Smart Psycho Pharmaceutical Drug Atarax and Methanol Binary Mixture Dielectric Characterization for Understanding Of Molecular Structure

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Abstract

The dielectric relaxation study for hydroxyzine hydrochloride (Atarax) and Methanol binary mixture has been carried out using the time domain reflectometry (T.D.R.) technique at temperature 283K, 288K, 293K and 298K and at different concentration, in the frequency range of 10MHz to 50Ghz. Further, Fourier transform and least squares fit method and Debay model have been used to obtain dielectric parameter viz. static permittivity, relaxation times. Excess permittivity, excess inverse relaxation time, Kirkwood correlation factor. Bruggeman factor and thermodynamic parameters have been obtained from the complex permittivity spectra. The investigation shows the systematic change in dielectric parameters of the system with change in temperature and concentration. There is almost linear relationship between the values of εs, however τ is nonlinear suggest weak intermolecular interaction. And its excess parameters values are positive and negative respectively. fB shows small deviation from ideal behavior. The geff values are greater than unity for all temperature suggests parallel orientation of electric dipole and gf deviates from unity indicate interaction between two components of mixture. The molar enthalpy of activation represents need of energy is nonlinear and entropy also nonlinear. Arrhenius shows change in activation energy of the system. The results obtained are used to interpret the nature and kind of solute-solvent interaction.

1 Introduction

The study of the dielectric behavior of liquid is very significant in understanding the structure and molecular interactions in the liquid. The dielectric constant specifies the solvent's ability to decrease the field strength of the electric field surrounding the charged particle impressed with it. This decrease is then compared with the field strength of the charged particle in vacuum (Mohsen-Nia et.al.2010). Macroscopic parameters such as dielectric constant have been extensively used for explanation of solvent effects. The dielectric constant is one of the fundamental properties that must be known to utilize theories of electrolyte solutions (Wang, P. et.al. 2001). The dielectric constant is

an important physicochemical parameter, as it is related to many important physical and biological applications (Nelson S. O.et.al. 2006, Nelson S. O.et.al. 1980, Dennis S. et al. 2006, Fakhree M. A. A. et al.2010, Gorman W. G. et al.1963,Shukla A. K. et al. 2000). the dielectric constant of a solvent is a relative measure of its polarity and its measurements are often used for evaluation of the characteristics of the liquid solutions (Hansen J. P. et al.1986).

2 Literature Survey

Blood is a highly important functional body fluid, it delivers oxygen to the vital parts, it transports nutrients, vitamins, and metabolites and it also is a fundamental part of the immune system. Therefore the precise knowledge of its constituents, its physical, biological, and chemical properties and its dynamics is of great importance. Especially its dielectric parameters are of relevance for various medical applications (E. H. Grant et al. 1978). Drug solubility in water and organic solvent plays an important role and affects many pharmaceutical processes. Maybe changes in dielectric constant of the medium have a dominant effect on the solubility of the ionizable solute in which higher dielectric constant can cause more ionization of the solute and results in more solublization (AAPS Pharma, SciTech 2010).

The blood serves as the principal transport medium of the body, carrying oxygen, and nutrients, messages to the tissues and waste product and CO_2 to the organs of excretion. In other words, blood is described as a fluid connective tissue. The blood plays many important roles in coordinating the individual cells into a whole complex organism. (K. Asami et al) reported the electrical properties like membrane capacitance Cm, dielectric increment ΔK, of yeast cells suspended in KCl solution by bridge method in the frequency range of 1kHz to 100 MHz. The Cm was obtained to be 1.6 μF/cm2. They also developed the yeast cell model to explain their results. Schwan (H. P. Schwan et al. Vol. 120. 1985) studied the electrical properties of biological cells and tissues at very low frequencies and discussed the mechanisms responsible for such properties. Schwan (H. P. Schwan et al. Vol. 110. 1985) analyses the dielectric data of biological material obtained from advanced dielectric techniques. He proposed three major and distinct relaxation effects which characterise the total dielectric response from d.c. to GHz, and several minor ones are superimposed. Schwan (H. P. Schwan et al. Rindi pelunum press.1985) summarised the electrical properties of biological cells and tissues over the total investigated frequency range. He also discussed mechanisms responsible for observed frequency dependencies and indicated the most possible sites for electromagnetic field interactions. Schwan (H. P. Schwan et al.1988) studied dielectric properties such as dielectric increment, membrane capacitance of biological cells by electro rotation method. He summarised biological effects of non-ionizing radiation, which is closely related to electro physiology. Pethig (R. Pethig et al. CRS press) analyses proton transport in proteins along with pH effects on protein structure. He reviewed the work on electrical and dielectric properties of protein at low hydration content to indicate proton transport. Takashima et al (R. Pethig et al.1988) measured dielectrophoretic properties of micrococcus lysodeiktcus in the frequency range of 20 Hz to 4 MHz as a function of ionic strength of suspending electrolyte. They concluded that low frequency DEP response is dominated by electrical properties of cell wall. The existing dielectric theories are insufficient in explaining the results, as they do not consider the inhomogeneous and charge structure of the organism. (Hawkes and Pethig 1988) noted dielectric properties of lysozyme-compressed powder as a function of hydration and pH at which the samples were lyophilized. They concluded that the dielectric dispersion in α-region appear in the range 104 Hz to 105 Hz for lysozymes of hydration ranging from 5 - 20 % weight water is related to the state of ionization of acidic and basic groups in the protein structure. (K.R. Foster and Schwan 1989) presented a very useful review of the work done on dielectric properties of tissues and biological particles in the past. It is a historical survey on electrical properties of biological materials. Various dielectric relaxation mechanisms and

