

Dielectric Relaxation And Molecular Interaction Studies Of PEG With Non-Polar Solvents Using TDR Technique

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Abstract: The complex dielectric permittivity measurements of Poly Ethylene Glycol (PEG) $HO[CH_2-CH_2-O]_n H$ of molecular weight 400 ($n=9$) $g\ mol^{-1}$ with carbon tetra chloride (CCl_4), benzene and 1,4-dioxane mixtures has been carried out at different concentration in mol/lit at 25°C and in the frequency range of 10 MHz to 30 GHz using Time Domain Reflectometry (TDR). The least squares fit method has been used to obtain the static dielectric constant, relaxation time, and Bruggeman factor for binary mixtures. The Kirkwood–Frohlich theory is applied to compute the dielectric constant for the mixtures. It adequately reproduces the experimental values of static dielectric constants for the binary mixtures. The excess parameters confirm that the heteromolecular hydrogen bonding interaction between alcohol-alcohol and alcohol- CCl_4 , benzene and 1,4-dioxane pairs very significantly in the mixtures. The Bruggeman model for the nonlinear case has been fitted to the experimental dielectric data for the mixtures.

Keywords: dielectric relaxation, time domain reflectometry, ethylene glycol.

I. Introduction

The dielectric properties of a medium is measured as a function of frequency in Dielectric spectroscopy (DS). It is based on the interaction of an external field with the electric dipole moment of the sample, often expressed by permittivity. The Dielectric Spectroscopy is especially sensitive to interference polarization and intermolecular interactions. The properties between individual constituents of complex material, characteristic of bulk properties and dielectric relaxation studies of various surfactants in aqueous solutions studied using TDR [1-3].

In low frequency, the measurement of conductive systems was strongly limited due to electrode polarization. To determine various relaxation parameters on dielectric spectrum over a wide frequency range, one step pulse (Time window) method is used. The successful development of Time Domain Spectroscopy (TDS) method and [4-8] Broad Band Dielectric Spectroscopy (BDS) have radically changed the attitude towards DS making it an effective tool for investigation of solids and liquids on the macroscopic, microscopic and mesoscopic levels.

Dielectric relaxation studies are very useful in understanding the structure of polymers. The properties of chemically homologous series of polymer molecule are of special interest because changes in properties are related to chain length and temperature and very useful to study the structural properties of polymers. Poly Ethylene Glycol (PEG) is of great significant in industry, in biomedicine, cosmetics, food additives and plasticizer. The general chemical formula of PEG is $HO(CH_2CH_2O)_nH$, where n is the number of repeat units. The dielectric relaxation study with a variation in chain length is impartment in binary system to study the effect of molecular dynamics and shape of the dielectric relaxation curve in the room temperature. Low molecular weight polymers have good solubility in polar and non polar solvents. As oxygen atoms and OH groups are present in the molecular chain and at the ends of the chain due to which these molecules have several hydrogen bonding giving rise to several confirmations in non polar mixtures. Extensive experimental attempts [9] have been made to study the absorption behavior of PEG molecules and their oligomers at radio waves and microwaves to understand their dielectric behavior. Earlier, the dielectric and thermodynamic properties of Polyethylene glycols have been studied to know the effect of chain length and the variation of temperature on the configuration of PEG by knowing the configuration of Polyethylene oxide (PEO) chain by measurements of NMR spectra [10-12], IR spectra [13,14], X-ray diffraction studies [15], the dipole moments [16], dielectric loss [17-19] and by theoretical determination of energies [16,20] associated with rotational isomeric state about skeletal bonds of the molecules of PEO, its oligomers and polymers.

The dielectric constant is one of important physicochemical properties of the mixed solvents, which enhance most of the biological, pharmaceutical, chemical, physical, analytical, laboratory industrial application [21]. The dielectric study of PEG on addition of dioxane provides information about breaking of molecular structure in the systems. In binary mixtures of PEG with benzene and CCl₄ the hydroxyl groups are present on the terminal carbon atom and neighboring carbon atom, therefore the strong intermolecular interaction, possible through hydrogen bonds, results in specific dynamics properties compared to PEG with 1,4- dioxane. Dielectric characterization has great potential in studying the H-bond interactions: dipolar alignments, hydrogen bond connectivity, and stoichiometric ratio of stable adduct formation in mixed solvents [21-23]. Crossely et al. [24] have examined the dielectric permittivity and loss for a series of glycols. It is observed that the relaxation times for the glycols are almost independent of alkyl chain length. The study of dielectric relaxation in glycol mixtures, a correlation between the PEG and non-polar solvents obtained from the values of the relaxation time and the number density of molecules. This can be interpreted as an indication that only one hydroxyl group in the PEG is active in the main relaxation process. This implies that the other end of the PEG, the ethylene molecule remains “anchored” to another molecule by the hydrogen bond. This reduces the flexibility at the molecular level leading to a slower relaxation than in normal glycols.

Main purpose of this study is to understand the dielectric behavior of PEG in terms of hydrogen bond and modification of hydrogen bonding due to presence of other non-polar molecules. In the present work dielectric measurements of the mixtures of PEG with benzene, CCl₄ and 1, 4-dioxane solvents using TDR method are reported. The complex dielectric permittivity in the frequency range of 10 MHz to 30 GHz has been determined. The static dielectric constant, dielectric loss, Bruggeman factor, Kirkwood correlation factors, excess dielectric permittivity, relaxation time have been determined for this binary mixtures at 25°C.

