

Dielectric relaxation and molecular interaction investigation of glycolic acid-water mixture using time domain reflectometry

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The complex spectra of glycolic acid (GA) and water mixture have been measured by time domain reflectometry (TDR) in the frequency range 10 MHz to 30 GHz at various temperatures for entire concentration. Dielectric relaxation time (τ), static dielectric constant (ϵ_0) and dielectric permittivity at low frequency (ϵ') and at optical frequency (ϵ'') have been determined from measured complex spectra (ϵ^*) using non-linear square fit method. Conductivity, Kirkwood and effective Kirkwood correlation factor of mixture calculated from the determined dielectric parameters have been used to find the alignment of dipoles between molecules. Thermodynamic parameters enthalpy, entropy and Gibb's free energy have been determined which enable the direction of reaction. Excess permittivity of glycolic acid-water mixture has also been determined which confirms the molecular interaction. Macroscopic parameters such as density and viscosity of mixture have been determined at room temperature. FTIR spectral characterization concedes the solute-solvent interaction.

Keywords: Dielectric parameters, Dielectric relaxation, Excess properties, Thermodynamic parameters, TDR

1 Introduction

Hydrogen bonding plays considerable role in many life processes. Hydrogen bonded molecular interaction in biological structure, importance, key process and applications were discussed earlier¹⁻⁴. Dielectric relaxation is a significant parameter to determine molecular interaction between the molecules^{5,6}. Dielectric relaxation spectroscopy (DRS) is an efficient tool to study the dielectric behaviour of liquid mixture. Glycolic acid (GA) is the simplest carboxylic acid which contains highly polar organic groups. The carbonyl C=O and hydroxyl O-H are the two important H-bonding sites of GA. In addition to this methyl group of the structure possibly produces bonding. Relatively electro negative of oxygen creates permanent dipole moment even it is covalently bonded with carbon.

GA is an important intermediate in the photo respiratory carbon oxidation cycles in plant and algae⁷. The structure of GA chemically potent

has wide range of applications in industries, pharmaceutical and cosmetics. GA predominantly used in chemical peeling of skin⁸. GA is available naturally in sugar beet. The crystal structure of GA was studied by Pijper *et al.*⁹. Blom and Bauder studied the microwave spectrum of GA in the frequency range 48-60 GHz^{10,11}. Godfrey *et al.*¹² studied the structure of GA with jet expansion spectroscopy and AAT conformer. The lowest energy conformers of GA studied theoretically by Flock and Ramek¹³. The microwave spectroscopy and IR spectroscopy study show diversified rotamers and distinctive structural conformations^{14,15}. IR and Raman calculations on these rotamers and conformations with frequency assignment of glycolic acid are given by Mary *et al.*¹⁶. In recent study Gu *et al.* studied the H-bonds in alpha hydroxy carboxylic complexes and also studied interaction of GA with dimer^{17,18}.

2 Experimental Method

2.1 Material and sample preparation

Glycolic acid was obtained from sigma Aldrich, having a purity of 99.9% and used without further

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purification. HPLC water with 1 μ S conductivity was used as a solvent. The GA and water solution was prepared very carefully to obtain homogenous medium. The mixtures were prepared by weighing appropriate amounts of the constituents of in a suitable flask. In order to avoid the uptake of moisture from the air, solution should be kept in tightly packed flask. The solutions were prepared carefully and maintained at room temperature for about 10 to 15 min before conduct of experiment.

2.2 Dielectric measurement and data analysis

The solutions were prepared carefully and maintained at a desired temperature for 10-15 min before taking the measurements. The dielectric permittivity (ϵ'), dielectric loss(ϵ''), static dielectric constant (ϵ_s), relaxation time (τ) and dielectric constant at infinite frequency (ϵ_∞) were measured using time domain reflectometry (TDR) in the frequency range from 10 MHz to 30 GHz. TDR setup consists of source, transmission line and sampling oscilloscope. By using source a step pulse of 200 mv with 18 ps incident and 20 ps reflected pulse time produced. Transmission line consists of 50 Ω semi rigid copper coaxial cable in which inner diameter of outer cable is 2.2 mm and outer diameter of inner cable is 0.51 mm. Sampling model 80E08 in Tektronix serial analyzer has been used which produces accurate oscilloscope measurement in 10 MHz to 30 GHz. Reflected pulse without sample $R_1(t)$ and with sample $R_x(t)$ are recorded in digitized 2000 points time window of 5 ns. The difference, sum of reflected pulse are $p(t)=[R_1(t)-R_x(t)]$ and $q(t)=[R_1(t)+R_x(t)]$, respectively. The frequency transformation of $p(t)$ is $p(\omega)$ and $q(t)$ is $q(\omega)$ where $p(\omega)$, $q(\omega)$ are reflection coefficients¹⁹⁻²⁴.

