

Study of structuredness and hydrogen bonding in binary solutions using microwave technique

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Dielectric and volumetric information of alcohols with different solvents provide eminent information regarding heteromolecular interactions and hydrogen bonding nature between diverse entities. Ordering of heteromolecules in their pure form as well as in binary mixture is studied thereby obtaining the Kirkwood correlation factor. Luzar model predicts the bonding between the hetero molecules such that the concentration dependent heteromolecular interactions study of isopropyl alcohol with dimethylsulfoxide and water liquid solutions with variations in the physiochemical and volumetric parameters has been revealed. The dielectric permittivity of the binary liquid mixtures have been obtained at 9.685 GHz using X-band microwave technique at room temperature.

Keywords: Static dielectric constant (ϵ'), Dielectric loss (ϵ''), Microwave conductivity, Excess permittivity, Kirkwood correlation factor, Structuredness

Assessment of structural properties using molecular interaction study under the influence of microwave frequency has considerable importance in pharmaceutical and chemical industries to know the physiochemical parameters due to intermolecular interactions executed among the diverse molecules that affects the chemical reactions. The evaluation and spectroscopic analysis of physical and chemical properties were done using time domain¹⁻⁴ and frequency domain, which has achieved finest results in the analysis of dielectric parameters to signify heteromolecules in the mixture. Frequency domain technique (FDT)⁵⁻⁹ plays a remarkable role in perceptive of molecular assets and intermolecular exchanges through hydrogen bonding in binary liquids⁹⁻¹⁰. It is the most convenient method that provides exact static dielectric constant (ϵ') values, especially of non-polar molecules in microwave region. In present context exposure of microwaves to binary solutions of Isopropyl Alcohol (IPA) with Dimethylsulfoxide (DMSO) and IPA with water causes variations in degree and structural behavior of

solute-solvent such that it recognizes the diverse interactions in dielectric properties. The properties such as static dielectric permittivity, dielectric loss, microwave conductivity (σ), penetration depth, loss tangent, excess permittivity, Bruggeman factor, pH and density (ρ) have been obtained to reveal the effect of structural and intermolecular interactions in the solutions. Thus the structuredness¹¹ or ordering of hetero molecules in its pure form as well as in binary solution is revealed using Kirkwood correlation factor. Luzar model recognizes the hydrogen bonding among the hetero molecules in terms of predicted number of hydrogen bonds, enthalpy, bonding energy and polarizability, respectively.

Isopropyl Alcohol [(CH₃)₂CHOH] is an example of secondary alcohol, non-toxic, colorless, flammable organic compound¹². It is widely used as a solvent and for anaesthesia, as a preservative in biological specimen, for etching, in eyeglass cleaning, electrical contacts, in heads of audio and video tapes, DVD and in lenses of optical disc, thermal paste remover for CPUs heat-sinks and other IC packages. It has use as

a major ingredient in gas dryer fuel additives, as a common ingredient in products such as antiseptics, disinfectants, hand sanitizer and detergents *etc.* Dimethyl sulfoxide [(CH₃)₂SO] is colorless polar aprotic solvent, water soluble, has garlic-like taste and dissolves in polar, nonpolar compounds and is soluble in a wide range of organic solvents¹³⁻¹⁵.

S. Guo *et al.*¹⁶ recently investigated intermolecular hydrogen bonding of isopropanol-water clusters using DFT calculations. Study concludes that, redshifts of O-H bond tensile peak, indicative of weakened O-H tensile strength and interaction among hydrogen bonds is strengthened with increase in water molecules. T. Sato *et al.*¹⁷ studied the dielectric relaxation spectroscopy of 2-propanol-water mixtures with a conclusion that the motion of individual alcohol molecules can be detected due to their relatively large size, they possess a single H-bond with high concentration. Kaatze *et al.*¹⁸ investigated dielectric spectrum of DMSO with water as a function of composition and concluded that the static permittivity reflects antiparallel dipole moment ordering. Ritzoulis¹⁹ investigated excess properties with negative values and Kirkwood correlation parameters of IPA+DMSO solutions, concluded the negative values of excess permittivity and no dipole interactions for DMSO and that of IPA the values greater than unity suggest antiparallel orientation of molecules.