dielectric dispersions in tissues are described. Dielectric properties of some tissues like muscle, bone, blood are summarised. (Basharath Ali et. al. 2007) studied anisotropy in permittivity and resistivity of fresh and oven dried ox muscle and heart tissues. They reported that anisotropy in permittivity and resistivity was significant in fresh tissues, while it was lacking in dry tissues. Further, dielectric constant, dielectric loss and conductivity were high and resistivity was low in fresh samples when compared to oven dry tissues. (Basharath Ali et. al. 2008) investigated dielectric parameters (dielectric constant, dielectric loss, conductivity or resistivity) of different types of tissues of liver, kidney and brain of the animal Ox at 1 KHz frequency. They attributed significant variation in these parameters to the extent of hydration, molecular composition, presence of certain elements in traces, structural and morphological differences in cells and tissues, and concluded that structural constituents and molecular composition of tissues have integrated activity in influencing the dielectric properties of tissues.

A drug molecular interaction is an important phenomenon in physiological media. Dielectric study provides information regarding the molecular interaction. The chemicals used in the present work were psychopharmaceutical drug Hydroxyzine hydrochloride (Atarax), Atarax reduces activity in the central nervous system. Atarax is used as a sedative to treat anxiety and tension. (http://www.chemspider.com/chemical-structure .82634.html) And Methanol (https://en.wikipedia.org/wiki/methanol). Due to its antagonistic effects on several receptor system in the brain, atarax has strong anxiolytic and mild antiobsessive as well as antipsychotic properties.(Simons FE et al. 1984) Several reactions have been noted in manufacturer guidelines – deep sleep, incoordination, sedation, calmness, and dizziness have been reported in children and adults, as well as others such as hypotension, tinnitus, and headaches.(UCB South-Africa et al.2004)it is synthesized by the alkylation of 1-(4-chlorobenzohydril) piperazine with 2-(2-hydroxyethoxy)ethyl chloride (H. Morren 1959). The information regarding interaction between the components in the liquids as well as the orientation of the dipoles in the mixture reported by (A. Pratima et.al. 2014). The interaction of alcohol and amide binary mixture was attributed to some sort of molecular interaction which may take place between the alcohols and substituted amides (A. Arunkumar et. al. 2016)

3 Experimental

3.1 Chemical and Sample Preparation

The chemical used in the present work is Atarax $C_{21}H_{29}Cl_3N_2O_2$ and methanol CH_3OH are of spectroscopic grade, obtained commercially with 99% purity and used without further purification. The solutions were prepared at six different compositions in steps of 20 % by volume. These volume fractions are converted to mole fractions for further calculations. Using this volume percentage the weight fraction is calculated (P. B. Undre et al. 2007) as

$$X_A = \frac{V_A \rho_A}{[(V_A \rho_A) + (V_B \rho_B)]} \qquad (1)$$

where, V_A and V_B are the volume and ρ_A and ρ_B is the density of liquid A(Atarax) and B (Methanol) respectively.

3.2 Time domain reflectometry setup and data acquisition

The Tektronix DSA8300 sampling oscilloscope sampling main frame with the dual channel sampling module 80E10B has been used for time domain reflectometry. The sampling module provides 12ps incident and 15ps reflected rise time pulse. The coaxial cable used to feed pulse has 50 Ohm

impedance, inner diameter of 0.28mm and outer diameter of 1.19mm. Sampling oscilloscope monitors changes in pulse after reflection from end of line. Reflected pulse without sample $R_1(t)$ and with sample $R_x(t)$ were recorded in time window of 5 ns and digitized in 2000 points. To minimize the signal to noise ratio the signal reflected is obtained from 512 samples after an optimum average of 100 times for each record. The subtraction [$p(t) = R_1(t) – R_x(t)$] and addition [$q(t) = R_1(t) + R_x(t)$] of these pulses are done in oscilloscope memory. These subtracted and added pulses are transferred to PC through compact disc for further analysis (manual of T.D.R.).

