Dielectric Measurements Of Nicotinic Acid In Aqueous Solutions Using Frequency And Time Domain Techniques

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Abstract

Dielectric relaxation properties for nicotinic acid in water medium were carried out using time domain reflectrometry (TDR) technique at 25^0C, 20^0C, 15^0C and 10^0C in the microwave frequency range from 10 MHz to 50 GHz. The relaxation behaviour of aqueous solutions of nicotinic acid has been illustrated by using Debye model. The static dielectric constant (ε_0), dielectric constant at high frequency (ε_∞) and relaxation time (τ in ps) extracted from the complex permittivity spectra using least squares fit method. Hydrogen bonding interactions for aqueous solution of nicotinic acid studied through the determination of Kirkwood correlation factor (g^{eff}) and thermodynamic parameters (enthalpy (ΔH) and entropy (ΔS)). Complex relative dielectric permittivity spectra of nicotinic acid in water medium were obtained using in the frequency domain (LCR) technique in the frequency range of 20Hz to 2MHz for different concentrations at 25^0C. The complex dielectric function $\varepsilon^*(\omega)$, dielectric loss (tan δ), electric modulus $M^*(\omega)$, electrical conductivity $\sigma^*(\omega)$ have been studied.

Keywords: *Time domain reflectometry; Frequency domain technique; Kirkwood correlation factor*

I. Introduction

Nicotinic acid (NA) is also known as Vitamin B3, is an important nutrient and safe for human body. Nicotinic acid (niacin) has long been used for the treatment of lipid disorders and cardiovascular disease [1]. Nicotinic acid's structure includes a pyridine ring and a carboxylic acid group, making it polar and capable of forming hydrogen bonds with water molecules. These intermolecular interactions significantly affect its dielectric behavior in aqueous solutions. The niacin has many applications as medicines, nutrients and drugs, but there are less reports and information in literature about its electrical characteristics and properties. The NA has wide and vast applications in biological, nutrients and food safety as basic vitamin B3. However, there is too lack in electrical characterization and applications reports rather than its biological applications reports. The aqueous solution of nicotinic acid give very effective information about molecular interactions. Therefore, the study of aqueous solution of nicotinic acid having great importance in the field of molecular physics [1].

Dielectric relaxation studies play a crucial role in understanding the behavior of molecules in different environments, particularly in solution mediums like water. Nicotinic acid, also known as niacin or vitamin B3, is an essential organic compound with diverse biological functions. Studying its dielectric relaxation in a water medium provides valuable insights into its molecular dynamics and interactions. When dissolved in water, nicotinic acid exhibits interesting dielectric relaxation properties, which offer its structural and dynamic characteristics. This study investigates into the dielectric relaxation of aqueous solutions of nicotinic acid and focuses on the relationship between this biomolecule and its surrounding solvent. Aim of the present work is to enhance our understanding of its solution properties, ultimately contributing to broader visions into the behaviour of biomolecules in aqueous environments. The dielectric relaxation study of nicotinic acid in aqueous media has received considerable attention [2-5]. Malathi et.al. [5] reported Microwave dielectric relaxation parameters of aqueous Nicotinamide (vitamin B3) using time domain technique.

In the present paper, investigating the hydrogen bonding interaction and dielectric relaxation properties for nicotinic acid in water medium in the microwave frequency range from 10MHz to 50 GHz using time domain reflectometry technique at 25^0C, 20^0C, 15^0C and 10^0C. From the complex permittivity spectra, the static dielectric constant (ε_0) and relaxation time (τ) were obtained by least square fit method. Kirkwood correlation factor and thermodynamics parameters were also determined to study the heterogeneous interactions through hydrogen bonding. Also in the present study, the complex permittivity spectra were obtained for different concentration of water in molar solution of nicotinic acid at 25°C using precision LCR meter in the frequency range of 20Hz to 2MHz. The electrical and dielectric properties of the aqueous solution are represented in terms

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of complex dielectric function ε*(ω), electrical modulus M*(ω), electrical conductivity σ*(ω). This data might be useful to characterize and analyze pharmaceutical material, food processing industry and in solubility prediction method in aqueous solution [6-7].

II. Materials And Methods

Time domain reflectrometry technique

Nicotinic acid in powder form were obtained commercially from S. D. Fine Chemical Limited, India and was used without further purification. Water was carefully purified by deionization and double distillation and was used for the measurement immediately after the purification. The solutions were prepared at room temperature for different mole fraction water. The dielectric measurements were carried out using TDR[8-9].