The complex permittivity spectra $\epsilon^*(\omega)$ was obtained from reflection coefficient by applying a bilinear calibration method^{25,26}:

$$\rho^*(\omega) = \frac{cp(\omega)}{j\omega dq(\omega)} \quad \dots (1)$$

The Havriliak-Negami expression includes the Cole-Cole ($\beta = 1$), Davidson-Cole ($\alpha = 0$) and Debye ($\alpha = 0$, $\beta = 1$) relaxation models. A non-linear least square fitting method was used to determine the values of the dielectric parameters Debye relaxation model²⁷.

$$\epsilon^*(\omega) = \epsilon_\infty + \frac{(\epsilon_s - \epsilon_\infty)}{[1 + (j\omega\tau)^{1-\alpha}]^\beta} \quad \dots (2)$$

with ϵ_s and τ as fitting parameters, α and β are distribution parameters. A nonlinear least-square fit method was used to determine the values of the dielectric parameters.

2.3 Viscosity and density measurement

The prepared solution (Glycolic acid-Water mixture) on individual concentrations is filled and it reaches the mark on the tube. The viscometer is now placed in a constant position and maintained at a constant temperature (303K). Time measurement carried out for all concentrations of an accuracy of 0.01 s and repeated for 2-3 times to minimize the error. The Ostwald viscometer is a function both of these dynamic viscosity and density known as the kinematic viscosity.

Relative viscosity of the solution can be measured by the equation:

$$\eta_r = \eta_w \left(\frac{t_s \rho_s}{t_w \rho_w} \right) \quad \dots (3)$$

η_r is dynamic viscosity, ρ_s is density of solution, ρ_w is density of water, t_s is time of flow for solution and t_w is time of flow of water.

2.4 FTIR studies

The FTIR spectrum of pure glycolic acid and mixture in various concentrations were recorded by Perkin-Elmer FTIR spectrophotometer at room temperature. Pure glycolic acid mixed with KBr and spectra were recorded in the entire frequency region. ZnSe cuvette is used for spectral measurement on mixture solutions. The precession of the equipment on measurement is approximately $\nu=1 \text{ cm}^{-1}$.

3 Results and Discussion

3.1 Dielectric parameters

3.1.1 Static dielectric constant and relaxation time

The complex permittivity of GA-water mixture has been measured as a functional frequency, temperature and concentration. The complex permittivity spectrum of aqueous GA is shown in Fig. 1. In spectra, overlap of dielectric permittivity and loss takes place above 8GHz and then decreases to maximum frequency range. We restrict ourselves in the lower concentration and mid frequency region; at the same our system of frequency range was restricted up to 8 GHz, *i.e.*, 10 MHz to 8 GHz is sufficient for water molecular interaction systems. Binding of water molecules in all activity sites with glycolic acid was considered in a

systematic fashion. The static dielectric constant (ϵ_0) of mixture depends on the potential energy surface of solute-solvent molecules. The static dielectric constant (ϵ_0) of mixture studied from 0.001 mol/lit to 0.008 mol/lit with increase in steps of 10^{-3} mol/lit. The static dielectric constant of GA increases with increase in concentration up to 0.005 mol/lit and then decreases gradually for particular temperature shown in Fig. 2.