3.3 Data Analysis

The time dependent data were processed to obtain complex reflection coefficient spectra, $\rho^*(\omega)$ over the frequency range from 10 MHz to 50 GHz using Fourier transformation (C. E. Shannon 1949, H. A. Samulan 1951) as

$$\boldsymbol{\rho^*(\omega) = \left[\frac{c}{j\omega d}\right]\left[\frac{\rho(\omega)}{q(\omega)}\right]} \qquad (2)$$

Where, $\rho(\omega)$ and $q(\omega)$ are Fourier transforms of [R_1 (t) - R_x (t)] and [R_1 (t) + R_x (t)], respectively. C is the velocity of light, ω is angular frequency and d is the effective pin length and j = root (-1). The complex permittivity spectra (S. Mashimo et al. 1989) $\varepsilon^*(\omega)$ were obtained from reflection coefficient spectra $\rho^*(\omega)$ by applying a bilinear calibration method. The experimental values of $\varepsilon^*(\omega)$ are fitted by Debye equation (P. Debye1929).

$$\boldsymbol{\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_0 - \varepsilon_\infty}{1 + j\omega\tau}} \qquad (3)$$

where, ε_0, ε_∞ and τ as fitting parameters. The value of ε_∞ was kept to be constant as the fitting parameters are not sensitive to ε_∞. A non-linear least squares fit method used to determine the values of dielectric parameters.

3.4 Permission Excess permittivity and excess inverse relaxation time

Information regarding to solute- solvent interaction may be obtained by excess properties *i.e.* static dielectric constant and relaxation time in the mixtures. The excess permittivity is defined as

$$\boldsymbol{\varepsilon_0^E = (\varepsilon_0)_m - [(\varepsilon_0)_A X_A + (\varepsilon_0)_B X_B]} \qquad (4)$$

Where, X is the mole fraction and the subscript m, A and B represent mixture, solute and solvent respectively. The excess permittivity provides qualitative information about multimer formation in the mixture

and, the excess inverse relaxation time defined as

$$\boldsymbol{\left(1/\tau\right)^E = \left(1/\tau\right)_m - \left[\left(1/\tau\right)_A X_A + \left(1/\tau\right)_B X_B\right]} \qquad (5)$$

Where, $\left(1/\tau\right)^{E}$ is the excess inverse relaxation times, which represent the average broadening of dielectric spectra. Information regarding the dynamics of solute solvent interaction obtained from this excess property is as (S. B. Sayyad et al. 2011).

3.5 The Bruggeman factor

Bruggeman mixture formulae (D.A.G. Bruggeman 1935, U. Kaatze 1987) can be used as evidence of molecular interaction in binary mixture. The Bruggeman modified equation for mixture is given by expression.

$$f_B = \left(\frac{\varepsilon_{0m}-\varepsilon_{0B}}{\varepsilon_{0A}-\varepsilon_{0B}}\right)\left(\frac{\varepsilon_{0A}}{\varepsilon_{0m}}\right)^{1/3} = 1 - \emptyset_B \qquad (6)$$

According to this equation linear relationship is expected which will give a straight line when f_B plotted against $\emptyset_B$. Any deviation from this linear relation indicates molecular interaction.

3.6 The Kirkwood Correlation factor

Kirkwood correlation factor (A. C. Kumbharkhane et al 1993) 'g' is also a parameter containing information regarding orientation about parallel or antiparallel alignment of dipoles. The effective angular correlation g^{eff} between molecules is calculated using modified form of equation.

$$\frac{4\pi N}{9KT}\left[\frac{\mu_A^2\rho_A\varphi_A}{M_A} + \frac{\mu_B^2\rho_B\varphi_B}{M_B}\right] g^{eff} = \frac{(\varepsilon_{0m}-\varepsilon_{\infty m})(2\varepsilon_{0m}-\varepsilon_{\infty m})}{\varepsilon_{0m}(\varepsilon_{\infty m}+2)^2} \qquad (7)$$

Where μ is the dipole moment in Debye, ρ is the density at temperature T. M is molecular weight, K is Boltzmann constant, N is Avogadro's number, $\emptyset_A$is volume fraction of liquid A, $\emptyset_B$ is volume fraction of liquid B.

The Kirkwood Correlation factor g is also a parameter containing information regarding orientation of electric dipole in polar liquids. The g for the pure liquid is given by the expression

$$\frac{4\pi N\mu^2\rho}{9KTM} g = \frac{(\varepsilon_s-\varepsilon_\infty)(2\varepsilon_s+\varepsilon_\infty)}{\varepsilon_s(\varepsilon_\infty+2)^2} \qquad (8)$$

Where μ is the dipole moment, ρ is the density at temperature T, M is the molecular weight, K is the Boltzmann constant, and N is Avogadro number (Sayyad S. B. 2012).