The complex permittivity spectra were studied using TDR technique [10-13]. The Tektronix model no. DSA8300 Digital Serial Analyzer sampling mainframe along with the sampling module 8E10B has been used for the dielectric measurement using TDR technique. A repetitive fast rising voltage pulse was fed through coaxial line system of impedance of 50Ω. All measurements are carried out in open load condition. Sampling oscilloscope monitors the changes in step pulse after reflection from the end of line. Reflected pulse without sample R_1 (t) and with sample R_x (t) were recorded in time window of 5 ns in the memory of the oscilloscope and transferred to the computer for further analysis. The selection of proper time window, sampling rate and bilinear calibration method suggested by Cole et al. were useful to measure the accurate complex permittivity at higher frequencies [14]. The temperature of the thermal system was regulated by a refrigerating cooling system with an accuracy of ±0.5⁰C. Further the Fourier transformation of the pulses and data analysis were done earlier to determine complex permittivity spectra using nonlinear least squares fit method [14-15]. The experimental setup of Time Domain Reflectometry is shown in Fig. 1.
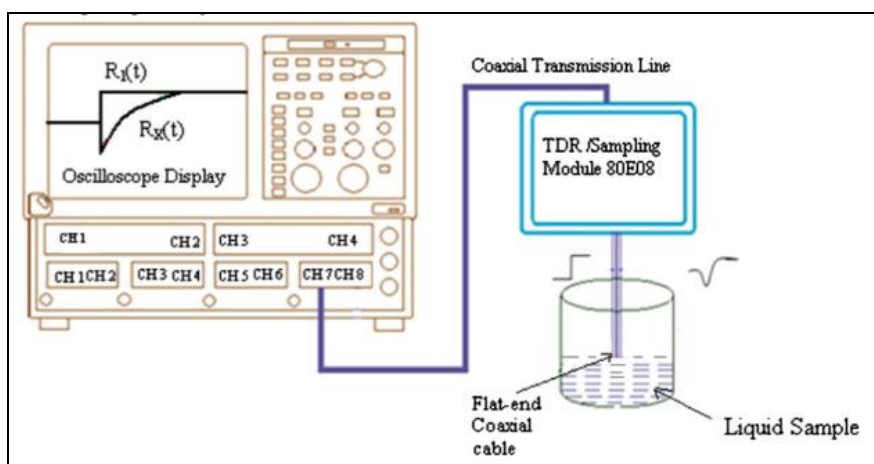
Figure 1. Experimental set up of TDR

Frequency domain technique

The complex relative dielectric function ε*(ω) of aqueous solution of nicotinic acid were determined by using Agilent E4980A precision LCR meter. A four terminal dielectric test fixture, Agilent 16452A was used for capacitance and resistance measurement in the frequency range 20Hz to 2MHz. The capacitance and parallel resistance of the liquid dielectric test fixture without and with samples were measured to compensate for a short [16]. The test fixture correction coefficient was also considered to cancel the effect of stray capacitance during the evaluation of the value of the complex dielectric function. The complex dielectric function ε*(ω) of the materials is determined using Eq.1

$$\varepsilon^*(\omega) = \varepsilon'(\omega) - j\varepsilon''(\omega) = \alpha \left[\frac{C_p}{C_o} - j \frac{1}{\omega C_o R_P} \right] \tag{1}$$

Where ω = 2πf is the angular frequency and α is the correction coefficient of the cell.

III. Result And Discussion

Complex permittivity using TDR technique

The real part ε′(ω) and imaginary part ε″(ω) of dielectric permittivty of aqueous solution of Nicotinic acid at 25⁰C, were measured using TDR technique as shown in Fig. 2. It is observed that at the relaxation

