International Journal of Sciences: Basic and Applied Research (IJSBAR)

ISSN 2307-4531
(Print & Online)

http://gssrr.org/index.php?journal=JournalOfBasicAndApplied

Dielectric Behavior of Binary Mixture of 2, 3-Dichloroaniline with 2-Methoxyethanol at 20^0 C

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Abstract

Densities, viscosities, refractive indices, dielectric constant $(\in')$ and dielectric loss $(\in'')$ of 2,3-Dichloroaniline (2,3-DCA) and 2-methoxyethanol (2-ME) for different mole fractions of 2,3-Dichloroaniline in binary mixture have been measured at single microwave frequency 10.985 GHz at 20^0C by Surber method at microwave X-band. The values of dielectric parameters $(\in' and \in'')$ have been used to evaluate the molar polarization (P_{12}) and loss tangent $(\tan\delta)$, excess permittivity $(\Delta\in'')$, excess dielectric loss $(\Delta\in'')$.We have also calculated excess viscosities $(\Delta\eta)$, excess refractive index (ΔRI) and excess activation energy (ΔE_a) have also been estimated. These parameters have been used to explain the formation of complexes in the system. It is found that dielectric constant $(\in')$, dielectric loss $(\in'')$, loss tangent $(\tan\delta)$, molar polarization (P_{12}) varies non-linearly but activation energy (E_a), viscosity (η),density (ρ), and refractive index (n) varies linearly with increasing mole fraction in binary mixture of 2,3-Dichloroaniline (2,3-DCA)+2-methoxyethanol (2-ME). Hence, solute-solvent molecular associations have been reported.

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Keywords: Molecular interaction of polar liquids, 2,3-Dichloroaniline, 2-Methoxyethanol, binary mixture, excess parameters.

1. Introduction

Hydrogen bonds in 2-ME are responsible for the striking behavior, and in some instances provide evidence for the role of 2-ME in the sequence of chemical reactions. Hydrogen bonding in 2-ME is believed to be highly cooperative i.e. interaction of 2-ME molecules with a cluster of hydrogen bonded molecules is more likely than interaction with a single molecule to give a dimmer. Binary and ternary representations and prediction of intermolecular interactions with liquid dynamics by using reliable models. Dielectric studies on mixture of polar liquids either in pure state or in inert solvents have been a subject of interest because they provide useful information regarding the molecular complex formation in solution [1]. Molecular mixtures bring about changes in thermodynamic properties like entropy, free energy and also in physical properties like density, molar volume, refractive index, dielectric permittivity etc [2].

Dielectric studies about binary mixtures are helpful to understand the intermolecular interactions in mixture due to hydrogen bonding and dipole-dipole interaction. Molecules containing -OH group forms hydrogen bonding with moleculesin mixture are important aspect of molecular orientation and molecules interaction [3-8]. Dielectric behavior and hydrogen bond molecular interaction of formaamide with mono, dia and tri-hydricalcohols are studied [9]. Dielectric relaxation of binary mixture of chloro substituted and flouro substituted anilines with 1- propanol using TDR technique is studied at different temperatures [10].

A survey of earlier literature shows that 2,3-DCA+2-ME binary mixture could provide useful information regarding the molecular interaction and formation of complexes, it also help to study the hydrogen bonding in the binary mixture. A dielectric investigation of binary mixture containing interacting molecules helps to detect the formation and composition of complexes in them. Experimental investigation of dielectric properties of polar liquids from microwave absorption in binary mixtures in earlier investigation shows viscosity, density, refractive index, thermodynamic parameters and dielectric parameters does not changes linearly. The deviation from linearity of these parameters is termed as excess parameters, which are useful to understand nature of bonding between binary mixtures The molecular complex formation can be investigated by studying molar polarization, excess viscosity, excess dielectric constants, excess activation energy and excess refractive index etc.

2. Materials

2,3-Dichloroaniline (GC Grade) is obtained from Sigma-Aldrich. 2-Methoxyethanol (AR Grade) were obtained from M/S Sd. Fine chemical, Mumbai, India without further purification the two liquids according to their proportions by volume were mixed well and kept 6h in well stoppered bottles to ensure good thermal equilibrium. These liquids used as solute and solvent.