The aim of present article is to investigate heteromolecular interactions and structuredness in homogenous mixtures of IPA+DMSO and IPA+Water such that IPA gets completely mixed in water and DMSO. The effect of high frequency on polarization and structural properties in these solutions was studied using FDT at room temperature such that these molecules possess different values of polarity index (PI).

Experimental details

Materials

Isopropyl Alcohol and Dimethylsulfoxide were purchased from Thermo Fisher Scientific Pvt. Ltd. Mumbai with 99.00% of purity and distilled water is used without further purification. The probable structural bonding between IPA+DMSO and IPA+Water is shown in Fig. 1.

Measurements

Static dielectric parameters were measured at 9.685 GHz frequency by means of frequency domain

technique using X-Band microwave bench. Analysis of data with procedure of frequency domain technique using X-Band microwave bench was systematically explicated formerly⁵⁻⁹. The densities of pure liquids and the binary mixtures were measured by using pycnometer, thereby taking doubly distilled water as a reference liquid. pH meter used was of Syntronics make and standardized before its experimental use.

Results and Discussion

Static dielectric constant

Dielectric parameters were obtained using X-band microwave technique. The relative permittivity of binary liquid mixture is obtained using the equation⁵⁻⁹ as,

$$\epsilon = \epsilon' - j\epsilon'' \quad \dots (1)$$

where, ϵ is the relative permittivity, ϵ' is the relative real permittivity and ϵ'' is relative loss and j is imaginary number with $\sqrt{-1}$ value. The static dielectric permittivity and loss is calculated by using the equations as⁵⁻⁹,

$$\epsilon' = \left(\frac{\lambda_0}{\lambda_c}\right)^2 + \left(\frac{\lambda_0}{\lambda_d}\right)^2 \quad \dots (2)$$

$$\epsilon'' = \frac{2}{\pi} \left(\frac{\lambda_0}{\lambda_d}\right)^2 + \left(\frac{\lambda_g}{\lambda_d}\right)^2 \frac{d\rho_{mean}}{dn} \quad \dots (3)$$

where, ϵ' is dielectric constant, ϵ'' is dielectric loss, λ_0 is free space wavelength, λ_d is wavelength in dielectric cell, λ_g is guided wavelength, $\lambda_c = 2a$ is cut-off wavelength ('a' is width of waveguide). To eliminate losses value, $\frac{d\rho_{mean}}{dn}$ is used, and is taken from slope of graph plotted between several mean values of ρ_n versus n values⁵⁻⁹.

In IPA+DMSO and IPA+Water binary mixtures, the value of static dielectric constant and dielectric loss goes on decreasing with increasing V_{IPA} , which is an indicative of net change in polarization with transition of elongated molecular aggregation into spherical aggregation structure²⁰, as tabulated in Table 1. These variations of V_{IPA} cause changes in ϵ' and is the effect of exchanges among unlike molecules which may create structural changes²¹⁻²³ thereby increasing heterogeneous interactions in solutions.

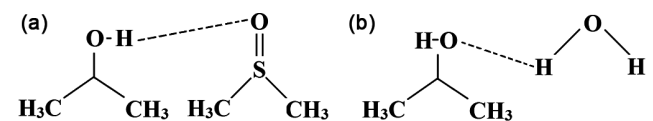


Fig. 1 — Probable bonding between (a) IPA+DMSO and (b) IPA+ Water.