II. Experimental

2.1. Materials

The poly(ethylene glycols) of number average molecular weight 400 g/ mol is obtained from Sigma Aldrich India. Benzene, CCl₄ and 1,4 Dioxane were obtained from Merck India Pvt. Ltd., and were used to prepare combination of binary mixtures without further purification. The dielectric permittivity ϵ' and dielectric loss ϵ'' of pure liquid PEG's binary mixtures at emperatures 25°C were measured using TDR employed in the frequency region 10 MHz to 30 GHz. The detailed analysis and procedures of the TDR system are explained below. The mole fractions of the mixture constituents were determined by using their individual volume in the mixture, the room temperature density and the molecular weight.

2.2. Measurements and Data Analysis

The complex permittivity spectra were studied using Time Domain Reflectometry (TDR) method [25, 26]. The basic TDR setup consists of broad band sampling oscilloscope, TDR module and coaxial transmission line. The Tektronix DSA8200 sampling oscilloscope with 30 GHz bandwidth and TDR module 80E08 with step generator unit was used. A 200 mV step pulse with 18 ps incident pulse and 20 ps reflected pulse time and 200 KHz repetition rate passes through coaxial 50 Ω lines. All measurements are carried out in open load condition. Sampling oscilloscope monitors changes in step pulse after reflection from sample. 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. Temperature of the sample was controlled electronically within $\pm 1^\circ\text{C}$.

The step pulses recorded without sample $R_1(t)$ and with sample $R_x(t)$ were subtracted and added to get

$$p(t) = [R_1(t) - R_x(t)] \quad (1)$$

$$q(t) = [R_1(t) + R_x(t)] \quad (2)$$

The complex reflection coefficient $\rho^*(\omega)$ over a frequency range of 10 MHz to 30 GHz were determined as follows:

$$\rho^*(\omega) = \frac{cp(\omega)}{jwdq(\omega)} \quad (3)$$

where $p(\omega)$ and $q(\omega)$ are Fourier transforms of $p(t)$ and $q(t)$ obtained using summation and Samulon methods [27] respectively, c is the velocity of light, ω is angular frequency and d is effective pin length (0.09 mm).

$$\epsilon^*(\omega) = \epsilon_\infty + \left[\frac{(\epsilon_0 - \epsilon_\infty)}{[1 + (J\omega J_0)^{1-\alpha}]^\beta} \right] \quad (4)$$

The complex permittivity spectra $\epsilon^*(\omega)$ is obtained from reflection coefficient spectra $\rho^*(\omega)$ by applying the non – linear least square fit method suggested by Cole [25, 26]. The frequency dependent complex permittivity spectra $\epsilon^*(\omega)$ of PEG-400 with their respective binary mixtures of benzene, CCl₄, and 1,4, Dioxane for different concentrations in liquid state at temperature 25°C are shown as real part (ϵ') and imaginary part (ϵ'') . It has been found that ϵ_0 , ϵ' , ϵ'' and ϵ_∞ are the linear functions of the concentration of binary mixture solute in dilute concentrations used for analysis of dielectric relaxation data and the values are recorded in Table.1. It is also found that the variations in relaxation time τ , g_f and g^{eff} values in binary mixtures are also calculated in various non-polar solvents. The Kirkwood correlation factor provides useful information regarding the orientation of dipoles. The dielectric constant can be used to calculate Kirkwood correlation factor g defined in general by

$$g = 1 + z \cos \gamma \tag{5}$$

where z is the average number of nearest neighbors and γ is the angle between the dipole moments of the neighboring molecule, Kirkwood correlation factor is greater than 1 are indicative of a tendency towards parallel alignment of dipoles, values less than 1 are obtained when anti parallel alignment is favored and a value unity is expected when orientational ordering is absent.

III. Results And Discussion

The complex permittivity spectra (real (ϵ') and imaginary (ϵ'')) as a function of log frequency (GHz) at 25°C for Poly (ethylene glycol) with benzene, CCl₄ and 1,4,dioxane for various concentrations are shown in Fig.(1a - 3b), and the Variation of excess permittivity (ϵ_∞), dielectric permittivity(ϵ'), static permittivity (ϵ_0),relaxation time (τ_{ps}) and dielectric loss (ϵ'') verses concentration in mol/lit at 25 °C for Poly (ethylene glycol) having molecular weight 400gmol⁻¹ at 25 °C with beneze,CCl₄, 1, 4, dioxane are shown in Fig.(4a - 4e) respectively. Both static dielectric constant and relaxation time increases with increase in concentration. The frequency dependence of the complex dielectric permittivity and the excess properties are reported in Table.1.

