates that formation of cooperative field to rotate dipole faster, while negative in NMA reach region which indicates the formation of linear structure (non cooperative field) results the slower rotation of dipoles. Kirkwood factor (g^{eff}) g_r, Bj coefficient and thermodynamic parameters give systematic behavior of solute solvent for different concentrations and temperature at high frequency.

Key words: Time domain reflectometry, Dielectric parameters, Excess parameters, Kirkwood correlation factor, Brugman factor and Thermodynamic parameter.

INTRODUCTION:

Dielectric relaxation studies on binary mixtures are important for understanding the hydrogen bonding and inter molecular interaction in the mixture. Dielectric study yields the Kirkwood correlation factor, which is strongly correlated with the solute solvent interaction these parameters give systematic change in structure of solute and solvent at different temperature for different concentrations.

Dielectric parameters for Ethanol-tetrahydrofuran, ethanol-dimethyl formamide, ethanol-dimethyl sulfoxide, ethanol-2ethoxy ethanol, ethanol-aniline, ethanol-nitrobenzene, ethanol-nitro toluene, were studied by our earlier group [21,24,33]

All the above systems showed systematic change in the dielectric parameters with Concentration and temperature

The aim of present work is to report a dielectric study on ethanol – N Methyl Acetamide mixtures conducted using Time Domain Reflectometry [TDR] at the frequency range 10MHz to 20GHz

The dielectric parameters viz. the static dielectric constant ε_0 relaxation time τ was determined. The Kirkwood correlation factor, excess dielectric parameters, B_j Coefficient and thermodynamic parameters obtained for 15^{0}c, 25^{0}c, 35^{0}c & 45^{0}c in order to Predict structural information about the binary mixtures

2. EXPERIMENTAL SETUP

Ethanol [AR grade] N Methyl acetamide [AR grade sd. Fine chem. Limited Mumbai] was used without further purification.

The solutions were prepared by mixing N Methyl Acetamide and ethanol with different concentrations at room temperature assuming ideal mixing behavior Composition in step of 10% by volume with in a 0.02% error limit.

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The complex permittivity spectra were studied using TDR [1]. A 200mv pulse from a tunnel diode pulse generator block with a rise time of 25ps to 45ps and repetitive frequency of 1kHz was transmitted along a 3.5mm transmission line with characteristic impedance of 50 Ohms. The sample cell was placed at the end of the transmission line, after the sample head.

The length of transmission line which connects pulse generator and Sampling head was about twice of the transmission line which connects sampling head and cell so that the incident pulse was well separated from the reflected pulse. The required cell was designed in such a way that its impedance matched with the transmission line

A Techtronic's 7854 sampling oscilloscope was used to sample the reflected pulse. The time window used for the experiment was kept at 500ps/division or 5ns. The reflected pulse was digitized with 1024 points in the selected time window. The oscilloscope is transfer to computer through 1.44MB floppy.

First, the reflected pulse R_1 (t) & R_x (t) without and with a sample in the cell respectively, were recorded. Time referencing was done by matching small reflection due to a particular discontinuity at the beginning of the cell in R_1 (t) & R_x (t) subtraction and addition of these pulses were done in the memory of the oscilloscope after proper time shifting. The subtracted pulse and added pulse were transferred to the computer for further analysis

The temperature controller system with a water bath and a thermostat was used to maintain the constant temperature with an accuracy limit of $\pm 1^0$c. The sample was surrounded by an insulating container, through which water obtained at a constant temperature using temperature controller system was circulated. The temperature at the cell was checked using an electronic thermometer

3 Data analysis:

The time dependent data were processed to obtain complex reflection coefficient spectra $\rho^*(\omega)$ over the frequency range from 10MHz to 20GHz using Fourier transformation [42,43]

$$\rho^*(\omega) = (c/j\omega d) [p(\omega)/q(\omega)] \quad \ldots(1)$$

Where ω is the angular frequency, p(w)and q(w) are the Fourier transform of $R_1(t)$-$R_x(t)$ and $R_1(t)$+$R_x(t)$, respectively, c is the velocity of light, d is the effective pin length and

$$j = -1^{1/2}$$

The complex permittivity spectra $\varepsilon^*(\omega)$ were obtained from reflection coefficient spectra $\rho^*(\omega)$ by applying bilinear calibration method [1] in the calibration process, pure liquids were used as a calibrating liquid