3.7 The Kirkwood Correlation factor

The thermodynamic parameters such as molar energy of activation ΔH and molar entropy of activation ΔS were obtained by using the Eyring rate equation (H. Eyring 1936)

$$\tau = (h/kT)exp[(\Delta H - T\Delta S)/RT] \qquad (9)$$

4 Result and Discussion

4.1 Permittivity and Relaxation Time

The static permittivity (ε_0) and relaxation time (τ) for the binary mixture as given in Table1, obtained by fitting experimental data with the Debye equation at four different temperatures. In this study, the variation in the static permittivity and relaxation time of Atarax with Methanol is as shown in Fig (1) and (2) respectively. It shows nonlinear variation after 60% of mole fraction of ethanol in the solution with change in mole fraction. This suggests that the intermolecular association is taking place in this region.

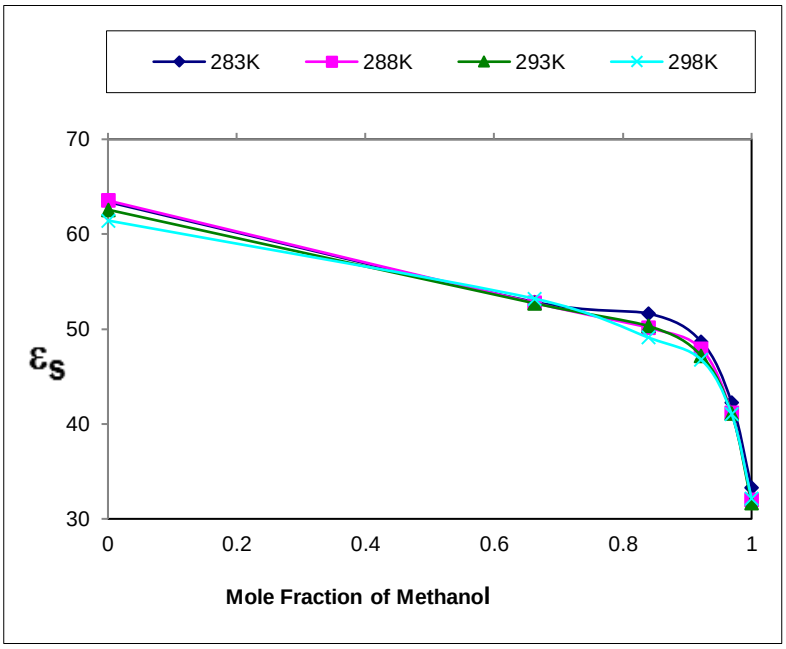


Figure 1: Variation of static dielectric constant (ε_s) as a function of mole fraction of Methanol at temperatures 283, 288, 293 and 298K.

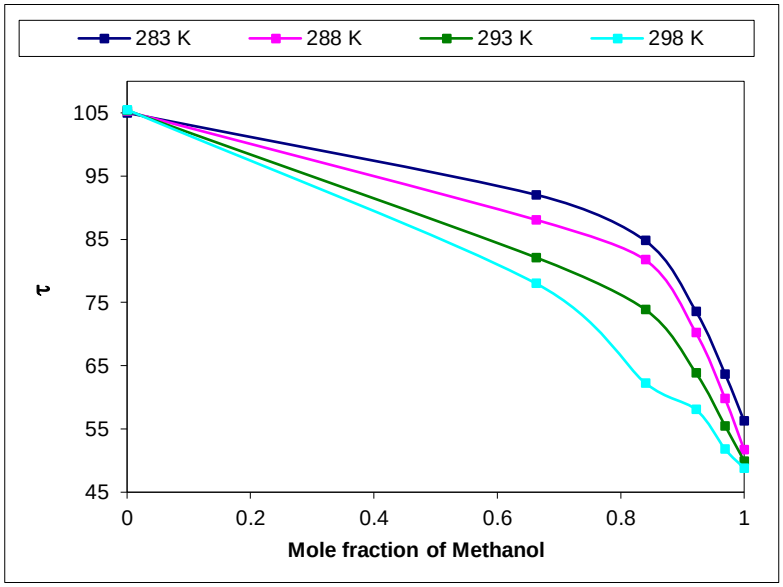


Figure 2: Variation of relaxation time (τ) as a function of mole fraction of Methanol at temperatures 283, 288, 293 and 298K.

4.2 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 Methanol at different temperatures is shown in fig (3) and (4)

The variation of excess permittivity (ε_0^E) and excess inverse relaxation time $(1/\tau)^E$ with the mole fraction of Methanol with Atarax at different temperature is shown in figs. 3 and 4. The excess permittivity, values are positive for all concentrations of Ethanol in Atarax at all temperature. Except at 283K for 20% of Methanol. This indicates parallel alignment of dipole in the system and formation of monomer, which increases total number of dipoles.