Table 1 — Dielectric and volumetric parameters of IPA+DMSO and IPA+Water solutions at room temperature

V_{IPA}	ϵ'	ϵ''	σ^a	P_d^b	Tan δ	ρ^c	pH	g^{eff}
IPA+DMSO solutions								
0	47.84	13.50	0.7117	0.100	0.762	0.9990	2.59	1.199
0.2	38.83	10.4	0.5482	0.111	0.267	0.9320	2.46	1.197
0.4	27.11	8.26	0.4352	0.134	0.304	0.9170	2.40	1.063
0.6	20.04	5.45	0.2875	0.155	0.272	0.8891	2.02	1.066
0.8	17.51	5.19	0.2736	0.166	0.296	0.8513	2.77	1.438
1.0	15.45	4.45	0.2349	0.177	0.288	0.8136	2.89	2.647
IPA+Water solutions								
V_{IPA}								
0	78.78	23.56	1.2418	0.078	0.299	1.0365	6.81	2.875
0.2	60.42	18.11	0.9544	0.897	0.299	1.0171	6.68	2.588
0.4	47.84	15.14	0.7982	0.100	0.316	1.0072	5.67	2.498
0.6	38.37	14.07	0.7417	0.111	0.362	0.9938	5.11	2.589
0.8	27.11	10.72	0.5648	0.134	0.395	0.9648	3.56	2.611
1.0	15.45	4.45	0.2349	0.177	0.288	0.8136	2.89	2.647

^aUnit: S/m; ^bUnit: m.; ^cUnit : gm/cm³

The effect of PI^{24-25} is also seen on the dielectric constant values such that with decrease in polarity index, dielectric constant value of the liquid goes on decreasing that is PI is in the order as Water>DMSO>IPA and ϵ' is in the order as Water>DMSO>IPA, respectively. Thus the dielectric constant of the systems used for study follows the effect of PI.

Microwave Conductivity, Loss tangent and Penetration depth

The values of Microwave conductivity (σ), Loss tangent and Penetration depth were obtained using the following relations as⁵⁻⁹,

$$\sigma = \frac{f\epsilon''}{1.8 * 10^{11}} \quad \dots (4)$$

$$\text{Tan}\delta = \frac{\epsilon'}{\epsilon''} \quad \dots (5)$$

$$P_d = \frac{\lambda_0 \sqrt{\epsilon_r'}}{2\pi\epsilon_r'} \quad \dots (6)$$

where, σ is microwave conductivity, f is microwave frequency, ϵ' is dielectric constant, ϵ'' is dielectric loss, λ_0 is free space wavelength, P_d is penetration depth and ϵ_r is relative permittivity.

The microwave conductivity is a function of dielectric loss and in above two solutions, it decreases with increase in V_{IPA} and compared to both the systems the conductivity of pure Water is greater than that of pure DMSO. Ratio of dielectric loss to the permittivity of material is the Loss tangent^{5, 26}. For IPA+DMSO, value of loss tangent is least at $V_{IPA} = 0.2$ and it is extreme for pure DMSO. In IPA+Water solutions, the value of loss tangent is maximum at 0.8

V_{IPA} and minimum for pure IPA. The depth, at which power gets reduced to 1/e of its surface value, is nothing but penetration depth^{5, 26}. Penetration depth value goes on increasing for both systems with increase in V_{IPA} .

Density and pH

For present systems, density(ρ) of the liquid mixtures is obtained using the formula for density as,

$$\rho = \frac{m}{V} \quad \dots (7)$$

where, ρ is density, m is mass and V is volume of solution.

Density of solutions goes on decreasing with increase in V_{IPA} in both the systems. As IPA having less density than water and DMSO both such that increasing V_{IPA} , causes decrease in density of mixture thereby decreasing the values of dielectric constant of mixture in both the systems²⁷, results in dependency of dielectric constant on the density of the mixture. pH is the concentration of hydrogen ions, that is a scale used to measure the nature of molecules such as its acts as base or acid. The pH values of the solutions given in Table 1 shows that for IPA+DMSO solution, the values remain unchanged, that is insignificant and that of for IPA+Water, pH value decreases with increase in V_{IPA} which is indicative of increasing acidic strength in the mixture.