The experimental values of $\varepsilon^*(\omega)$ were fitted with the Debye equation [34,35,36]

$$\varepsilon^*(\omega) = \varepsilon_\infty + \varepsilon_0 - \varepsilon_\infty / 1 + (j\omega\tau) \quad \ldots.(2)$$

Where ε_0, ε_∞ and τ are fitting parameters in equation (2), ε_0 is the static dielectric constant, ε_∞ is the high frequency dielectric constant and τ is the relaxation time. A non linear least square fit method (44) was used to determine the values of the dielectric parameters.

ie $\varepsilon^*(\omega)$ were obtained from reflection spectra $\rho^*(\omega)$. The examples of $\rho^*(\omega)$ and $\varepsilon^*(\omega)$ spectra are shown in fig 1(a) & 1(b) respectively

Permittivity and relaxation time

The frequency dependent complex permittivity ε^*= ε'-j ε'' data was obtained by using calibration process, by using dielectric parameters of raw data. 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 [22,39,41].

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

Conc of NMA	ε_0				T			
	288	**298**	**308**	**318**	**288**	**298**	**308**	**318**
0	26.20	24.3	23.51	20.11	146.3	126.54	115.2	105.5
0.0783	27.12	26.41	25.82	25.01	179	168.23	161.8	127.5
0.1604	30.33	29.55	28.41	27	184.2	175.12	170.2	135.2
0.2467	38.11	36.57	35.02	34.52	205.7	196.02	189.9	150.4
0.3375	45.56	44.12	42.22	42.08	233.4	222.15	215.2	170.1
0.4332	55.41	54.72	53.22	52.2	265.3	255.25	242.1	195.2
0.5341	66.46	65.64	64.11	63.07	318.3	301.64	285.2	228.1
0.6407	78.32	78.29	76.03	75.76	381.3	358.79	332.1	268.1
0.7535	92.25	93.57	91.05	90.89	465.6	432.49	399.2	320.2
0.8731	105.58	107.07	104.11	103	560.2	520.89	472.1	389.3
1	122.65	120.12	117.89	115.98	675.3	586.04	515.9	458.4

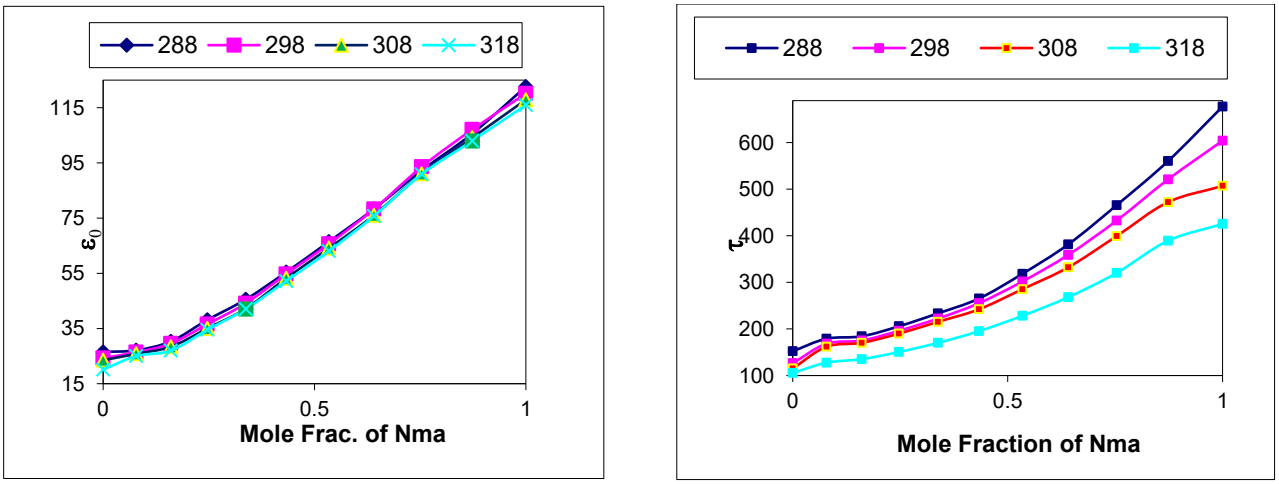

Fig.1: Variation of static dielectric constant (ε_s) and relaxation time (τ) as a function of mole fraction of N Methyl acetamide in Ethanol at temperatures 288, 298, 308 and 318K.