From figure (3) it can be seen that $(\varepsilon_s)^E$ is positive for all concentration of Methanol in the mixture for all temperature studied. This indicates that the molecules of mixture may form multimers structures in such a way that the effective dipoles get reduced. This is due to the opposite alignment (antiparallel) of the dipoles in the mixture.

The behavior in $(1/\tau)^E$ is quite different as can be seen from figure (4) the all values of $(1/\tau)^E$ are positive, but for lower concentration of Methanol increases and then decreases at higher concentration of Methanol at all temperatures. This suggests that at lower concentration of Methanol the molecular interaction produces hindering field making effective dipole rotation slower. But at higher concentration of Methanol the molecular interaction produces a cooperative field and the effective dipoles have more freedom of rotation.

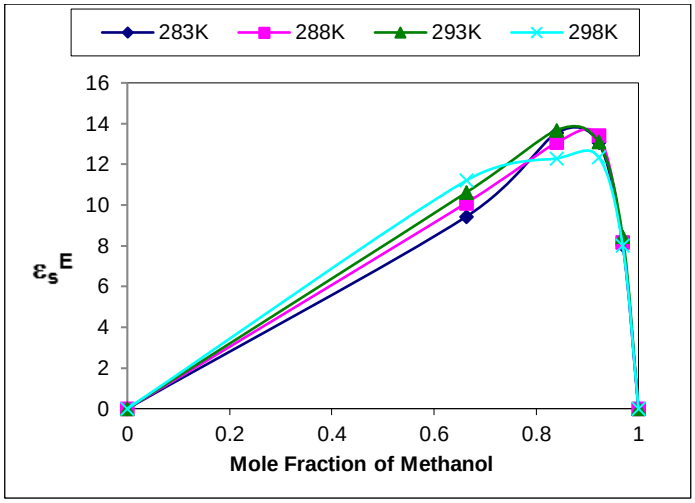


Figure 3: Variation of excess permittivity (ε_s^E) as a function of mole fraction (x_2) of Methanol at temperatures 283, 288, 293 and 298K.

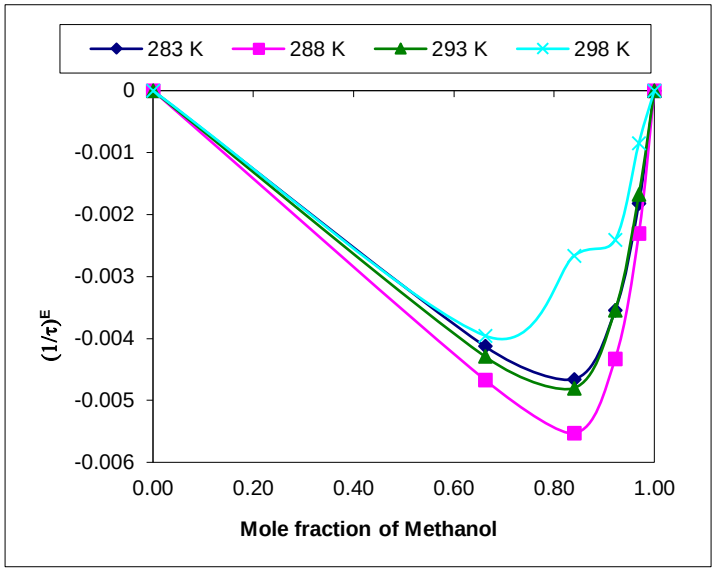


Figure 4: Variation of excess inverse relaxation time $(1/\tau)^{E}$, as a function of mole fraction (x_2) of Methanol at temperatures 283, 288, 293 and 298K.

4.3 The Bruggeman Factor

The experimental values together with ideal and theoretical values of Bruggeman factor plotted against volume fraction of Methanol in the mixture are as shown in figure (5). It can be seen from this plot that f_B shows a deviation from the ideal Bruggeman behavior. This confirms the intermolecular interaction in the mixture.