frequency of the real part ε′(ω) of complex permittivity spectrum of aqueous solution of Nicotinic acid decreases with increase in the frequency. It is due to the less effect of applied field which has been cancelled by the dipoles.
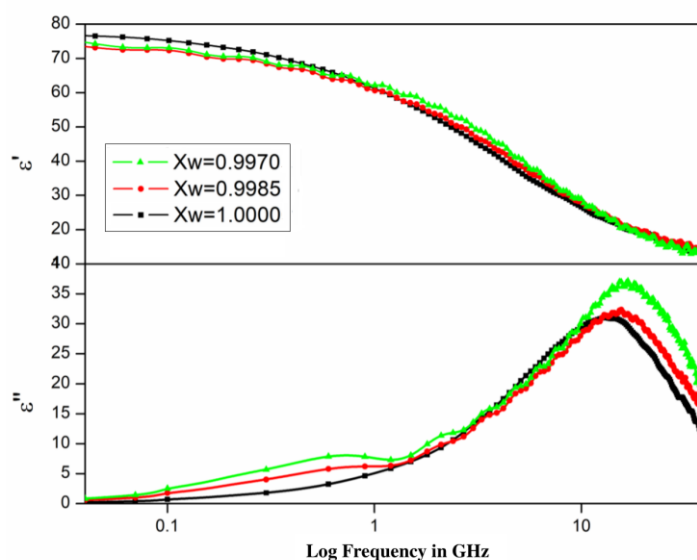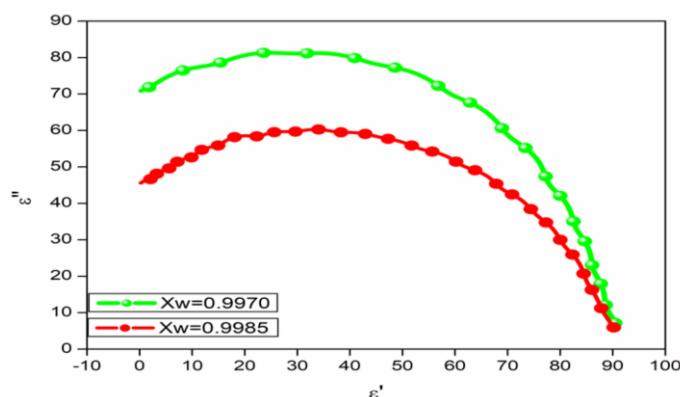

Figure 2. Plot of dielectric permittivity (ε') and dielectric loss (ε") for aqueous solution of nicotinic acid with different mole fraction of water (X$_W$) at 25^{0}C.

The complex permittivity ε*(ω) data were fitted by the non-linear least squares method to the Debye model [17].

$$\varepsilon^{*}(\omega) = \varepsilon_{\infty} + \frac{\varepsilon_0 - \varepsilon_{\infty}}{1 + j\omega\tau} \qquad (2)$$

where, ε_0 is the static dielectric constant, ε_{∞} is the permittivity at high frequency, τ is the relaxation time in pico-second. In this study the aqueous solution of Nicotinic acid shows Debye type dispersion. Therefore the complex permittivity spectra ε*(ω) of aqueous solution of Nicotinic acid were fitted in Debye model using non-linear least squares fit method to determine the dielectric relaxation parameters. The real part (ε′) of complex permittivity will decrease when the relaxation frequency is reached since less of the applied field is cancelled by the dipoles as the frequency is increased. In an imaginary part (ε″) of the complex permittivity spectra the peak value occurs at this frequency since most of the energy lost at that point[18]. The Cole-Cole plot shows dielectric loss ε″(ω) for aqueous solutions of Nicotinic acid at different mole fraction of water as a function of dielectric dispersion ε' (ω) in Fig. 3.


Figure 3. Cole-Cole plot for nicotinic acid in water medium at 25^{0}C.

Dielectric permittivity spectra using frequency domain technique
The frequency domain spectroscopy using high precision LCR meter is utilized to extract the dielectric properties of corresponding liquid samples at low frequencies. The dielectric permittivity (ε′) spectra for

aqueous solutions of nicotinic acid are shown in Fig. 4. A sharp fall in ε′ values with frequency is observed in the low frequency region. This suggests the presence of ionic impurities as in the case of most of the polar materials. At a particular frequency, the ε′ curve becomes frequency independent. The frequency independence of ε′ values of the studied samples is observed in the frequency range of 40 kHz—50 kHz. Hence, the static dielectric permittivity values are recorded at 2 MHz and reported in Table 1 along with refractive indices.

(a)