3. Measurements

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 by Surber [11] for microwave measurements are used. All the measurements are carried out at temperatures 20^0C 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 [12] and viscosity by LVDL, V-pro II Brook field viscometer with an accuracy of $\pm$1% (USA) [13]. 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. Refractive indices for sodium D-line were measured by using Abbe's refractometer, having accuracy up to the third place of decimal. The X-band microwave bench is used to measure wavelengths in the dielectric and the voltage standing wave ratio (VSWR).

We determined the density (ρ), viscosity (η), refractive index (n), dielectric constant (ε'), dielectric loss (ε"), loss tangent $(\tan\delta)$, activation energy (E_a) and molar polarization (P_{12}) of dilute solutions of binary mixture of 2,3-DCA+2-ME. All the measurements are carried out at temperatures 20^0C and the temperature is thermostatically controlled within $\pm 0.5^0$C. Microwave techniques have been used method suggested by Surber [11].

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

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

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

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 precision of measurements for the wavelength with the available X-band microwave bench is $\pm 0.001cm$ corresponding to this accuracy value, the error in the

measurements of ε' is estimated. For simplification, involved errors due to non zero impedance of the short circuit plunger are ignored. The errors of measurements are calculated by using the conventional method of error analysis [14]. Over all estimated accuracy of measurements for ε' and ε'' by this method is about $\pm 1\%$ and $\pm 5\%$ respectively. In order to determine the dielectric wavelength (λ_d) the movable short of the liquid cell was moved in and out and corresponding reflection coefficient was measured using crystal detector in the directional coupler [15].

The relationship between the reflected power and depth of liquid columns is given by a damped sinusoidal curve. The distance between two adjacent minima of this curve gives λ/2. Thus the knowing the values of dielectric wavelength (λ_d), free space wavelength (λ_0), cut off wavelength (λ_c) and waveguide wavelength (λ_g) were determined by Surber relations [11, 15]. To determined the $\varepsilon'$and ε'' were measured by reflectometric technique by measuring reflection coefficient from the air dielectric boundary of the liquid [16]. The ε' and ε'' for different mole fractions of 2,3-DCA in the binary mixture of 2,3-DCA+2-ME are measured at 20^0C.

The free energy of activation (E_a) of the viscous flow for the pure liquids and their binary mixtures is obtained by using the following equation [17].

$$\eta = \left(hN / V\right) \exp\left(E_a / RT\right) \qquad \text{…...……. (3)}$$

Where η is the viscosity and V is the molar volume of the binary liquid and other symbols have their usual meaning. The values of molar polarization of the binary mixture are obtained by using the formula [18].

$$P_{12} = \left(\frac{\varepsilon'-1}{\varepsilon'+2}\right)\left[\frac{M_1X_1 + M_2X_2}{\rho}\right] \qquad \text{…….. (4)}$$

Where M_1 and M_2 are the molecular weight, X_1 and X_2 is the mole fraction of the constituents of the binary mixture.

The excess dielectric properties such as excess permittivity $\left(\Delta \in '\right)$, excess loss factor $\left(\Delta \in ''\right)$,excess activation energy $\left(\Delta E_a\right)$ and excess viscosity (Δη) etc. can be obtained by using the relations of the form [19].

$$\Delta Y = Y_m - \left(X_1Y_1 + X_2Y_2\right) \qquad \text{…….. (5)}$$

When ΔY any excess parameter and Y is refers to the above mentioned quantities, that is, permittivity $\left(\in '\right)$, loss factor $\left(\in ''\right)$, activation energy $\left(E_a\right)$ etc. The subscripts m, 1 and 2 used in the above equation are respectively for the mixture, liquid (1) and liquid (2). X_1 And X_2 are the mole fractions of the two components in the liquid mixtures. Qualitative information regarding the solute-solvent interaction may be obtained by the excess permittivity as follows.

The equation (5) for the excess permittivity $(\Delta \in ')$ can be written as

$$\Delta \in ' = \in_m^{'} - \left(X_1 \in_1^{'} + X_2 \in_2^{'} \right) \qquad \text{……...……. (6)}$$

We have three cases, if

1. $\Delta \in ' = 0$: This indicates the solute and solvent do not interact at all.
2. $\Delta \in ' < 0$: This means that solute and solvent interact in such a way that the total effective dipoles get reduced. The solutes and solvents may form multimers leading to the less effective dipoles. In general, the negative excess permittivity indicates the formation of multimers in the binary mixtures. The excess permittivity $(\Delta \in ')$ is associated with the polarization.
3. $\Delta \in ' > 0$: In this case the solute and solvent interact in such a way that the effective dipoles tend to be more. This means the reduction of multimers either in solute or solvent due to the interactions between solute and solvent.