Table 1. reports the values of dielectric parameters obtained by fitting experimental data to Debye equation. It can be seen that by increasing the concentration of NMA in ETH the static permittivity of values increases towards NMA 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 were drawn by shirke et al. [37] for alkyl acetate alcohol system. It is found that in the NMA rich region the dielectric constant increases where as, when the concentration of ETH dominates in the mixture,the static permittivity

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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_2 group of amides.

The relaxation time shows continuous increase with increase in concentration of NMA in ETH and offers hindrance to the rotation of the molecule. The increase in relaxation time with increase in concentration of NMA is to be expected in view of the fact that hydroxyl group reorientation depends to some extent on the no of molecules of NMA to be added in mixture, due to viscosity of mixture get increases. The relaxation time increases with addition of NMA, which indicate that the degree of cooperativity for reorientation of the molecules increases with increasing length and bulk of the cluster increases. The relaxation time increases with increasing size of molecule, ie relaxation time is directly related to the size of molecules. 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.

b) Excess Permittivity and Excess Inverse Relaxation Time

The variation of Excess permittivity (ε^E) and Excess inverse relaxation time with change in mole fraction of NMA at different temperatures is shown in fig (2) and (3). 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 ε_0 & τ 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

$$\varepsilon^E = (\varepsilon_o - \varepsilon_\infty)_m - [(\varepsilon_o - \varepsilon_\infty)_A x_A + (\varepsilon_o - \varepsilon_\infty)_B x_B)]$$

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

1) $\varepsilon^E = 0$ indicates the solute and solvent do not interact at all
2) $\varepsilon^E < 0$ 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
3) $\varepsilon^E > 0$ 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.

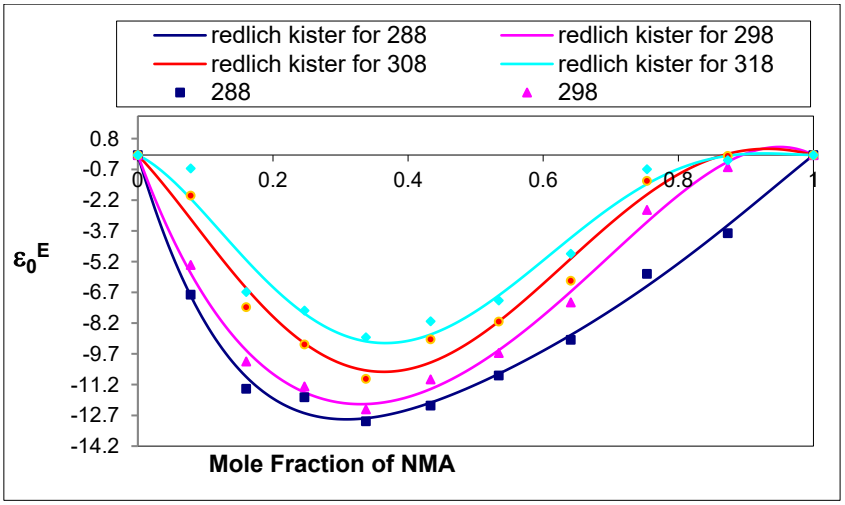

Fig 2. Variation of excess permittivity (ε^E) as a function of mole fraction (x_2) of NMA at 288, 298, 308 and 318K.

Fig 2. shows the plot of excess permittivity against mole fraction of NMA 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

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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

$$(1/\tau)^E = (1/\tau)_m - [(1/\tau)_A X_A + (1/\tau)_B X_B]$$

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 [45]

The information regarding the dynamics of solute solvent interaction gives from this excess property is as follows

1) $(1/\tau)^E = 0$ there is no change in the dynamics of solute solvent interaction
2) $(1/\tau)^E < 0$ the solute solvent interaction produces a field such that the effective dipoles rotate slowly.
3) $(1/\tau)^E > 0$ the solute solvent interaction produces a field such that the effective dipoles rotate faster. ie the field facilitates rotation of dipoles.

The variation of $(1/\tau)^E$ with mole fraction of NMA at 15, 25, 35 and 45^0c and at 11 concentration as shown in following fig.