(b)
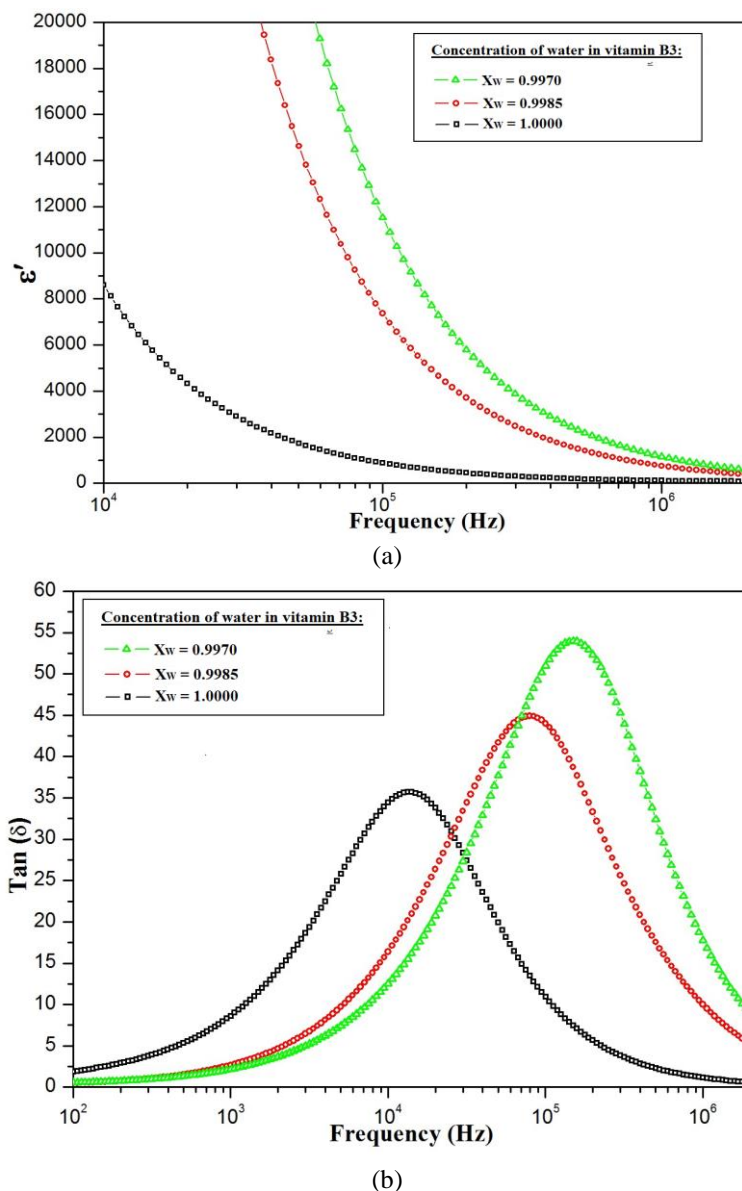
Figure 4. (a) Dielectric permittivity (e′) spectra for aqueous solution nicotinic acid using LCR (b) Spectra of loss tangent (tan δ).

Table 1. Static dielectric permittivity (ε_s) using LCR meter for Aqueous Solutions of nicotinic acid at different mole fraction of water (X_w) liquid samples at 25 °C.

X_W	$\varepsilon_{s(LCR)}$
0.9970	66.75(11)
0.9985	74.26(9)
1.0000	79.71(8)

Dielectric constant and Relaxation time

The values of dielectric parameters (ε_0, ε_∞, τ and β) for aqueous solution of nicotinic acid at 25^0C, 20^0C, 15^0C and 10^0C with different mole fraction of water(X_W) have been reported in Table 2.

Table 2. Dielectric relaxation parameter for aqueous solutions of nicotinic acid at different mole fraction of water (X_w) and temperature.

Temp = 25⁰C				Temp = 20⁰C			
X_W	ε_o	ε_∞	τ(in ps)	X_W	ε_o	ε_∞	τ(in ps)
0.9970	72.41(15)	3.00	8.79(14)		74.85(12)	3.00	9.16(11)
0.9985	72.99(8)	3.00	8.61(8)		74.01(8)	3.00	9.08(8)
1.0000	78.32(3)	2.04(3)	8.43(6)		79.74(8)	2.89(6)	9.62(3)
Temp = 15⁰C				Temp = 10⁰C			
X_W	ε_o	ε_∞	τ (in ps)	X_W	ε_o	ε_∞	τ (in ps)
0.9970	78.14(8)	2.00	9.50(7)		81.49(9)	2.00	10.30(7)
0.9985	75.64(9)	2.00	9.80(8)		78.59(9)	2.00	11.00(8)
1.0000	80.88(10)	2.85(4)	10.27(4)		82.89(3)	5.72(2)	11.32(1)

The errors in these parameters have been given in the brackets which shows an uncertainty in the last significant digits e.g. the static dielectric constant of water 79.74(8) means 79 ± 0.08. The increasing value of dielectric constant for the solution with increasing mole fraction of water (X_W) and systematic change in the dielectric parameters of the solution can be explained on the basis of molecular interactions.

The plot of ε_0, vs. mole fraction of water in Fig. 5 of aqueous solution of nicotinic acid shows the non-linear behaviour in all concentrations. The disruption or formation of hydrogen bonds between nicotinic acid and water molecules can influence the dielectric constant in a non-linear manner. The molecular structure of nicotinic acid, with its aromatic ring and carboxylic acid group, may contribute differently to the dielectric properties compared to water molecules.