The Kirkwood correlation factor g [28] provides the information regarding the orientation of the electric dipoles. For the pure liquid polymer, the Kirkwood correlation factor g may be obtained by the expression

$$g\mu^2 = \frac{9kTM}{4\pi\pi N} \left[\frac{(\epsilon_0 - \epsilon_\infty)(2\epsilon_0 + \epsilon_\infty)}{\epsilon_0(2 + \epsilon_\infty)^2} \right] \tag{6}$$

where, μ is the dipole moment, ρ is density at temperature T , M is the molecular weight, k is Boltzmann constant, N is Avogadro's number, ϵ_0 is static permittivity and ϵ_∞ is the dielectric permittivity at high frequency, often represented by the square of the refractive index ($\epsilon_\infty = n_D^2$). In associating molecules, the molecular relaxation time is affected by the intra and intermolecular hydrogen bonding. The departure of the Kirkwood correlation factor g from unity is a measure of the extent of intermolecular hydrogen bonding. For the molecules with parallel dipole moments g will be greater than unity, while for anti-parallel dipole alignment g will be smaller than unity. To examine the effect of intermolecular association on the dielectric relaxation time of these molecules, the values of Kirkwood correlation factor g have been determined.

We have assumed that the mixture can be represented by one correlation factor g^{eff} as follows:

$$\frac{(\epsilon_{0i} - \epsilon_{\infty i})(2\epsilon_{0i} + \epsilon_{\infty i})}{9\epsilon_{0i}} = \frac{4\pi\pi}{9kT} \left[\frac{\mu^2 \rho_1}{M_1} X_1 + \frac{\mu^2 \rho_2}{M_2} (1 - X_1) \right] g^{eff} \tag{7}$$

where 1 and 2 stand for PEG and non-polar solvent, X_1 is the volume fraction of PEG. The value of $g^{eff} > 1$ indicates average parallel orientation of electric dipole in a molecule. It can be seen that as PEG is added in non-polar solvents at regular steps in the mixture the value of g^{eff} increases is shown in Fig.6. We also assume that correlation factors of PEG and non-polar solvent molecules are affected by the same amount g_f in the mixture [29]. The Kirkwood–Frohlich equation for the mixture can be written as

$$\frac{(\epsilon_{0i} - \epsilon_{\infty i})(2\epsilon_{0i} + \epsilon_{\infty i})}{9\epsilon_{0i}} = \frac{4\pi\pi}{9kT} \left[\frac{g_1 \mu^2 \rho_1}{M_1} X_1 + \frac{g_2 \mu^2 \rho_2}{M_2} (1 - X_1) \right] g_f \tag{8}$$

The Kirkwood correlation factors g_1 , g_2 for individual species are modified by assuming for PEG – non-polar solvents mixture that two kinds of intermolecular hydrogenbonds exist. One is the hydrogen bond between the PEG - PEG molecules; another is the hydrogen bond between the PEG – non-polar solvent molecules. These new correlations (g_1 and g_2) are described by the relation as follows [30]:

$$g_1 = 1 + Z_{11} \cos \gamma_{11} + Z_{12} \cos \gamma_{12} \frac{\mu_2}{\mu_1} \quad (9)$$

$$g_2 = 1 + Z_{21} \cos \gamma_{21} \frac{\mu_1}{\mu_2} \quad (10)$$

where Z_{11} , Z_{12} and Z_{21} are the average number of particles forming the hydrogen bond with PEG–PEG, PEG–non-polar solvents, and non-polar solvents–PEG pairs, respectively. γ_{11} and γ_{21} are the angles between the neighbouring dipoles of PEG and non-polar solvents molecules. The values of g_1 and g_2 depend on the concentration of non-polar solvents in binary mixtures. The values of g^{eff} and g_f for PEG–non-polar solvents mixture are reported in Table 2. The observation shows that the g values are greater than unity in all these binary systems, which leads to the conclusion that the molecules associate to form multimers with parallel dipole moments. Dielectric measurements in the megahertz to gigahertz range are very important for investigating molecular dynamics of polymers, especially in solution state. One empirical relation, the Kohlrausch-Williams-Watt (KWW) function, has considerable use not only for the dielectric relaxation behavior of liquid polymers but also for the other polymer relaxation phenomena including volume and enthalpy relaxations, NMR and dynamical relaxations, Quasi elastic light scattering, small angled relaxation scattering, transient optical relaxation and time dependent fluorescence depolarization. Dielectric behavior of polymer at a frequency region higher than 100 MHz has not been examined satisfactorily. In case of liquid polymers the dielectric measurements in the higher frequency studies are very scarce. In general, polar polymers in dilute solutions of non-polar solvent show an absorption peak at a frequency between 100 MHz and 2 GHz, if they have dipolar component perpendicular to their chain contour [31]. Frequency obtained from TDR measurements peak does not depend on the molecular weight of the polymer chains [32]. Poly ethylene glycol shows the dielectric relaxation process in polar and non-polar solutions in the gigahertz region [33, 34]. The static permittivity of mixture with volume fraction of solute is given by Bruggeman mixture formula [35]

$$f_B = \frac{\epsilon_{0m} - \epsilon_{02}}{\epsilon_{01} - \epsilon_{02}} \left(\frac{\epsilon_{01}}{\epsilon_{0m}} \right)^{1/3} \quad (11)$$

It can be seen from Fig.(5a - 5c) that f_B is not a linear function of PEG volume fraction as predicted by Bruggeman equation. The Bruggeman equation may be modified for binary liquid [36] as follows;

$$f_B = \frac{\epsilon_{0m} - \epsilon_{02}}{\epsilon_{01} - \epsilon_{02}} \left(\frac{\epsilon_{01}}{\epsilon_{0m}} \right)^{1/3} \quad (12)$$

$$= 1 - [a - (a-1)X_A]X_A \quad (13)$$

where f_B is the Bruggeman dielectric factor. The ϵ_{0m} , ϵ_{01} , and ϵ_{02} are the static dielectric constant corresponding to mixtures, PEG with benzene, CCl_4 and 1,4-dioxane respectively, X_A is the volume fraction of PEG. From the above equation, a linear relation is expected from a plot of f_B versus X_A . In this equation, volume fraction X_A is changed by a factor $a-(a-1)X_A$ of the mixture; for $a = 1$ it corresponds to Bruggeman equation. The value of a can be determined by least squares fit method and is found to be 1.29, 1.16 and 1.65 for the binary systems respectively, its deviation from unity indicates the molecular interaction in the mixture.