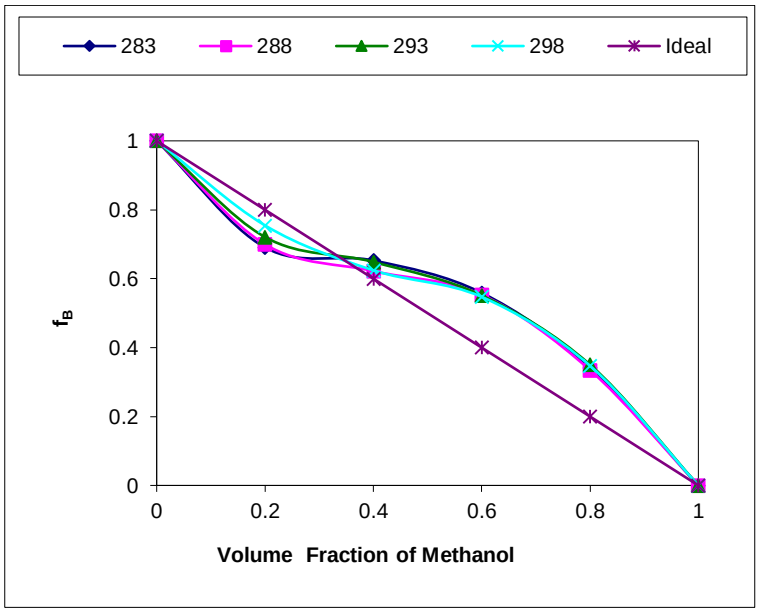


Figure 5: The Bruggeman plot for Atarax + Methanol as a function of volume fraction of Methanol at temperatures 283, 288, 293 and 298K.

4.4 The Kirkwood correlation factor

The structural information about the liquids from the dielectric relaxation parameter may be obtained using the Kirkwood correlation factor g_f. 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 2 and shown in fig. (6).

The variation in g_f with change in volume fraction of Methanol are given in table 2 and shown in fig (7). The amount of solute – solvent interaction can be accessed using these parameters.

The g^{eff} values confirm the formation of hydrogen bonding in pure Atarax system. These values are greater than unity at all temperatures suggesting parallel orientation of electric dipoles. The corresponding values for Methanol indicate weak dipole-dipole interaction. This results the formation of antiparallel arrangement of dipoles in the pure system of Methanol at 80% and 100% . From table 2 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.

The g^{eff} values can be observed from fig.6 are greater than unity for this binary mixture at all temperature, suggesting parallel orientation of electric dipole.

The gf values can be observed from fig. 7 are closure to unity for this binary mixture at all temperature, suggesting stronger interaction between the molecules.

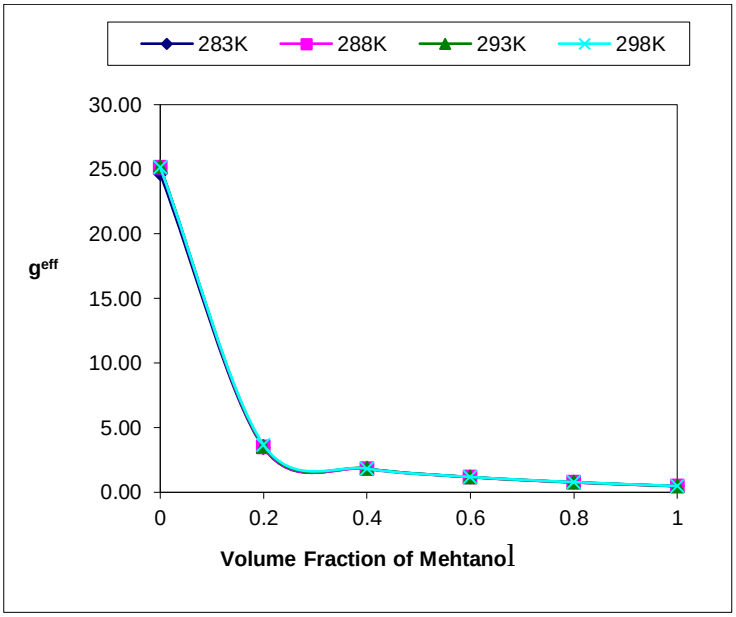
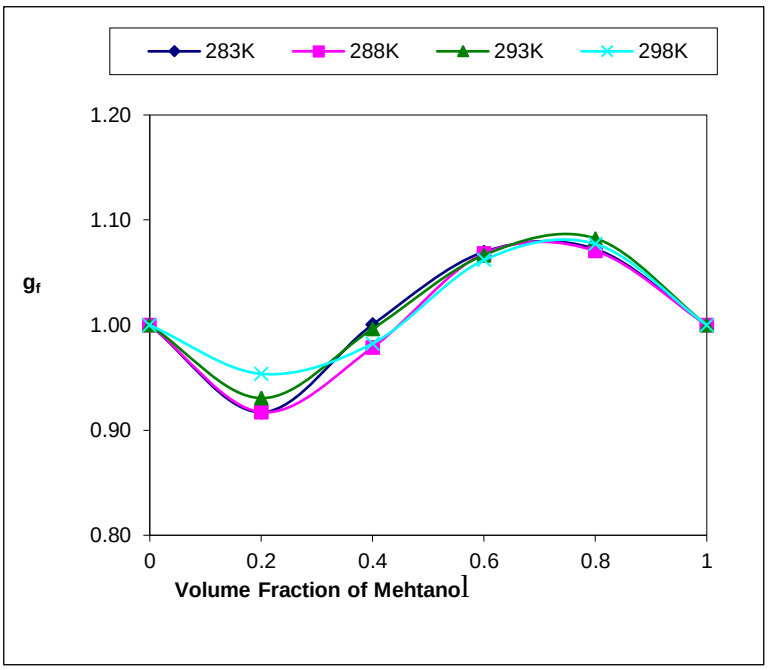