The values of static dielectric constant and relaxation time decrease with an increase in temperature. This may be due to a decrease in orientational correlation of dipole moments with an increase in temperature [12]. The value of dielectric constant observed lower at X_W = 0.9985, this is may be due to disruption of the hydrogen bonding network in the solution and that reduces the overall ability of the solution to store electric energy, hence lower the value of dielectric constant.

From Fig. 6, the increase in τ values with increasing mole fraction of water indicates that number of dipoles increases in the solution, the intermediate structure formed rotates slower there by giving the increase in values of τ in the solution. The dielectric properties will also get affected by temperature. This is due to the effect of temperature on polarization mechanism and charge mobility. From the Table 1, the values of τ are decreasing with an increasing temperature [19], this is due to there may be hydrogen bonding and other interactions between molecules. As temperature increases, these interactions can be weakened, allowing molecules to move more freely and reducing the time required for the system to relax.
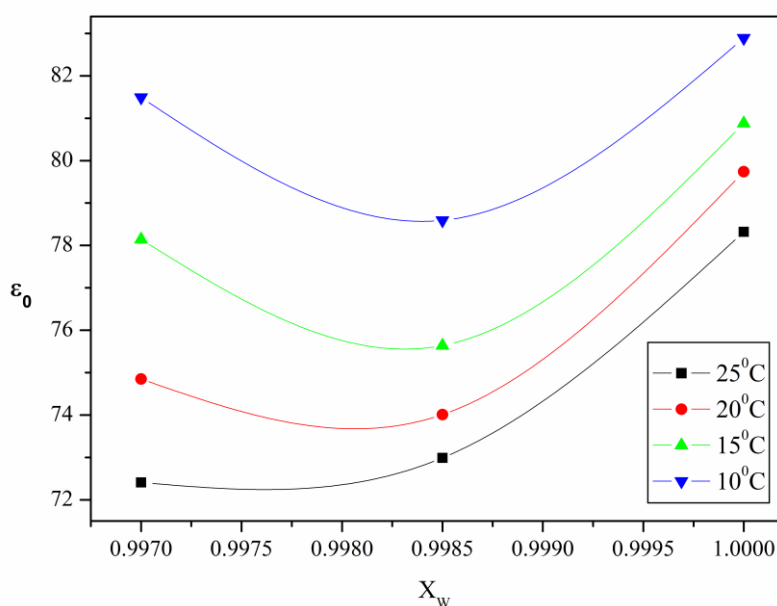
Figure 5. Static dielectric constant (ε_0) of vs. mole fraction of water (X_W) for aqueous solutions of nicotinic acid at different temperature.
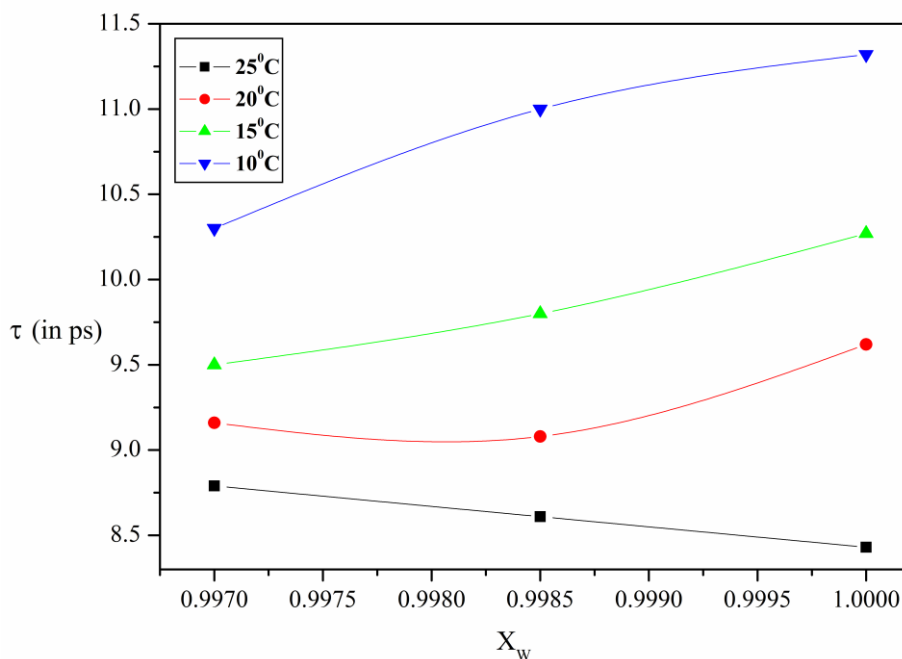
Figure 6. Relaxation time (τ) vs. mole fraction (X_w) of water for nicotinic acid in water medium at different temperature.