The static dielectric constant of mixture increases when reducing the temperature (303 K-288 K). This significantly reveals that two O-H stretching vibrations or transitions and is quite interesting because alcohol O-H exhibits intra molecular hydrogen bonding, while O-H group in acid is also available for intermolecular hydrogen bonding. These results may be expressively exhibits for understanding the bonding effects of clustering and nucleation in more general types of hydrogen bonded system and to

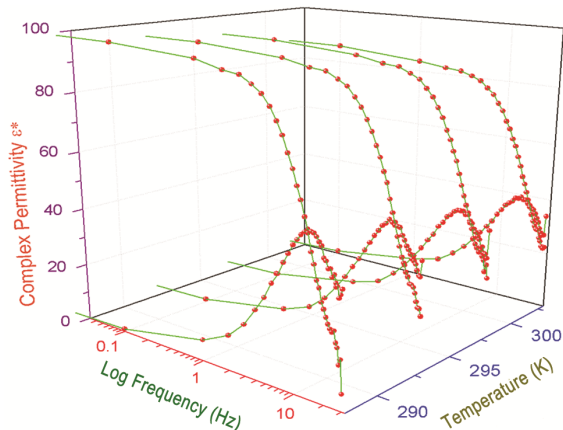


Fig. 1 – 3D plot of complex permittivity spectra of GA-water mixture for 0.002 mol/lit.

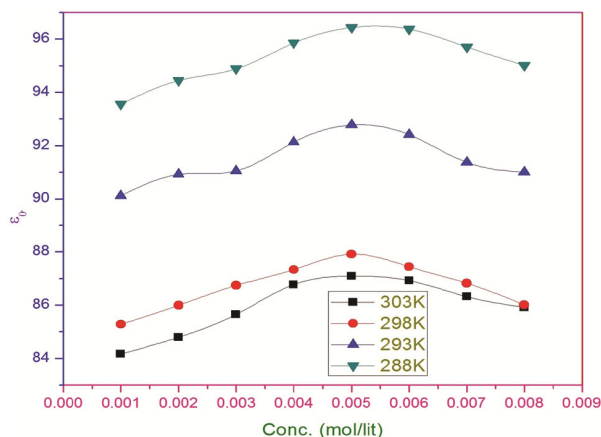


Fig. 2 – Plot between static dielectric constant versus concentration of glycolic acid-water mixture.

continued study of complex clusters in very high frequency techniques. This shows the formation and breaking of hydrogen bond network with one or two water molecules²⁸. Steinhoff *et al.*²⁹ reported the permittivity values of water in microwave region operated at GHz frequency. Fernandez *et al.*³⁰ measured the static dielectric constant of water and steam extends over a temperature range of 238 K to 873 K and measurement in the frequency range from MHz to GHz. The microwave studies show trans-GA with relative energy of $\approx 1200 \text{ cm}^{-1}$. The microwave spectroscopic studies show the lowest energy structure of GA to be symmetric with the alcoholic hydrogen group pointing toward the carboxyl oxygen of carboxyl group. This structure involves the intra-molecular bonding between the alcohol hydrogen and carboxyl oxygen.

The relaxation characteristics of GA-water mixture arises due to restricted reorientational motions of molecular electric dipoles. The relaxation time of water was found in various literature studies both on theoretical and experimental work with different frequency regions from kHz to THz^{31,32}. The dielectric relaxation properties of water are most accurately measured by Kaatze *et al.*³³ at different temperature and numerous dielectric parameters have been measured in broad frequency range. Kress *et al.*³⁴ estimate the molecular structure and intermolecular polarization data derived from Quantum-chemical studies. The calculated relaxation time significantly matches with literature values and it confirms the additive nature of solute-solvent interaction as shown in Fig. 3.

3.1.2 Kirkwood and corrective correlation Factor

The Table 1 represents the effective Kirkwood correlation factor g^{eff} provides useful information regarding the orientation of dipoles and is defined by:

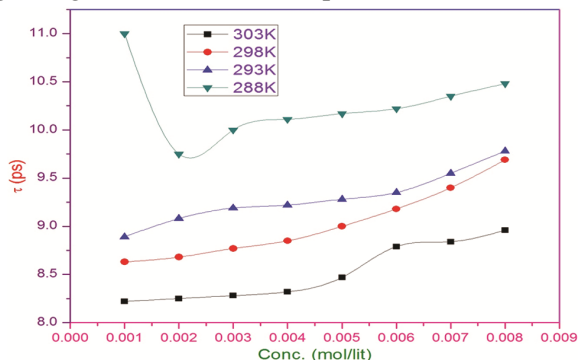


Fig. 3 – Plot between concentration versus relaxation time of GA-water mixture.

Table 1 – Activation energy (ΔF), Kirkwood correlation factor (g), effective Kirkwood factor (g^{eff}) and conductivity (σ) of GA-water mixture at various temperatures and concentrations.