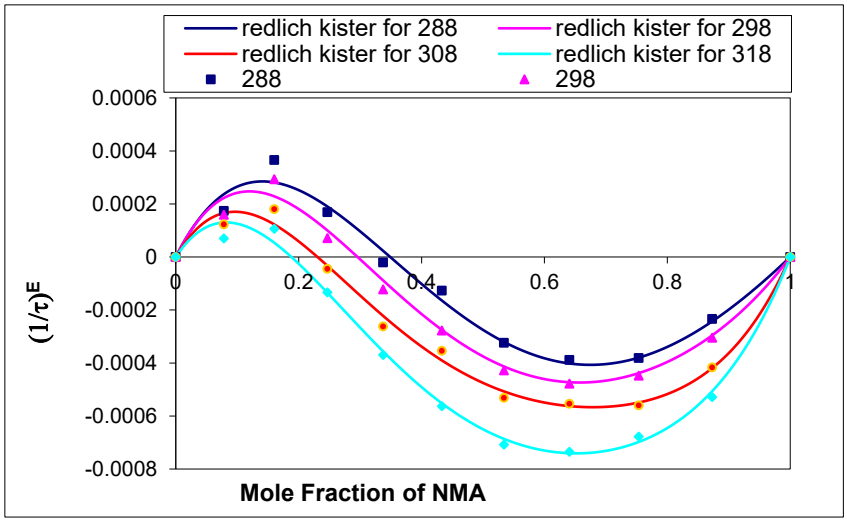

Fig 3. Variation of excess inverse relaxation time $(1/\tau)^E$, as a function of mole fraction (x_2) of NMA at 288, 298, 308 and 318K.

From fig 3 it can be seen that, for all the four-temperature excess inverse relaxation values are positive in ethanol reach region which indicates the formation of such structure which cooperate dipole to rotate faster, while negative in NMA reach region which indicates the formation of linear structure which rotates slowly under the influence of an external varying field. It indicates that addition of NMA to ETG has created a hindering field such that the effective dipoles rotate slowly.

The variation of Excess permittivity and Excess inverse relaxation time with change in mole fraction of NMA at different temperatures is shown in fig (2) and (3). 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 Bj values excess parameters at various concentrations were calculated and used to draw the smooth curves. The estimated values of Bj are

Table.2 The estimated values of Bj coefficients in Redlich-Kister equation for Ethanol- N Methyl acetamide mixture at different temperatures.

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Parameter	B_j coefficient	Temperature			
		288 K	298 K	308 K	318 K
$(\varepsilon_s)^E$	B_0	-44.802	-41.081	-35.759	-30.512
	B_1	29.456	40.881	44.356	42.218
	B_2	-24.556	4.3499	22.758	22.905
	B_3	13.386	12.403	-22.904	-33.892
$(1/\tau)^E$	B_0	-0.0011	-0.0015	-0.0019	-0.0026
	B_1	-0.0029	-0.0025	-0.0021	-0.0024
	B_2	0.0023	0.0023	0.0013	0.0014
	B_3	-0.0005	-0.0014	-0.0025	-0.0023

c) The Kirkwood correlation factor

The structural information about the liquids from the dielectric relaxation parameter may be obtained using the Kirkwood correlation factor gf [17]. This factor is also a parameter for obtaining information regarding orientation of electric dipoles in polar liquids. The values of g^{eff} are given in table (3) and shown in fig. (4).

The variation in g_f with change in volume fraction of N methyl acetamide are given in table (4) and shown in fig.4 The amount of solute – solvent interaction can be accessed using these parameters.

Table No.3 Kirkwood correlation factor g^{eff} values for ETH+NMA

Volume fraction of NMA	288	298	308	318
0	1.00	0.91	0.88	0.75
0.1	0.76	0.74	0.72	0.70
0.2	0.68	0.66	0.63	0.60
0.3	0.71	0.68	0.65	0.64
0.4	0.72	0.70	0.67	0.66
0.5	0.76	0.75	0.73	0.72
0.6	0.81	0.80	0.78	0.77
0.7	0.85	0.85	0.83	0.82
0.8	0.90	0.92	0.89	0.89
0.9	0.94	0.95	0.93	0.92
1	1.00	0.98	0.96	0.95

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Table No.4 Kirkwood correlation factor g_f values for ETH+NMA