Excess permittivity

Interaction among two constituents of liquid mixture is provided in terms of excess permittivity (ϵ^E) and is given as²⁸⁻²⁹,

$$\epsilon^E = (\epsilon')_m - [(\epsilon')_1V_1 + (\epsilon')_2(1 - V_1)] \quad \dots (8)$$

where, suffix m, 1 and 2 denote mixture, solute and solvent respectively, V_1 is volume fraction of solute.

As shown in Fig. 2, excess permittivity values for IPA+DMSO and IPA+Water binary mixtures are negative thereby indicating strong hydrogen bonded intermolecular interactions among the hetero molecules such that water and DMSO acts here as a structure breaker³⁰. Peak negative value for IPA+DMSO is at 0.6 and that of IPA+Water is at 0.2 V_{IPA} respectively. More negative values for IPA+DMSO indicates strong interactions while less negative value for IPA+Water reveals the less strong interaction than that of IPA+DMSO solutions.

Bruggeman factor

Information in terms of static permittivity of the mixture constituents is explored using Bruggeman factor, and is given by the relation as³¹,

$$f_B = \left[\frac{(\epsilon'_m - \epsilon'_1)}{(\epsilon'_2 - \epsilon'_1)} \right] \left[\frac{\epsilon'_2}{\epsilon'_m} \right]^{\frac{1}{3}} = 1 - V_2 \quad \dots (9)$$

Bruggeman equation has been modified for binary mixtures³² as,

$$f_B = \left[\frac{(\epsilon'_m - \epsilon'_1)}{(\epsilon'_2 - \epsilon'_1)} \right] \left[\frac{\epsilon'_2}{\epsilon'_m} \right]^{\frac{1}{3}} = 1 - [a - (a-1)V_2]V_2 \quad \dots (10)$$

where, f_B is Bruggeman factor, ϵ'_m , ϵ'_1 and ϵ'_2 represent static dielectric constant corresponding to mixture, solute and solvent, V_2 is volume fraction of solvent. In modified equation, V_2 is changed by a factor $[a - (a-1)V_2]$ of the mixture where 'a' is the interaction parameter.

The value of interaction parameter 'a' for IPA+DMSO systems is found to be 0.44, which suggests increased effective dipoles with strong intermolecular interactions³³ and for IPA+Water, it is

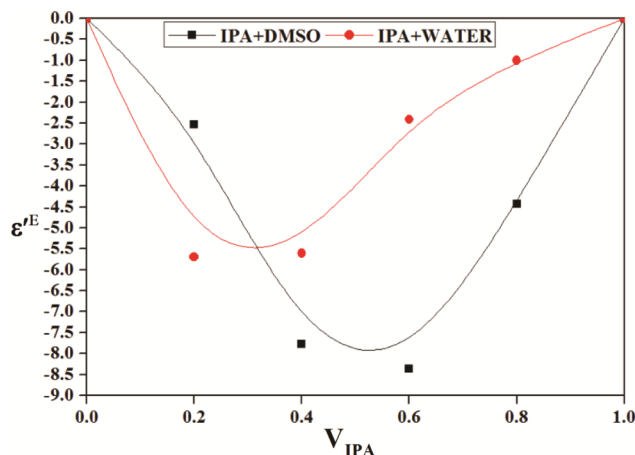


Fig. 2 — Variation of excess permittivity vs. V_{IPA} at room temperature.

1.43, with decreased dipoles with weak intermolecular interactions thereby suggesting decrease in effective volume fraction of IPA in Water³⁴ and intermolecular interactions are less strong than IPA+DMSO as compared in both solutions at room temperature, as shown in Fig. 3.