Temperature (K)	Concentration (mol/lit)	ΔF (k cal.mol ⁻¹)	g	g^{eff}	σ (m.mho)
303	0.001	8.23	1.00	1.93	4.08
	0.002	7.83	1.01	1.94	4.69
	0.003	7.61	1.02	1.96	6.92
	0.004	8.49	1.03	1.99	6.65
	0.005	8.42	1.03	2.00	8.07
	0.006	8.16	1.03	1.99	6.48
	0.007	7.34	1.02	1.98	8.14
	0.008	6.39	1.02	1.97	9.06
298	0.001	8.96	1.00	2.15	3.78
	0.002	9.5	1.01	2.28	4.49
	0.003	9.4	1.02	2.41	6.45
	0.004	8.9	1.02	2.53	6.33
	0.005	8.59	1.03	2.65	7.60
	0.006	7.66	1.03	2.33	5.68
	0.007	6.48	1.02	2.31	6.80
	0.008	5.04	1.01	2.28	9.20
293	0.001	9.83	1.00	2.44	3.31
	0.002	10.55	1.01	2.57	4.04
	0.003	10.26	1.01	2.63	6.22
	0.004	10.96	1.02	2.72	6.07
	0.005	11.46	1.03	2.79	6.61
	0.006	10.97	1.03	2.53	5.38
	0.007	9.74	1.01	2.49	6.69
	0.008	9.14	1.01	2.47	7.02
288	0.001	11.46	1.00	2.51	2.75
	0.002	12.28	1.01	2.57	3.65
	0.003	12.43	1.01	2.66	5.23
	0.004	13.11	1.02	2.81	5.47
	0.005	13.32	1.03	3.02	5.89
	0.006	13.05	1.03	2.72	4.76
	0.007	12.26	1.02	2.64	5.78
	0.008	11.48	1.02	2.57	6.26

$$g = 1 + Z \cos \gamma \quad \dots (4)$$

where Z is the average number of nearest neighbours and γ is angle between the dipole moments of the neighbouring molecules.

Kirkwood correlation factor determined by following expression:

$$g = \frac{9kTM}{4\pi\mu^2N\rho} \left[\frac{(\epsilon_s - \epsilon_\infty)(2\epsilon_s + \epsilon_\infty)}{\epsilon_s(2 + \epsilon_\infty)^2} \right] \quad \dots (5)$$

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_s is static permittivity and ϵ_∞ is the dielectric permittivity at high frequency ($\epsilon_\infty = n_D^2$). The molecular relaxation time is affected by the intra and intermolecular hydrogen bonding.

The effective Kirkwood correlation factor g^{eff} confirms inter-molecular association with the dielectric relaxation time assuming the mixture is represented by one correlation factor g^{eff} as follows:

$$g^{\text{eff}} = \frac{9kT}{4\pi N} \frac{(\epsilon_s - \epsilon_\infty)(2\epsilon_s + \epsilon_\infty)}{\epsilon_s(\epsilon_\infty + 2)^2} \left[\frac{M_A}{\mu_A^2 \rho_A X_A} + \frac{M_B}{\mu_B^2 \rho_B X_B} \right] \quad \dots (6)$$

Where X_A and X_B are volume fraction, M_A and M_B are molecular weight, ρ_A and ρ_B are density of solute and solvent, respectively. ϵ_s static permittivity of binary mixtures A and B.

When Kirkwood correlation factor g and $g^{\text{eff}} > 1$, there is a tendency towards parallel alignment of dipoles, < 1 significances antiparallel alignment is favoured and a value of unity is expected when orientational ordering is absent. In our system of work

g is slightly greater than one so that all the dipoles in glycolic acid mixtures towards parallel alignment. The g^{eff} of mixture is highly greater than 1 for entire concentrations due to all random oriented dipoles are aligned in parallel. The g^{eff} increases along with neighbouring dipole moments suggest that the increase in molecular size does not veil the effect of the decrease in their intermolecular association. This has been observed that the orientation ordering is parallel alignment of dipoles³⁵.