Kirkwood correlation factor from dielectric parameter

To understand the significance of association effect due to the hydrogen bonding, it is very useful to compute the values of Kirkwood correlation factor g for these binary mixtures using following expression [20-21],

$$\frac{(\varepsilon_0 - \varepsilon_\infty)(2\varepsilon_0 + \varepsilon_\infty)}{\varepsilon_0(\varepsilon_\infty + 2)^2} = g\mu^2 \frac{4\pi N\rho}{9\kappa TM} \qquad (3)$$

where μ, ρ and M correspond to the dipole moment in gas phase, density and molecular weight, respectively, k is the Boltzmann constant and N the Avogadro's number.

For binary mixture, the static dielectric permittivity needs to be considered as the dipole orientation correlation factor (g^{eff}). The modified form of Eq. (3) is used to study the orientation of the electric dipoles in the binary mixtures as follows[20-21],

$$\frac{4\pi N}{9kT}\left[\frac{\mu_x^2 \rho_x X_x}{M_x} + \frac{\mu_A^2 \rho_A (1 - X_x)}{M_A}\right] \times g^{eff} = \frac{(\varepsilon_{0m} - \varepsilon_{\infty m})(2\varepsilon_{0m} + \varepsilon_{\infty m})}{\varepsilon_{0m}(\varepsilon_{\infty m} + 2)^2} \qquad (4)$$

where M_X and M_A are molecular weight of water and aqueous solution of nicotinic acid respectively. ρ_X and ρ_A are corresponding densities, X_x is mole fraction of water. ε_{0m} and $\varepsilon_{\infty m}$ are the static dielectric constant and dielectric constant at high frequency of the mixtures. To calculate the values of g^{eff}, we have taken $\mu_A = 5.24D$ [5] and $\mu_x = 1.85D$ [22] for L-phenylalanine and water respectively. The values of ε_∞ are taken from the fitting data.

The values of g^{eff} for nicotinic acid in water medium at different temperature are reported in Table 3. It illustrates that g^{eff} values for all the concentrations greater than unity and that reveals parallel orientation of electric dipole among molecules. The g^{eff} values of aqueous solution of nicotinic acid at different temperature shown in Fig. 7. The g^{eff} values are decreasing with increasing the concentration of water indicates that there is stronger hydrogen bonded parallel dipolar ordering in the mixtures as compared to the g^{eff} values for pure water molecules.

Table 3. Kirkwood correlation factor (g^{eff}) for nicotinic acid in water medium at different temperature.

X_w	g^{eff}			
	25^0c	20^0c	15^0c	10^0c
0.9970	2.60(15)	2.64(15)	2.71(16)	2.78(16)
0.9985	2.62(15)	2.61(15)	2.62(15)	2.68(16)
1.0000	2.81(16)	2.82(17)	2.85(17)	2.84(17)
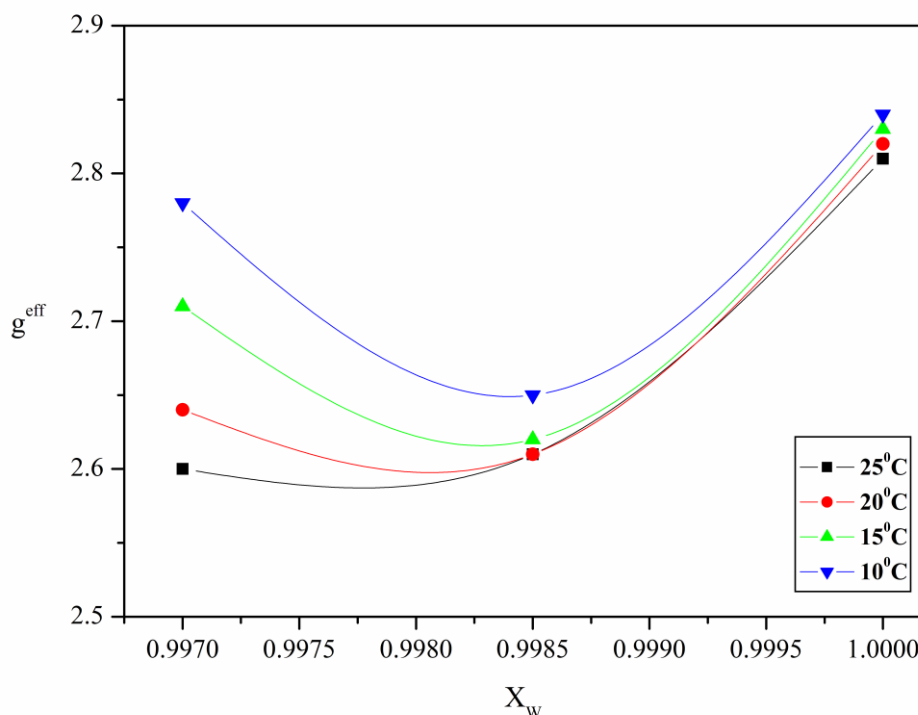
Figure 7. Plot of Kirkwood correlation factor (g^{eff}) with mole fraction water for nicotinic acid in water medium at different temperature.