Volume fraction of NMA	288	298	308	318
0	1	1	1	1
0.1	0.76	0.79	0.80	0.86
0.2	0.68	0.70	0.69	0.71
0.3	0.71	0.71	0.69	0.73
0.4	0.72	0.72	0.71	0.74
0.5	0.76	0.78	0.77	0.79
0.6	0.81	0.82	0.82	0.83
0.7	0.85	0.87	0.87	0.89
0.8	0.90	0.94	0.93	0.95
0.9	0.94	0.98	0.97	0.98
1	1	1	1	1

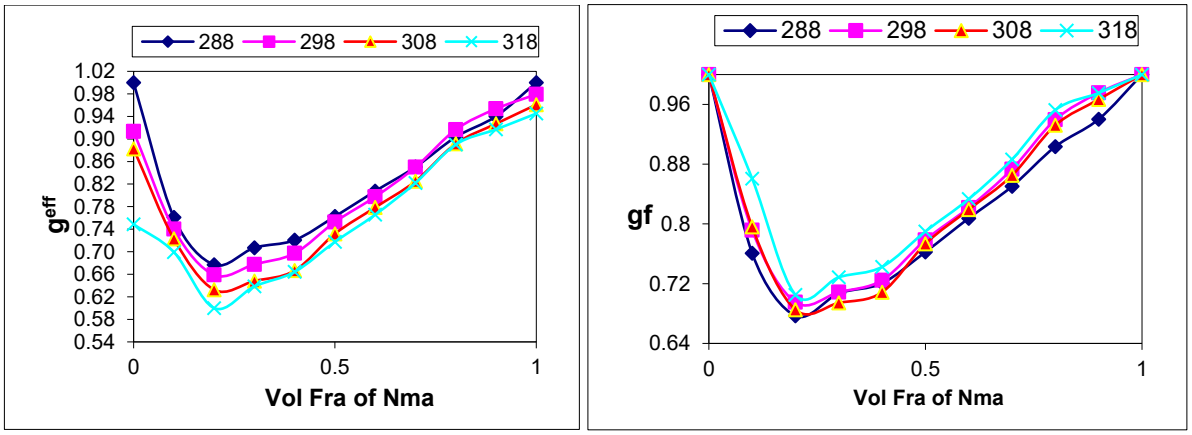

Fig No.4 Variation of Kirkwood correlation factor g^{eff} and gf with variation if volume

d) Thermodynamic parameters

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

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Table No.5 The activation enthalpy (ΔH) entropy (ΔS) of ETH + NMA binary mixture for various concentrations.

Vol. Fr. NMA	Enthalpy **ΔH**	Entropy **ΔS**
0	6618	-33.5
0.1	5440	-39.42
0.2	4674	-42.32
0.3	4776	-42.9
0.4	4852	-43.69
0.5	4797	-44.95
0.6	5429	-44.25
0.7	6038	-43.63
0.8	6567	-43.44
0.9	6484	-45.24
1	9404	-36.62