Figure 6: Variation of Kirkwood correlation factor g^{eff} with variation of volume fraction of Methanol in Atarax at temperatures 283, 288, 293 and 298K.



253

254 **Figure 7:** Variation of Kirkwood correlation factor g_f with variation of volume fraction of Methanol
255 in Atarax at temperatures 283, 288, 293 and 298K.

256 **4.5 Thermodynamic parameters**

257 The values of molar enthalpy of activation (ΔH) and molar entropy of activation (ΔS) at different
258 concentrations determined using Eyring rate equation are listed in table (3). The variation of molar
259 enthalpy of activation and molar entropy of activation with increase in volume fraction of Methanol
260 in the mixture are shown in fig (8). The Arrhenius plot for Atarax + Methanol system is shown in fig
261 (9). From table (3) it can be seen that the molar enthalpy of activation (ΔH) increases with increase in
262 volume fraction of Methanol in Atarax from -2.64 KJ/mol up to 18.46 KJ/mol. This means that more
263 energy is needed for group dipole reorientation with increase in volume fraction of Methanol in the
264 mixture. Negative value of molar entropy of activation (ΔS) with volume fraction of Methanol
265 indicates relatively high ordered arrangement of molecules in the activated state (Hasted J. B. 1973,
266 S. N. Helembe et al. 44. 1995, S. N. Helembe et al. 45. 1995, M. P. Lokhande et al. 1997).
267 The positive values of enthalpy with increasing concentration of Methanol suggest less energy is
268 required to achieve group dipole reorientation.

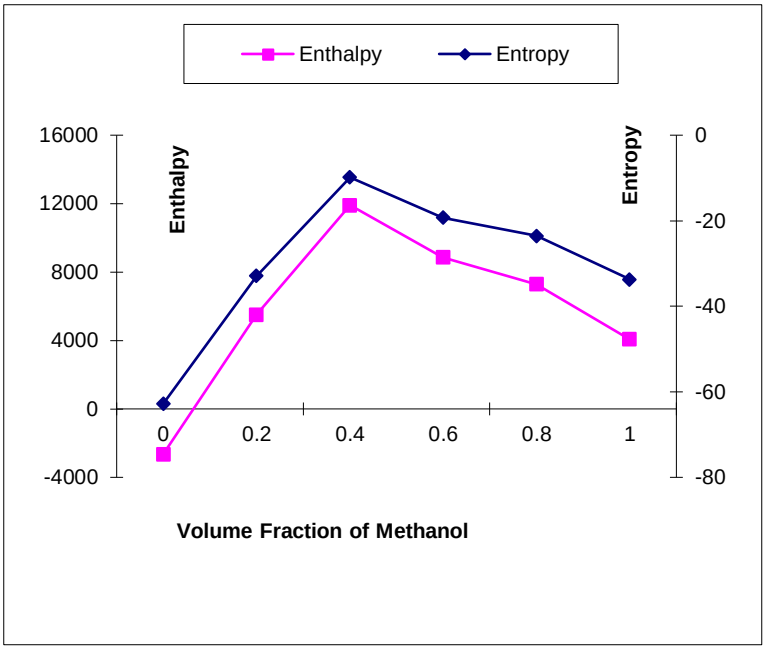


Figure 8: Enthalpy (ΔH) and Entropy (ΔS) of Atarax + Methanol Binary mixture.

). The Fig (9) shows that the plot with the steeper slope has a higher activation energy and the plot with the flatter slope has a smaller activation energy. This means that over the same temperature range, a reaction with a higher activation energy changes more rapidly than a reaction with a lower activation energy. The slope of Arrhenius plot changes with concentration, which shows the change in activation energy of the system (J. G. Berberain et al. 1986, S. M. Puranik et al. 1993The temperature dependence of relaxation time follows Arrhenius behavior. The temperature dependence of relaxation time follows Arrhenius behavior (S. B. Sayyad 2008).