Thermodynamics parameters

       The Eyring equation is used to describe the relationship between reaction rate and temperature. It is analogous to the Arrhenius equation, which also describes the temperature dependence of reaction rates. However, whereas Arrhenius equation can be applied only to gas-phase kinetics, the Eyring equation is helpful in the study of gas, condensed, and mixed-phase reactions [23]

       The Eyring equation gives a more precise calculation of rate constants and provides approaching into how a reaction progresses at the molecular level [24]. The thermodynamic parameters evaluated using Eyring equation is as follows [24]

$$\tau = \frac{h}{kT} \exp\left(\frac{\Delta H}{RT}\right) \exp\left(\frac{-\Delta S}{R}\right) \qquad (5)$$

where ΔS is the entropy of activation, ΔH is the activation energy in kJ/mol. τ is the relaxation time in ps and T is the temperature in (K), h is the Plank's constant and R is the gas constant. The results of activation energies are obtained by least square fit method and are reported in Table 4. The value of ΔH for water is 10.88 kJ/mol that is energy required to break single hydrogen bond [25]. However, it occurs low for $X_W = 0.9985$ and 0.9970 concentration, this reveals that the aqueous solutions of nicotinic acid requires less energy for rotation of molecules as well as to break hydrogen bonding than pure water. Also it suggests the hetero molecular association of nicotinic acid-water molecules.

Table. 4 Activation enthalpy ΔH(in (kJ/mol) and entropy ΔS (in J/mol k) of aqueous solution of nicotinic acid for different mole fraction of water.

X_w	ΔH	ΔS
0.9970	4.78(94)	0.223(006)
0.9985	8.08(76)	0.212(003)
1.0000	10.88(23)	0.233(003)

Electrical Modulus

       The electric modulus spectrum can be used to explore the relaxation process due to ionic conductivity taking place in liquids. The spectra of electric modulus can be utilized to overcome the space charge effect in the dielectric spectra of nicotinic acid-water mixture [26]. The values of complex electric modulus are evaluated from the equation:

$$M^*(\omega) = \frac{1}{\varepsilon^*(\omega)} = M' + jM'' = \frac{\varepsilon'}{\varepsilon'^2 + \varepsilon''^2} + j\frac{\varepsilon''}{\varepsilon'^2 + \varepsilon''^2} \qquad \textbf{(6)}$$

Figures 8(a and b) shows the real and imaginary parts of electric modulus of all the associating liquid samples. In lower frequency region ($<10^4$ Hz), the M′ values are very low and show negligible increase. Then M′ values increasing linearly with increase in frequency and approaching a constant value in the MHz region of frequency. The peak observed in M″ spectra correspond to a frequency (f_σ)which is associated with ionic conductivity relaxation time [27].
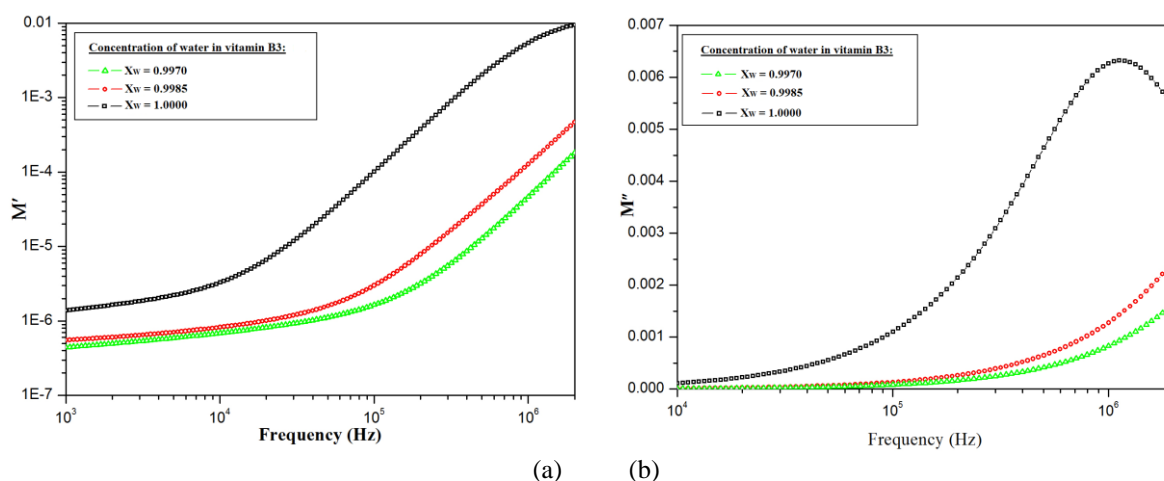
Complex electric conductivity (σ_{ac})