IV. Conclusion

The dielectric relaxation parameter for PEG-400 with their respective binary mixtures of benzene, CCl_4 , and 1,4, dioxane for different concentrations in liquid state has been measured using TDR method in the frequency range 10 MHz to 30 GHz at 25°C. The dielectric constant for the mixtures can be explained using hydrogen-bonded model by assuming the formation of hydrogen bonds between solute-solute and solute-solvent pairs. The orientation correlations between neighbouring molecules due to hydrogen bonding interaction are determined in terms of Kirkwood factors. A modification in the Bruggeman equation provides a better description of dielectric behavior in the mixtures. The study shows the influence of varying chain length on the molecular size, shape, dielectric relaxation and structural properties of the binary system. At the relaxation frequency the oscillation of the applied field becomes very fast for the molecules to have time to fully rotate. As compared to relaxation time of pure liquid, the relaxation time of binary mixtures is higher which suggest that the formation of complexes or presence of association in unlike molecules, which gives an idea about the behavior of dielectric constant and relaxation time with respect to mixture compositions. The g_f and g^{eff} values are found to be greater than unity shows the molecules are with parallel dipole moment and confirm that in these

binary systems the dipolar orientation has an activated state and more ordered. It also suggests that there is more hindrance to the rotation of the molecule clusters in binary mixture in comparison with the rotation of their homogeneous clusters. This also supports the conclusion drawn from the positive values of relaxation time. The proper choices of reference liquids for calibration are very useful to keep the errors in the values of these dielectric parameters within the desired limit.

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Table.1 Dielectric parameter of PEG binary system of various concentration at T=25⁰C

	mol/lit	ϵ'	ϵ''	ϵ''/ϵ'	$\epsilon''/\epsilon' \text{ps}^{-1}$	ϵ''/ϵ'
	0.1	2.01	2.13	2.12	14.84	0.008
PEG -	0.3	2.05	2.28	2.34	16.31	0.009
BENZENE	0.5	2.08	2.41	2.54	18.78	0.183
	0.7	2.18	2.48	2.70	21.21	0.216
	0.9	2.19	2.57	2.86	22.45	0.278
	0.1	1.96	2.21	2.23	14.55	0.043
PEG - CCl ₄	0.3	2.03	2.29	2.50	15.1	0.065
	0.5	2.05	2.37	2.53	17.65	0.154
	0.7	2.12	2.45	2.67	19.12	0.191
	0.9	2.14	2.53	2.90	21.2	0.262
	0.1	2.01	2.29	2.31	10.34	0.203
PEG -	0.3	2.11	2.44	2.49	10.72	0.290
DIOXANE	0.5	2.28	2.57	2.65	10.82	0.350
	0.7	2.37	2.66	2.78	11.31	0.433
	0.9	2.44	2.76	2.88	11.97	0.528

Table.2 Effective correlation factor and correlation factor of PEG with various volume fractions

	vol. fr. solute	g^{eff}	g_f
	0.038	1.67	1.00
PEG-BENZENE	0.099	2.36	1.22
	0.153	3.33	2.14
	0.200	3.94	2.72
	0.242	4.24	3.19
	0.038	1.39	1.00
PEG - CCl ₄	0.099	3.01	2.48
	0.153	4.36	2.64
	0.200	5.09	3.36
	0.242	5.84	3.92
	0.038	1.19	1.00
PEG -	0.099	1.38	1.07
	0.200	1.67	1.18
DIOXANE	0.153	1.52	1.13
	0.242	1.74	1.21