4. Results and Discussion

The values of density (ρ), viscosity (η), refractive index (n), dielectric constant (ε'), dielectric loss (ε''), loss tangent (tanδ) ,activation energy (E_a) and molar polarization (P_{12}) for viscous flow with increasing mole fraction (X) of 2,3-DCA for the binary mixtures of 2,3-DCA+2-ME are reported in Tables 1 at 20^0C.

The density of binary mixture of 2,3-DCA and 2-ME are increasing as mole fraction of 2,3-DCA in the binary mixture is increasing. This is expected because density of pure 2, 3-DCA is more than that of pure 2-ME. This variation of density as shown in Tables 1. From Tables 1 it can also be seen that refractive index (n) is increasing with increase in mole fraction of 2,3-DCA in the binary mixture. This increase in refractive index (n), viscosity (η) and activation energy (E_a) is further supported by the increase in density of the binary mixture with increase in mole fraction of 2,3-DCA.

The variation of dielectric constant (ε') versus mole fraction (X) of 2,3-DCA as shown in (fig 1).The values of dielectric constant increase with increase in mole fraction of 2, 3-DCA in the binary mixture. This can be attributed to the increase in hydrogen bonding in the mixture. This is because there is dipole-dipole interactions in pure 2, 3-DCA and strong hydrogen bonding as in pure 2-ME. Similar results have been reported by Vishwam [20]. From (fig 1). it shows deviation from linearity, indicating the complex formation in the binary mixture as suggested by Job [21]. The deviation is maximum at about X=0.2905 mole fraction of 2,3-DCA, hence formation of 1:2 complex in the binary mixture.

The variation of loss tangent (tanδ) and mole fraction (X) of 2,3-DCA as shown in (fig 2) which shows that the microwave energy absorption is maximum in the mixture than in pure liquids. The existence of prominent minima in the tanδ is X=0.8269 mole fraction of 2,3-DCA. An interactions causing association between two

types of molecules may be responsible for nonlinear behavior. Similar results have been reported by Kawle [22] for β-picoline and O-chlorophenol mixture.

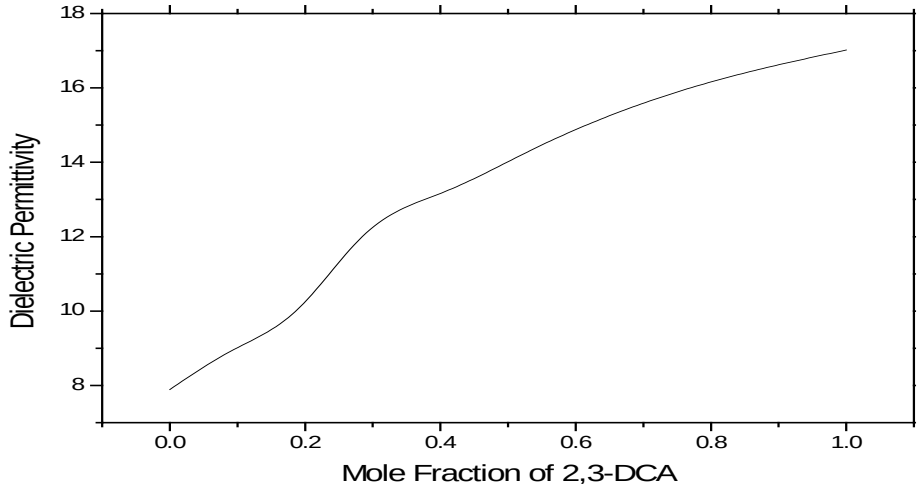

Figure (1):- Plot of Dielectric Permittivity (ε') with mole fraction of 2,3-DCA+2-ME