3.1.3 Excess dielectric properties

The excess static permittivity study of mixture gives the H-bond contribution to dielectric parameters of the mixture. The excess permittivity determined by following equation:

$$\epsilon^E = (\epsilon_s)_M - [(\epsilon_s)_w X_w + (\epsilon_s)_A (1 - X_w)] \quad \dots (7)$$

where the subscripts M , W and A represents static dielectric constant of mixture, water and solute, respectively. The mole fraction of water in glycolic acid is represented by X_w . The excess dielectric constant of mixture shows the nature of molecular interaction in solution state. It also confirms the molecular interaction. The ϵ^E value provides the information, if ϵ^E of the mixture is zero the constituents do not interact and they have ideal mixing manner. When ϵ^E values lesser than zero the constituents interact so as to reduce the total number of effective dipoles contributed in the mixture dielectric values. If suppose $\epsilon^E > 0$ indicates that the constituents of a mixture interact in such a way that there is an increase in number of effective dipoles contributed in the mixture.

The ϵ^E values of mixtures are positive and higher for all concentrations. This indicates parallel alignment of GA-water binary mixture and strong H-bond, respectively. The positive value shows easy electron transport between molecules. Therefore interaction takes place between two OH group of glycolic acid and water enhance complex formation. Excess dielectric permittivity values of mixture are reported in Fig. 4.

3.2 Density, viscosity and conductivity

The density and intrinsic viscosity for glycolic acid and water mixture are about 1.0059 kg/m³ and 0.9200 cP, respectively. The determined density and viscosity of binary mixture reported in Fig. 5. The formation of strong H-bonding between glycolic acid + water, and viscosity increases linearly due to formation of

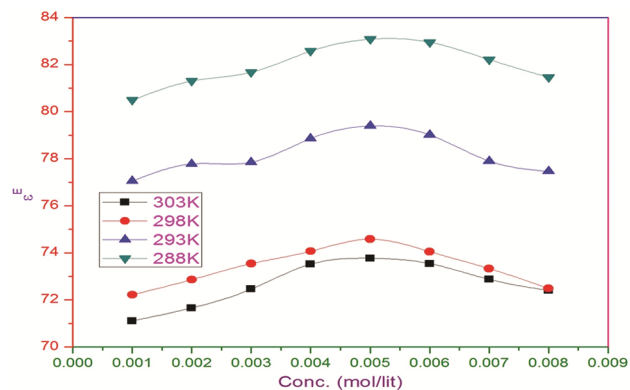


Fig. 4 – Plot between concentration versus excess permittivity of GA-water mixture.

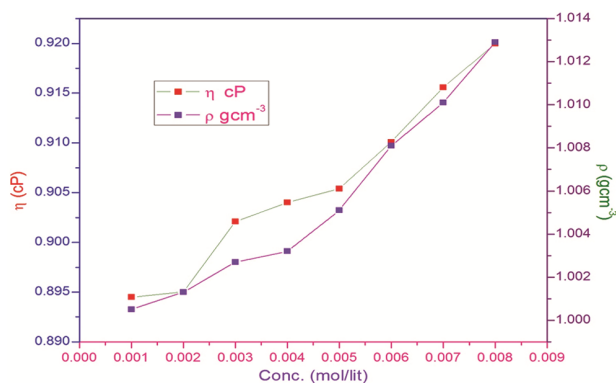


Fig. 5 – Plot between concentration versus viscosity (η) and density (ρ) of GA-water mixture.

more cluster or coagulation takes place and group molecular rotation increases the time period of rotation. The formation of strong hydrogen bonding and different structural arrangements causes increase in the viscosity and relaxation time of a molecule.

Conductivity of GA-water mixture determined following expression:

$$\sigma = \frac{C\epsilon}{d} \frac{R_1(t) - R_x(t)}{R_1(t) + R_x(t)} \quad \dots (8)$$

where $R_1(t)$ and $R_x(t)$ values of reflected pulses at time $t=\infty$ without and with samples, respectively, and ϵ is the permittivity of free space. The calculated conductivity values of mixture for various temperatures reported in Table 1

The ionic conductivity is maxima at mid concentration region and then decreases gradually. The electrical conductivity increases due to more number of water molecules which are connected with glycolic acid and then increases the ionic charge concentration in solution.

3.3 Thermodynamic studies

Tinsay and Siraj³⁶ studied the kinetics mechanism of GA mixture and measured different thermodynamic parameters. The thermodynamic properties of glycolic acid in condensed state have not been studied recently so far. The formation of intra molecular H-bond in glycolic acid-water complex may be affected upon H-bond with water. The formation of significant impact on the intermolecular H-bonding network with water binds to glycolic COOH group. The intermolecular H-bond involves water OH acceptor and acid OH donor groups.