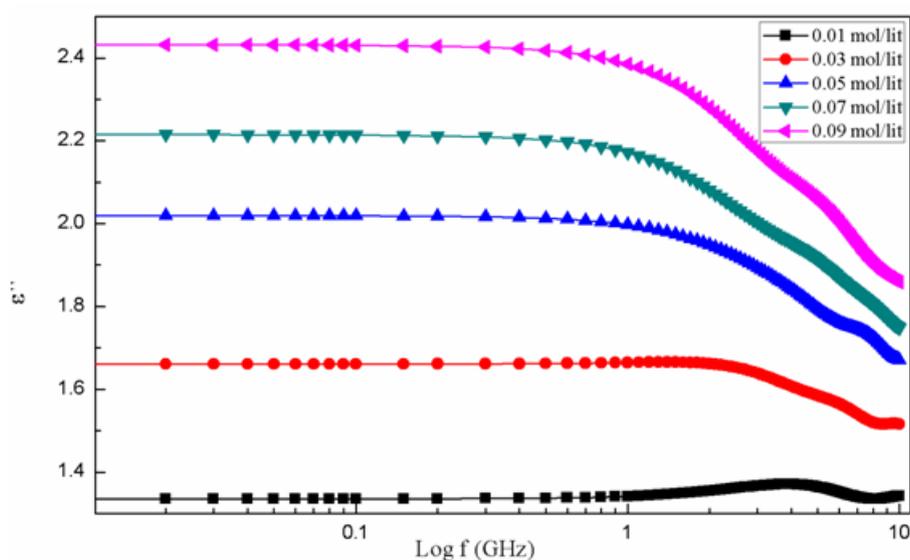


Fig.1a

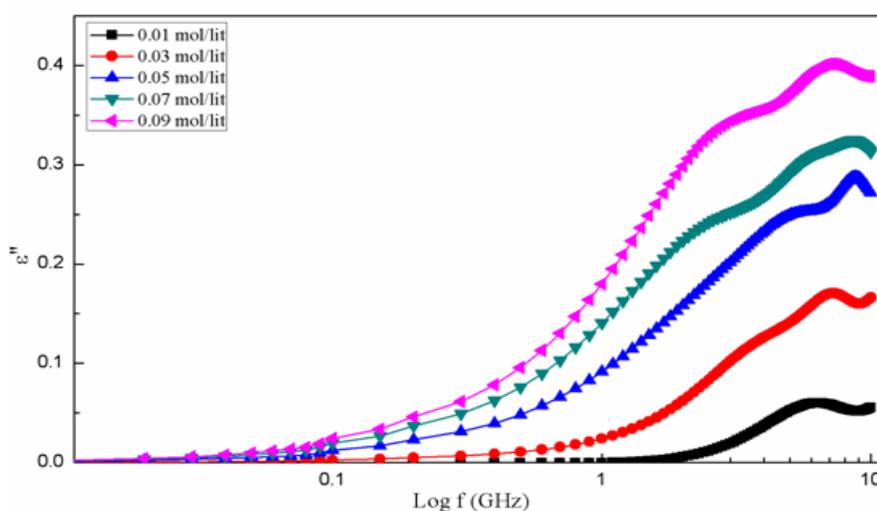


Fig. 1b

Fig. 1a and 1b. Variation of ϵ' and dielectric loss (ϵ'') as a function of log frequency in (GHz) at 25 °C for Poly (ethylene glycol) with benzene for various concentrations.

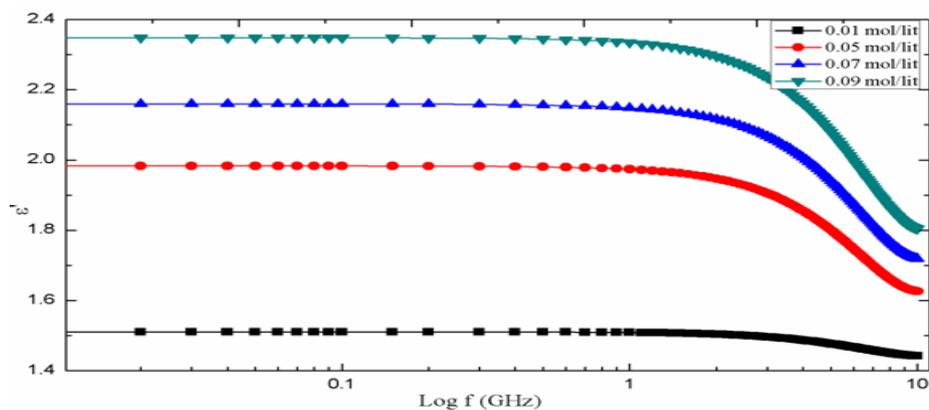


Fig.2a

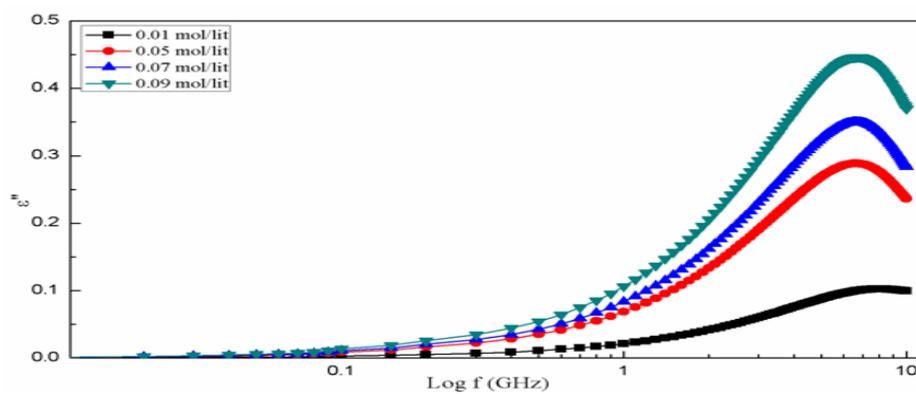


Fig.2b

Fig. 2a and 2b. Variation of dielectric permittivity (ϵ') and dielectric loss (ϵ'') as a function of log frequency in (GHz) at 25 °C for Poly (ethylene glycol) with, CCl_4 for various concentrations.