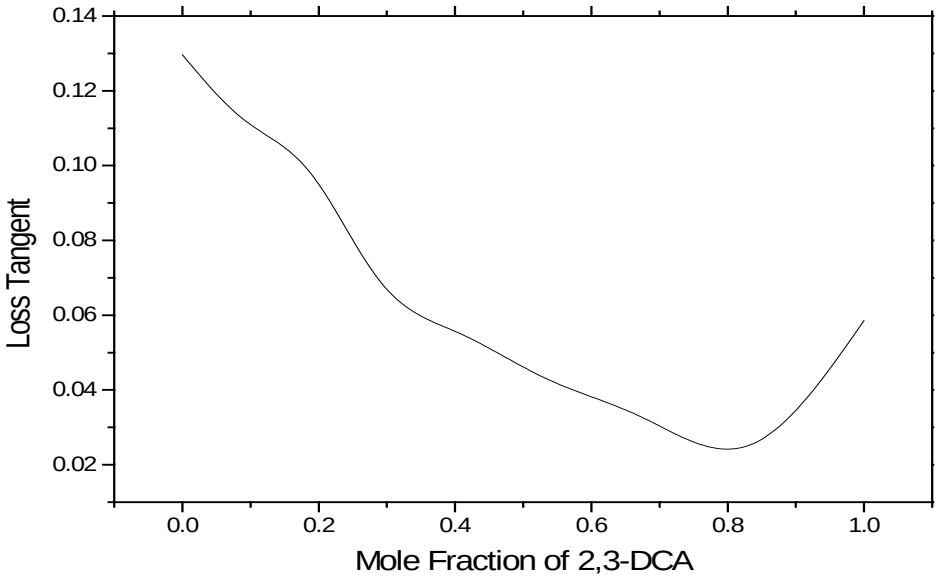

Figure (2):- Plot of Loss Tangent (Tanδ) with mole fraction of 2,3-DCA+2-ME

The variation of molar polarization (P_{12}) versus mole fraction (X) of 2,3-DCA in the binary mixture is represented in the (fig 3). The intersection of straight lines at X=0.4 representing separate region of high and low concentration of 2,3-DCA can be interpreted as a point of maximum concentration which corresponds to 1:1 complex for the system. The amount of complex present is responsible for the shape of polarization curve. Thus

this result regarding the formation of complex is supported by our earlier conclusion made for the tanδ against mole fraction of 2, 3-DCA curve for the system. Strong positive deviation from additivity in molar polarization (P_{12}) is an indication of tendency towards formation of a miscibility gap. The same results have been reported by Narwade [23].

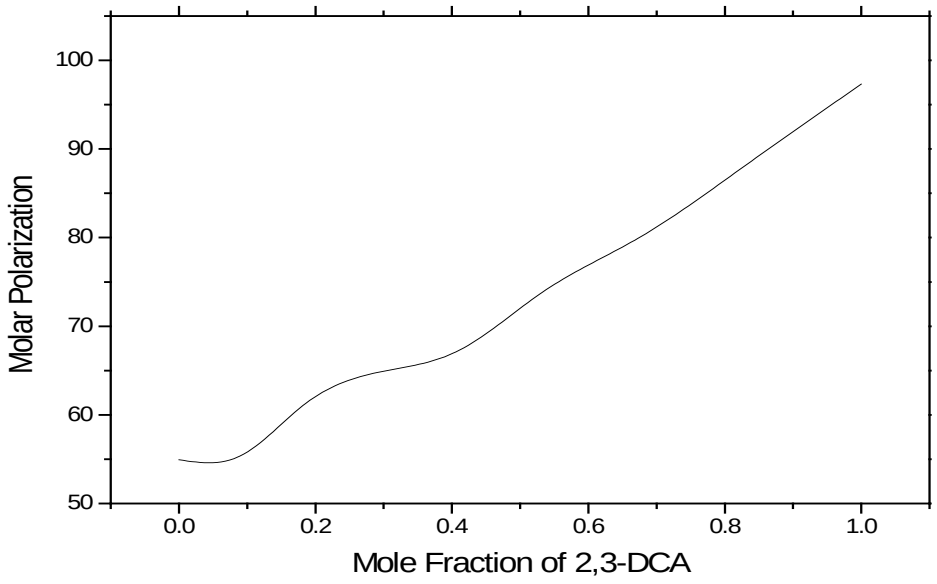

Figure (3):- Plot of molar Polarization (P_{12}) with mole fraction of 2,3-DCA+2-ME

The excess parameters of binary liquid system are a measure of deviation from the ideal behavior of the mixture and are more sensitive towards the molecular interactions in the liquid mixture [10]. The values of excess dielectric permittivity (Δε'), excess viscosity (Δη), excess refractive index (Δn) and the excess activation energy (ΔE_a) for 2,3-DCA+2-ME are reported in Table 2.