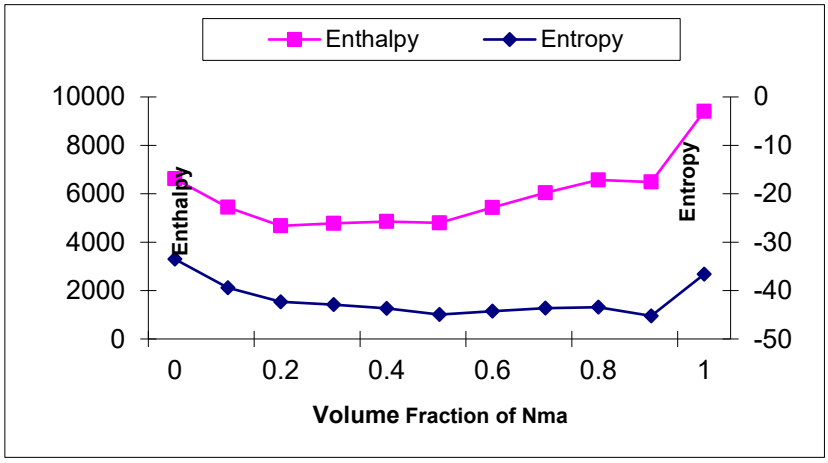

Fig.No. 5 Enthalpy (ΔH) and Entropy (ΔS) of ETH+NMA Binary mixture

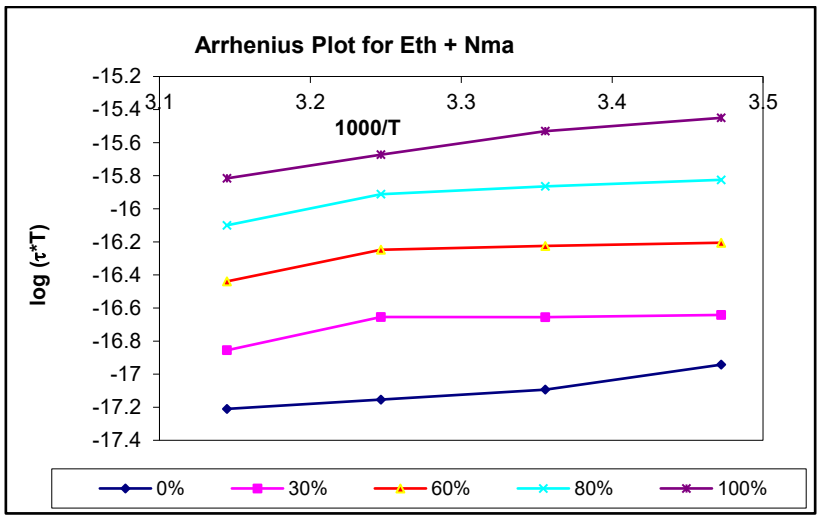

Fig.No.6 Arrahenius plot of ETH+NMA Binary mixture.

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The arrhenius plot of ln(1/t) vs (1000/T) are plotted in fig (6) for various concentrations. The ln (1/T) values increases with increase in concentration of NMA all plots having almost similar slope, which indicates that at all concentrations the activation energy remain almost same.

Discussion:

Static Permittivity and Relaxation Time

From fig.(1a) it can be seen that there is an linear relationship between the values of static dielectric constant (εs) and concentration of NMA in the mixtures. And from fig.(1b) it can be seen that the relationship for the relaxation time is nonlinear. This suggests weak intermolecular interaction due to exposed charge distribution in ETH and NMA molecules.

Excess Parameters

From figure (2) it can be seen that $(\varepsilon s)^E$ is negative for all concentration of NMA in the mixture for all temperature but small positive values are obtained at 90% and above concentration of NMA. This indicates that the molecules of mixture may form multimers structures in such a way that the effective dipoles get reduced, and above 90% of NMA concentration field is quite cooperative and total effective dipoles get increased.

The behavior in $(1/\tau)^E$ is quite different as can be seen in figure (3) The values of $(1/\tau)^E$ are positive for lower concentration of NMA and becomes negative at higher concentration of NMA at all temperatures. This suggests that at higher concentration of NMA the molecular interaction produces hindering field making effective dipole rotation slower. But at lower concentration of NMA the molecular interaction produces a cooperative field and the effective dipoles have more freedom of rotation.

Kirkwood correlation factor

The value of geff is 1 for ETH at 288 K which decreases to 0.75 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 NMA are 1.00 and 0.95 respectively indicating weak dipole-dipole interaction resulting formation of antiparallel dipoles in the pure system of NMA. From table.(4) the value of gf is unity for an ideal mixture and deviation from unity may indicate interaction between two components of the mixture. The gf 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.

Thermodynamic Parameters

From table (5) it can be seen that the molar enthalpy of activation (ΔH) decreases with increase in concentration of NMA in ETH up to equal concentration from 6.618 KJ/mol to 4.797 KJ/mol. This means that less energy is required for group dipole reorientation with increase in volume fraction of NMA in the mixture. The value of molar enthalpy of activation increases from4.797 to9.404 KJ/mol with increase the concentration above 50% of NMA in ETH suggest more energy is required to reorient dipole in NMA rich region. All negative value of molar entropy of activation (ΔS) with volume fraction of NMA indicates relatively high ordered arrangement of molecules in the activated state at equal concentration. Arrhenius plot for the system is shown in fig (4.1.8) 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 (ΔH) 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.

CONCLUSION:-

The dielectric parameter (**ε**) excess parameter (**τ**), the Kirkwood correlation factor have been reported for ETH – NMA mixture for different concentrations and temperatures. These data provide information regarding solute solvent interaction in liquid. The investigated values of dielectric parameter show systematic change in dielectric values with increase in temperature and concentration. The negative excess dielectric parameter values indicate the slower rotation of effective dipoles of the system. The dielectric constant and relaxation time increases with increases with increase in concentration of NMA where as decrease with increase in temperature. These studies suggest a interaction between ETH and NMA

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