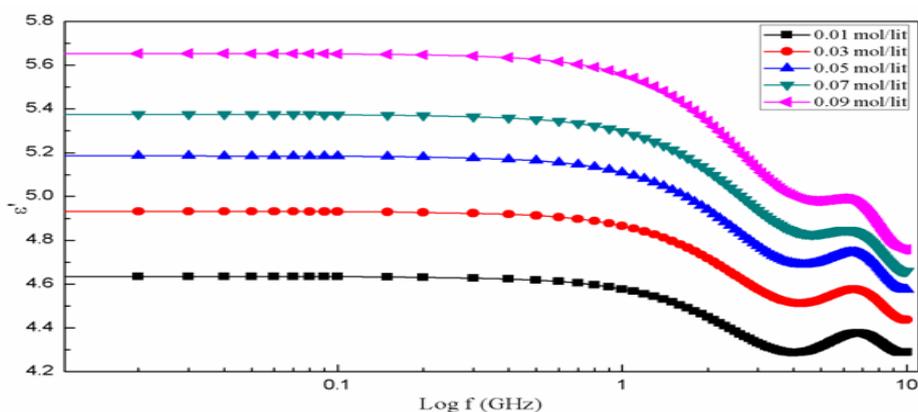


Fig.3a

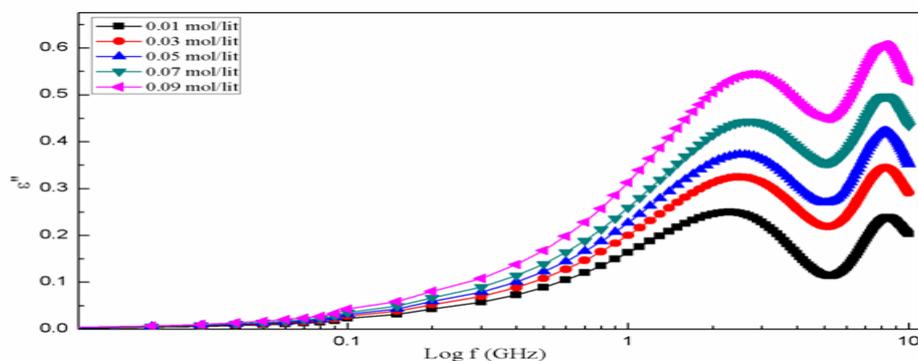


Fig.3b

Fig. 3a and 3b. Variation of dielectric permittivity (ϵ') and dielectric loss (ϵ'') as a function of log frequency in (GHz) at 25 °C for Poly (ethylene glycol) with 1, 4, dioxane for various concentrations.