The variation of excess dielectric constant (Δε') versus mole fraction (X) of 2,3-DCA as shown in (fig 4). From figure Δε' is positive over entire concentration rang of mole fraction of 2,3-DCA. Investigation on concentration dependent excess dielectric constant (Δε') values of mixed dipolar solvents provides information regarding change in number of parallel alignment dipoles contributed to the dielectric polarization. The strength of hydrogen bonded interactions and the molar ratio of stable adduct.The similar results have been reported by Vijaya Krishana [24]. The maximum peak of excess dielectric constant (Δε') at X=0.4056 mole fraction of 2,3-DCA.

The variation of excess viscosity (Δη) versus mole fraction (X) of 2,3-DCA as shown in (fig 5). From figure it shows that excess viscosity (Δη) values are positive for the whole composition range. This is due to charge transfer and hydrogen bonding interactions which leads to the formation of complex species between unlike molecules thereby resulting in positive (Δη) values. The similar results have been reported by Narwade [23]. The maxima of Δη occurs at X=0.5321 mole fraction of 2,3-DCA.

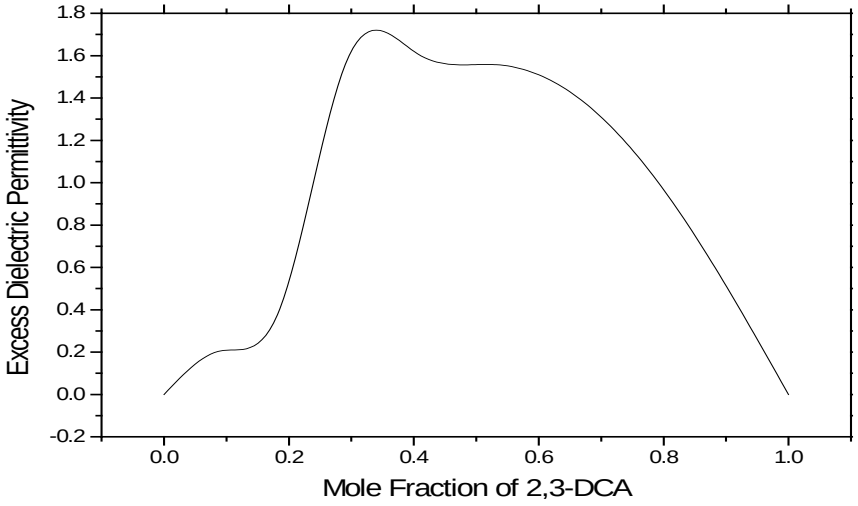

Figure (4):- Plot of excess dielectric constant ($\Delta\varepsilon'$) with mole fraction of 2,3-DCA+2-ME.

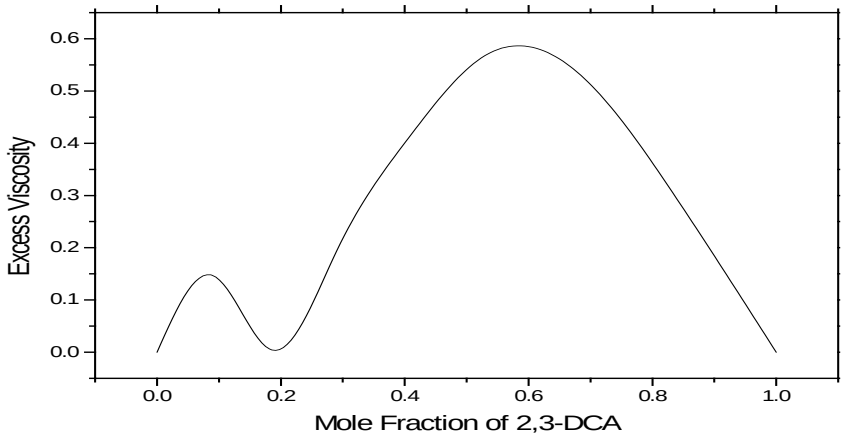

Figure (5):- Plot of excess viscosity ($\Delta\eta$) with mole fraction of 2,3-DCA+2-ME.