Dielectric strength $\Delta\epsilon = \epsilon_s - \epsilon_\infty$ is the difference between static dielectric constant and static dielectric permittivity at optical frequency. Sengwa *et al.*³⁷ studied the dielectric strength of aqueous propylene glycol. Activation energy $\Delta F = \epsilon_s - \epsilon'$ is the energy difference between the reactants and the transition energy state where the activated complex is formed. The calculated dielectric strength and activation energy are reported in Fig. 6 and Table 1, respectively. The complex formations of glycolic acid are an unstable species when formation needs no additional energy to precede the reaction. The energy difference between the reactants and the product is due to change in enthalpy, entropy and this will lead to creation of Gibb's free energy. The values of enthalpy ΔH are a measure for the amount of binding energy that is gained in the transition state to the ground state observed by Emel'yaneko *et al.*³⁸ also studied about discrete thermodynamic parameters of glycolic acid.

The calculated enthalpy ΔH and entropy ΔS for GA-water mixture reported in Fig. 7. The transformation energy or entropy ΔS are a measure for the differences in disorder between the transition state and the ground state. The amount of heat transfer

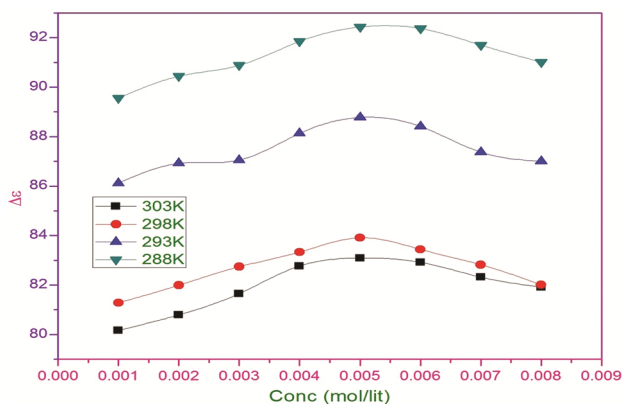


Fig. 6 – Plot between concentration versus dielectric strength of GA-water mixture.

of the reaction indicates entropy is increasing or decreasing. The increasing entropy shows positive ΔS (or) negative ΔS . The calculated ΔS show near the values of unity with positive give entropy is increasing from the reaction and is more disordered. The positive value of ΔS indicates the formation of complex in the rate determining step.

The Gibb's free energy of activation ΔG determines rate at which a certain activated complex^{39,40} is slow or fast which can be obtained by the following expression:

$$\Delta G = \Delta H - T\Delta S \quad \dots (8)$$

Our system of binary mixture shows a negative values of ΔG suggested that the formation of activated complex is high and the direction of reaction. The calculated values show slight increase and decrease on increase in the concentration in Fig. 8. The large negative value observed in this reaction suggests a considerable degree of charge separation of (or) from the activated complex. The negative value decrease from (or) to increasing the concentration shows rate

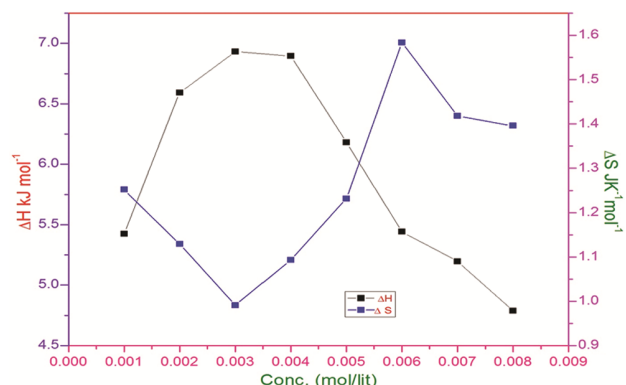


Fig. 7 – Plot between concentration versus enthalpy (ΔH) and entropy (ΔS) of GA-water mixture for room temperature.