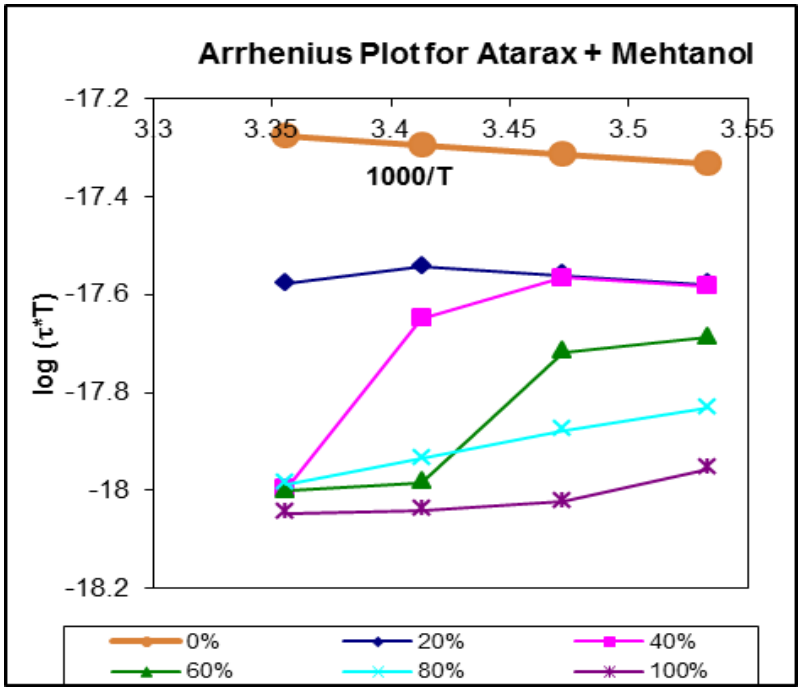


Figure 9: Arrehenius Plot of Atarax + Methanol Binary mixture.

4.6 Conclusion

The static permittivity and relaxation time both decreases with increasing concentration of Methanol, indicates molecules rotate easily, which leads to decrease in relaxation time.
The excess permittivity (ε_0^E) values are positive and more deviation in Methanol rich region shows strong monomeric structure form in this region. The values of excess inverse relaxation time $(1/\tau)^E$ shows effective dipole rotate slowly. The Bruggeman factor f_B shows a small deviation to lower side from the ideal Bruggeman behavior at 20% of volume fraction of Methanol, indicate reduction of effective volume value of Bruggeman parameter get larger than one. This confirms the weak intermolecular interaction in the mixture in this region and in remaining region strong interaction. The g^{eff} values in the Methanol dominate region confirm antiparallel orientation of electric dipoles. The values of g_f deviates from unity indicate interaction between two components of mixture. The molar enthalpy of activation represents need of energy is nonlinear and entropy also nonlinear. Arrhenius shows change in activation energy of the system. The results obtained are used to interpret the nature and kind of solute-solvent interaction.

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Table: 1.: Temperature dependent dielectric parameters for binary mixture of Atarax + Methanol.

Mole Fraction of Methanol	283 K		288 K		293 K		298 K	
	εs	τ (ps)	εs	τ (ps)	εs	τ (ps)	εs	τ (ps)
0	63.4	105	63.55	105.2	62.58	105.4	61.44	105.5
0.6626	52.86	82.05	52.76	82.09	52.72	82.13	53.21	78.02

0.8396	51.64	81.78	50.15	81.75	50.32	73.9	49.11	51.28
0.9218	48.69	73.62	47.9	70.22	47.2	63.87	46.77	51.08
0.9691	42.25	63.67	41.16	59.77	41.1	55.48	41.02	51.81
1	33.27	56.25	32.03	51.7	31.69	49.94	32.12	48.81

368

369 **Table 2:** Kirkwood Correlation factor (g^{eff}) and (g_f) for Atarax + Methanol

Volume fraction of Methanol	**283K**		**288K**		**293K**		**298K**	
	g^{eff}	**g_f**	**g^{eff}**	**g_f**	**g^{eff}**	**g_f**	**g^{eff}**	**g_f**
0	24.63	1	25.13	1	25.17	1	25.13	1
0.2	3.46	0.92	3.51	0.92	3.57	0.93	3.66	0.95
0.4	1.83	1.00	1.81	0.98	1.85	1.00	1.83	0.98
0.6	1.18	1.07	1.18	1.07	1.18	1.07	1.19	1.06
0.8	0.77	1.07	0.76	1.07	0.78	1.08	0.79	1.08
1	0.48	1	0.47	1	0.48	1	0.49	1

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371 **Table: 3.** Activation Enthalpy (ΔH) and Entropy (ΔS) of Atarax + Methanol binary mixture for
372 various concentrations.

Volume fraction of Methanol	**ΔH (KJ/mole)**	**ΔS (KJ/mole)**
0	-2.636	-0.062
0.2	5.520	-0.032
0.4	11.909	-0.098
0.6	8.864	-0.019
0.8	7.298	-0.023
1	4.069	-0.033

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