The variation of excess refractive index (Δn) versus mole fraction of 2,3-DCA as shown in (fig 6). From figure the values of Δn are positive for entire concentration of mole fraction of 2,3-DCA in binary mixture of 2,3-DCA+2-ME and their magnitude increase with increase in substitution of aniline group on -OH group of alcohol. It may be noted that such values are due to electronic perturbation of the individual molecules during mixing and therefore depend very much on the nature of mixing molecules. The extent of positive deviations maximum at X=0.5321 mole fraction of 2,3-DCA, thus this results supporting to our earlier conclusion in (fig 5) for excess viscosity of strong interactions in the systems of 2,3-DCA and 2-ME. Positive peak in Δn values indicate strong interactions between the componants of binary mixture. Similar results are already predicted by Nemmaniwar [19].

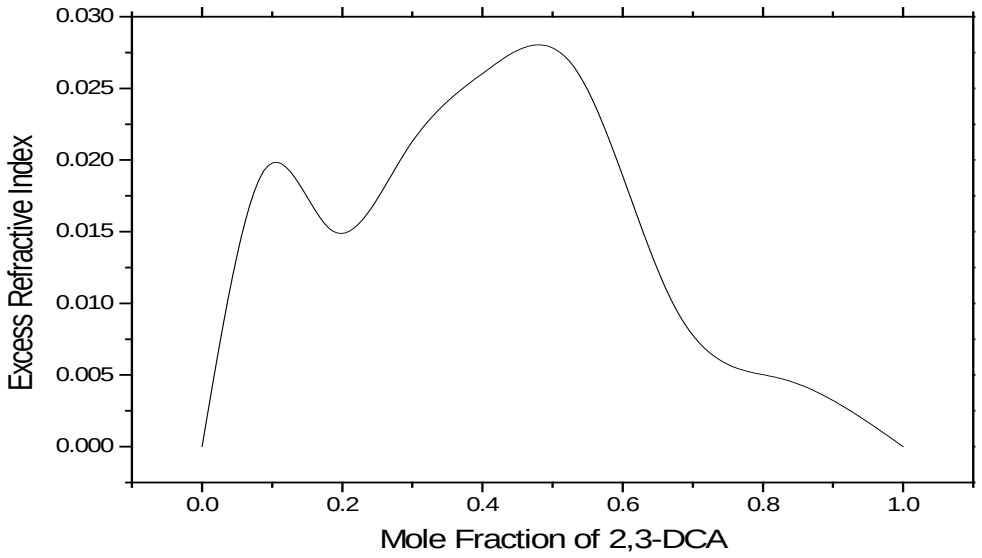

Figure (6):- Plot of excess Refractive Index (ΔRI) with mole fraction of 2,3-DCA+2-ME.

The variation of excess activation energy (ΔE_a) versus mole fraction X of 2,3-DCA as shown in (fig 7). From Figure ΔE_a positive, the positive deviation of ΔE_a viscous flow in the present investigation is due to increase in the internal energy of viscous flow, thus this results supporting to our earlier conclusion in (fig 4) for $\Delta\varepsilon'$ of strong interactions in the systems of 2,3-DCA and 2-ME. Similar results are already predicted by Rana [10].