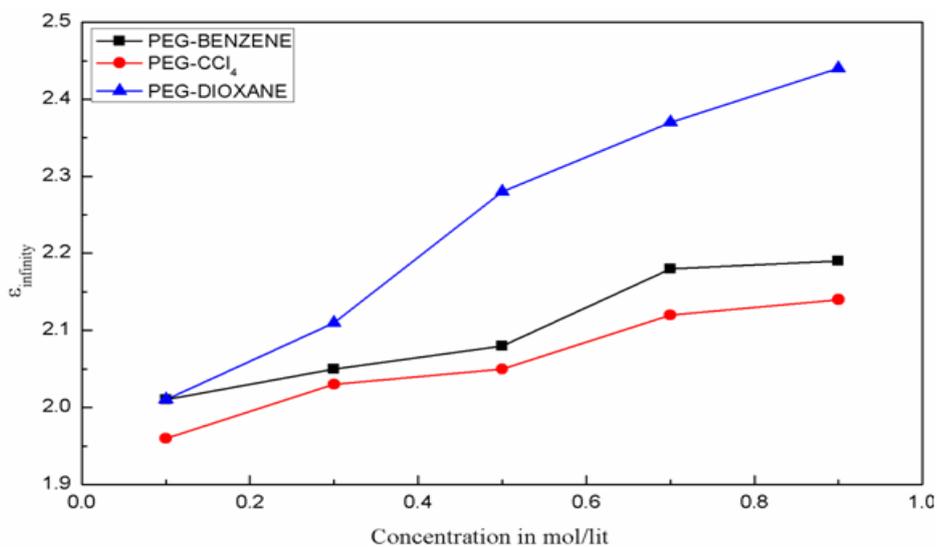


Fig.4a

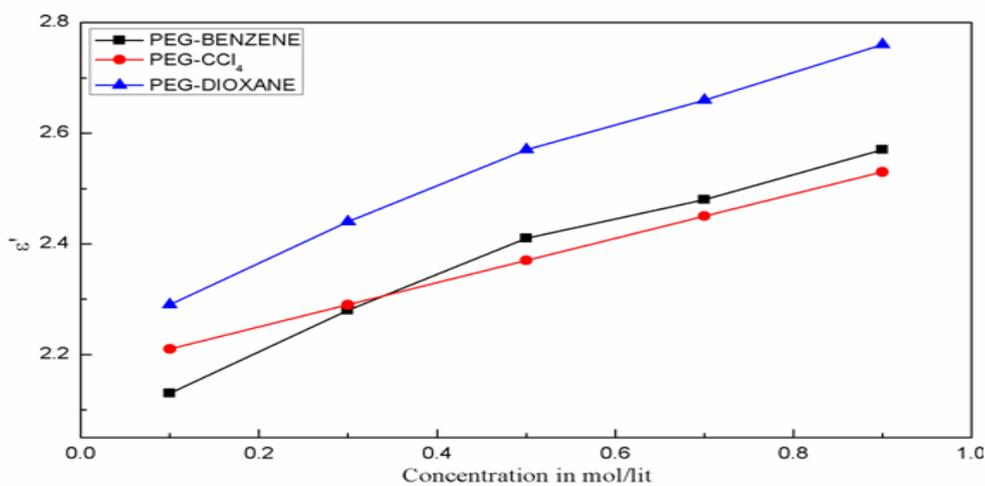


Fig.4b

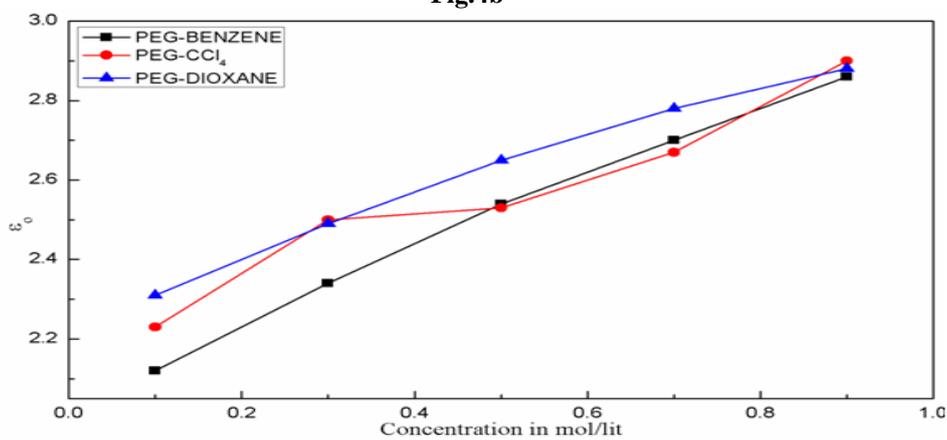


Fig.4c

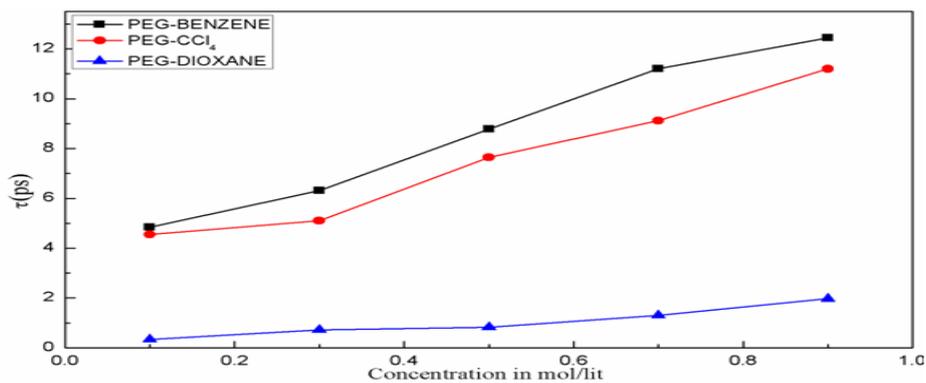


Fig.4d

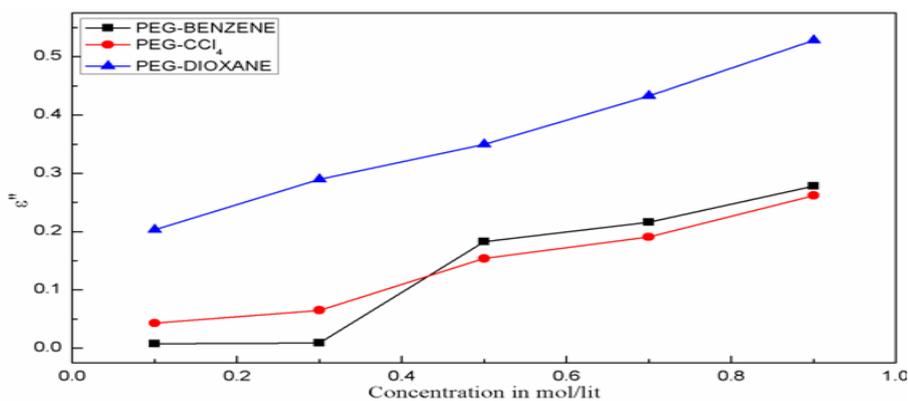


Fig.4e

Fig. 4a , 4b, 4c, 4d and 4e. Variation of permittivity at infinite frequency (ϵ_{∞}), dielectric permittivity(ϵ'), static permittivity (ϵ_0), relaxation time (τ_{ps}) and dielectric loss (ϵ'') verses concentration in mol/lit at 25 °C