The study of electric conductivity is useful to describe the ionic traces in polar compounds. The complex electric conductivity (σ^*) values for nicotinic acid-water system are calculated by using the relation [26],

$$\sigma^*(\omega) = \sigma' + j\sigma'' = \omega\varepsilon_0\varepsilon'' + j\omega\varepsilon_0\varepsilon' \qquad (7)$$

Where, σ′ and σ″ are real and imaginary part of A conductivity respectively. ε_0 is dielectric permittivity of free space (8.854×10^{-12} F m^{-1}). The plot for real part of AC conductivity (σ_{ac} in units of S/m) versus frequency applied is shown in figure 9.

At lower frequency, σ_{ac} values are very small. As the frequency increases, the values of σ_{ac} also increasing linearly and eventually reaches a plateau region of static values at near about 10^4Hz i. e. the AC conductivity values become frequency independent.

(a)   (b)

Figure 8. (a) real part of electric modulus vs. frequency (b) imaginary part of electric modulus vs. frequency for nicotinic acid in water medium at 25^0C.
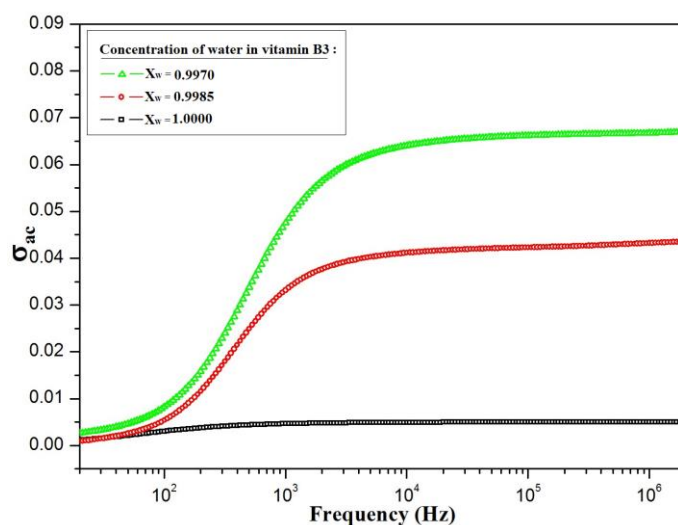
Figure 9. Complex conductivity vs. frequency for nicotinic acid in water medium at 25^0C.

IV. Conclusion

The dielectric measurements for nicotinic acid in aqueous solution have been done and their dielectric spectra is obtained using both LCR and TDR techniques. The study of dielectric spectra reveals how nicotinic acid behaves in water in terms of its electrical response. Using Debye model, microwave dielectric properties of aqueous solution of nicotinic acid have been carried out. Also investigating how the dielectric constant (ε_0), relaxation time (τ) are sensitive to the variations in mixture constituents of aqueous solutions of nicotinic acid. The observed shifts in dielectric relaxation times with changing temperatures suggest the presence of dynamic processes within the system. The Kirkwood correlation factor is greater than unity reveals parallel ordering of the electric dipole. Activation enthalpy confirms strong interaction takes place among aqueous solution of nicotinic acid. The non-zero complex conductivity values confirm the presence of ionic traces in the solutions. This data is valuable for understanding the molecular interactions, structural changes, and overall behavior of nicotinic acid in solution, contributing to fields such as chemistry, pharmaceuticals, and materials science. This study also contributes to the understanding of different types of vitamins in aqueous solutions and provides a foundation for future research in this domain.

Acknowledgements

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