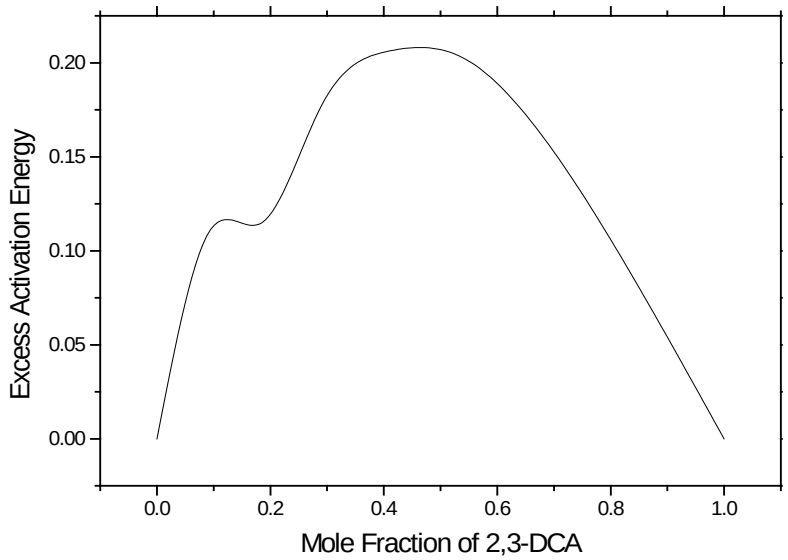

Figure (7):- Plot of excess Activation Energy (ΔEA) with mole fraction of 2,3-DCA+2-ME.

Table 1: Mole fraction (X) of 2,3-DCA density (ρ), viscosity (η), refractive index (n), dielectric constant (ε'), dielectric loss (ε''), loss tangent $(\tan\delta)$, activation energy (E_a) and molar polarization (P_{12})

X	ρ gm/cm^3	η cp	n	ε'	ε''	$\tan\delta$	Ea (Kcal/mol)	P_{12}
0.00000	0.9650	2.14	1.4041	7.8920	1.0231	0.1296	7.0837	54.94378
0.08883	1.0970	2.87	1.4407	8.9072	1.0024	0.1125	7.2721	55.33446
0.18532	1.1260	3.32	1.4553	9.9873	0.9847	0.09859	7.3656	61.27233
0.29051	1.2281	4.24	1.4813	12.1020	0.8346	0.06896	7.5227	64.78415
0.40563	1.3272	5.21	1.5094	13.2040	0.7292	0.05522	7.6549	67.10302
0.53214	1.3473	6.20	1.5342	14.3070	0.6181	0.04320	7.7666	73.78458
0.67184	1.3864	7.09	1.5450	15.4060	0.5071	0.03291	7.8527	79.88983
0.82690	1.3985	7.88	1.5700	16.2943	0.4061	0.02492	7.9205	87.9637
1.00000	1.4021	8.70	1.5990	17.0210	0.9962	0.05852	7.9841	97.32982

Table 2: Excess parameters $\Delta\varepsilon'$, $\Delta\eta$, ΔRI and ΔE_a along with mole fraction (X) of 2,3-DCA for the binary mixture liquid system of 2,3-DCA+2-ME at 20^0 C

X	**$\Delta\varepsilon'$**	**$\Delta\eta$**	**ΔRI**	**ΔEA**
0.00000	0.00000	0.00000	0.00000	0.00000
0.08883	0.20427	0.14728	0.01929	0.10842
0.18532	0.40351	0.00430	0.01508	0.11504
0.29051	1.55793	0.19425	0.02058	0.17742
0.40563	1.60900	0.40907	0.02624	0.20597
0.53214	1.55709	0.56916	0.02639	0.20376
0.67184	1.38077	0.54273	0.00996	0.16408
0.82690	0.85353	0.31554	0.00474	0.09226
1.00000	0.00000	0.00000	0.00000	0.00000

5. Conclusion

The dielectric constant, dielectric loss, loss tangent, viscosity, density, refractive index and excess parameters have been reported for 2,3-DCA+2-ME binary mixture at the various mole fraction at 20^0 C. This suggests the strong interaction between the alcohols and amine molecules. Density, viscosity, refractive index and activation energy is increasing with increase in mole fraction of 2,3-DCA in the binary mixture. This increase in density, viscosity, refractive index and activation energy is further supported by the increase in molar polarization of the

binary mixture with increase in mole fraction of 2,3-DCA. Dielectric constant, dielectric loss and loss tangent varies non linearly with mole fraction of 2,3-DCA in binary mixtures. Excess parameter $\Delta\varepsilon'$ and ΔE_a shows strong interaction at mole fraction X=0.4056 of 2,3-DCA in binary mixtures and the $\Delta\eta$ and ΔRI shows strong interaction at mole fraction X=0.5321 of 2,3-DCA in binary mixtures at 20^0C.

Acknowledgments

The authors thanks to the Principal, Yeshwant Mahavidyalaya Nanded for providing necessary laboratory facilities.

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