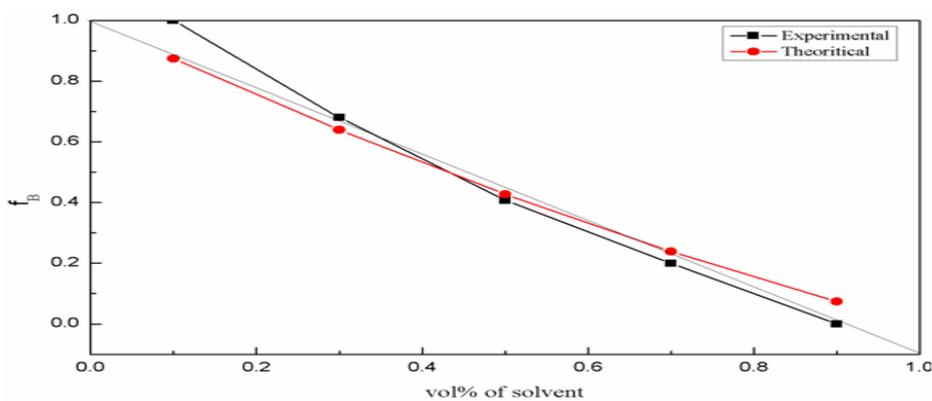


Fig.5a

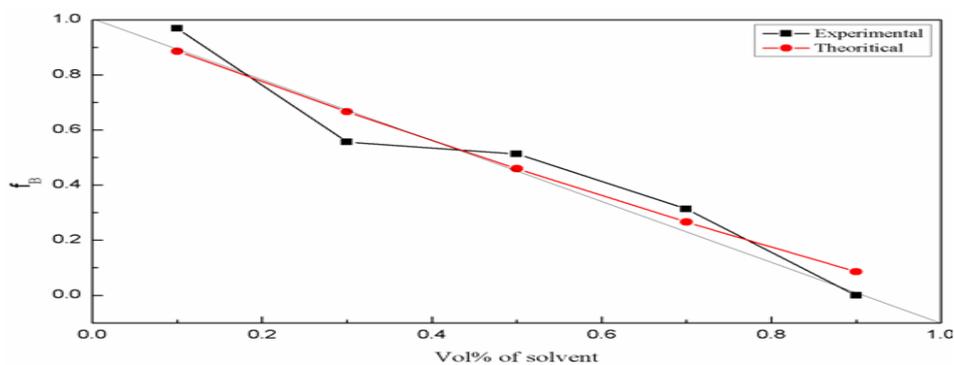


Fig.5b

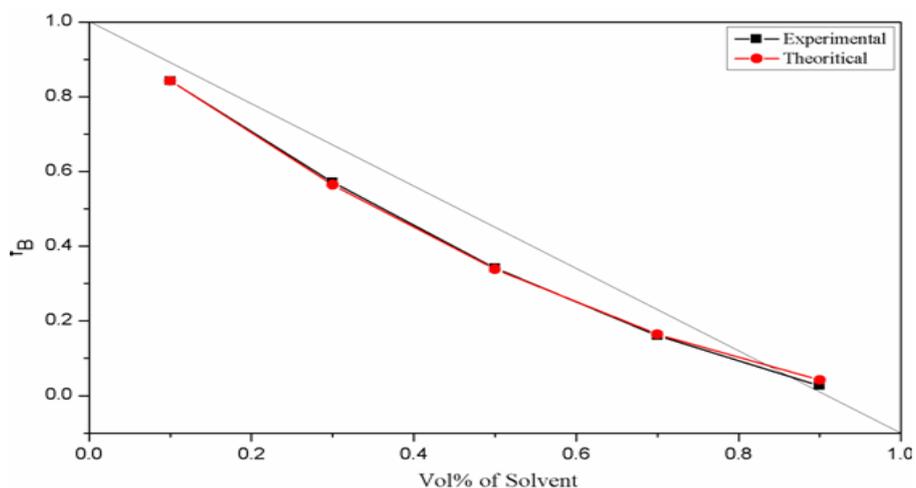


Fig.5c

Fig.5a, 5b and 5c variation of Bruggeman dielectric factor with volume % of PEG with benzene, CCl₄ and 1,4 dioxane

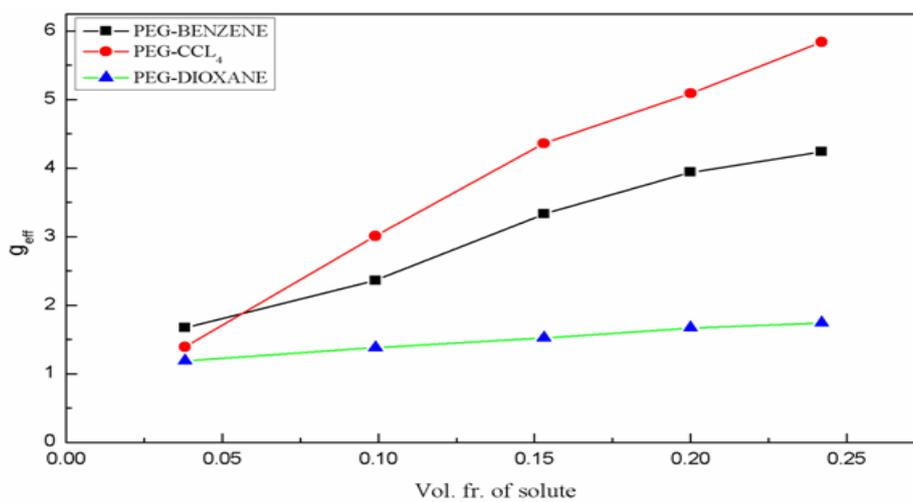


Fig.6

Fig.6 variation of Kirkwood effective correlation factor with volume fraction of PEG with benzene, CCl₄ and 1,4 dioxane