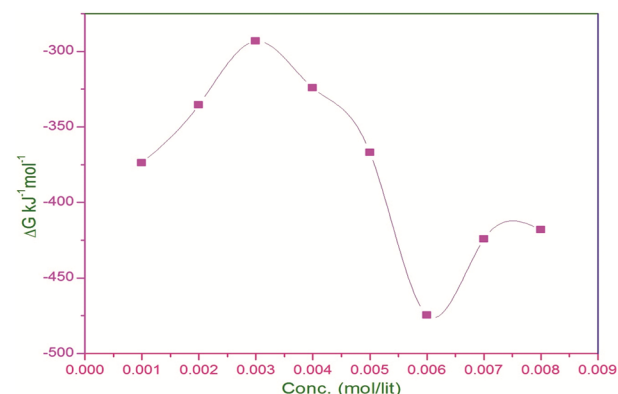


Fig. 8 – Plot between concentration versus Gibb's free energy of GA-water mixture.

and direction of the reaction become complex in the activated process is high. Based on thermodynamic parameters the mixture processes to involve large charge separation and results to increase the total number of charges between two ends of highly solvated molecules. The immobilization of large number of solvent molecules reduces entropy and Gibb's energy value.

3.4 FTIR analysis

The FTIR spectrum of glycolic acid+ water mixture is shown in Fig. 9. Carboxylic acid are specifically characterized by the –OH stretching frequency, the C=O stretch and –OH out-of-plane deformation and another C-O stretch with –OH in-plane deformation. Basically C=O stretching vibration in the carboxylic acid gives strong band at 1725 cm^{-1} but our observed values at 1735 cm^{-1} . Significantly it represents the wave number shifts from blue region to red⁴¹. The sharp and narrow band appears at 1637 cm^{-1} provides the H₂O deformation. In the case of acid species the average carbonyl double bond group $\nu_{\text{C=O}}$ at $1725\pm 12\text{ cm}^{-1}$ with maximum absorption of 0.25 molar au/vibrating group. On increasing the concentration of the solution to maximum, we observed 0.22 molar au/vibrating group. These values are matches with cabaniss *et al.*^{42,43}. The vibrations of the CH₂ group, CH₂ in-plane deformation appear at 1434.95 cm^{-1} , 1355.76 cm^{-1} and 1239.17 cm^{-1} specifically represent the CH₂ rocking and wagging mode of vibrations, respectively. Our $\nu_{\text{C-O}}$ appears at 1239.17 cm^{-1} with approximate intensity 0.1 molar au molar/vibrating group⁴⁴. The narrow band appears at 1091 cm^{-1} clearly states that stretching vibration of C-OH. The high and broad absorption peak appears at 3447.51 cm^{-1} and 2568.41 cm^{-1} , respectively. There are two main reasons for this broadening (i) the weakening of

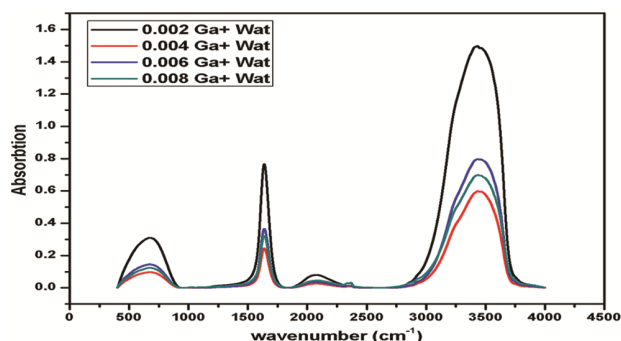


Fig. 9 – FTIR spectrum of GA-water mixture for various concentrations.

hydrogen-bonding network and (ii) formation of water clusters with solute molecules. The band 3447.51 cm^{-1} is –OH stretch of pure water, at the same time the water molecule that has not been strong bonded to the carboxyl group but bonded. The band 2071.86 cm^{-1} shows a slight hump with broaden region, which indicates the aqueous acid than the pure water. The assignments of mixture validate the bonding of GA-water

4 Conclusions

Molecular interactions between the solute-solvent molecules in all concentration of binary system have been interpreted in terms of inter and intra molecular hydrogen bonding through dielectric properties and thermo dynamical parameters with change in environmental condition such as temperature, concentration and frequency. The relaxation behaviour of mixture are explained with Debye model and determined from the values of time dependent relaxation response. The relaxation time increases with increase in concentration of glycolic acid. As the size of the molecule increases, molecular interactions become stronger. The Kirkwood correlation factor increases with increase in the molecular size of the liquid but decreases with increase in temperature. Kirkwood and effective Kirkwood correlation factors greater than unity indicates parallel alignment of solute-solvent molecules. The FTIR spectral study confirms H-bonded molecular interaction of GA with water.

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