Kirkwood correlation factor

Orientation of dipoles, which measures the extent of association and correlation between the hetero molecules, thereby ensuring the hindrance in the rotation is explained by Kirkwood. Effective Kirkwood correlation factor g^{eff} is calculated from modified Kirkwood equation as³⁵⁻³⁶,

$$\frac{(\epsilon'_m - \epsilon'_{\infty m})(2\epsilon'_m + \epsilon'_{\infty m})}{\epsilon'_m(\epsilon'_{\infty m} + 2)^2} = \frac{4\pi N}{9kT} \left[\frac{\mu_1^2 \rho_1}{M_1} V_1 + \frac{\mu_2^2 \rho_2}{M_2} (1 - V_1) \right] \times g^{\text{eff}} \quad \dots (11)$$

where, g^{eff} , ϵ'_m , $\epsilon'_{\infty m}$, μ_1 , μ_2 , ρ_1 , ρ_2 , M_1 , M_2 , N , K , T has their usual meanings and V_1 is volume fraction of solute.

Dipole-dipole orientations in the solution of diverse molecules are effectively understood by using Kirkwood correlation factor (g^{eff})³⁷⁻³⁸. g^{eff} for IPA, DMSO and Water at room temperature is found to be 2.647, 1.199 and 2.875 respectively. For IPA+DMSO and IPA+Water solutions, $g^{\text{eff}} > 1$ at room temperature indicates the strong interactions with antiparallel orientation of electric dipoles among hetero molecules³⁸.

The value of g^{eff} increases with increase in V_{IPA} in IPA+DMSO solutions thereby indicating strong interaction while in IPA+Water solutions, the value of g^{eff} decreases with increase in V_{IPA} such that it confirms weak interactions in IPA+Water than that of

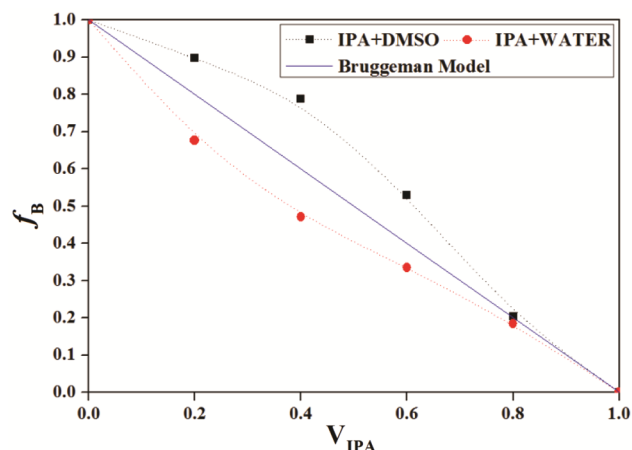


Fig. 3 — Variation of Bruggeman factor vs. V_{IPA} at room temperature.

IPA+DMSO solutions if the two systems are compared together³⁸.

The structuredness¹¹ or the ordering of molecules is studied from the Kirkwood correlation factor very well. In IPA+DMSO, the pure IPA and Water show structured nature while DMSO show unstructuredness or non-ordering of the molecules. The comparative study confirms the values of static dielectric constant and Kirkwood correlation factor, which are included in the Table 2 with orderedness of molecules. With increase in V_{IPA} , the structuredness of IPA molecule goes on increasing in case of IPA+DMSO while in case of IPA+Water, both are structured and ordering nature of Water is more than that of IPA, goes on decreasing.

g_1 and g_2 are the average correlation factors derived from a single value of ϵ' such that for separate class $i = 1$ and 2 and these values are obtained by using Luzar model with succeeding equations as⁴²⁻⁴³,

$$g_1 = 1 + Z_{11} \cos \Phi_{11} + Z_{12} \cos \Phi_{12} \left(\frac{\mu_2}{\mu_1} \right) \quad \dots (12)$$

$$g_2 = 1 + Z_{21} \cos \Phi_{21} \left(\frac{\mu_1}{\mu_2} \right) \quad \dots (13)$$

where, $Z_{11} = 2 \langle n_{HB}^{11} \rangle$, $Z_{12} = 2 \langle n_{HB}^{12} \rangle$ and $Z_{21} = 2 \langle n_{HB}^{21} \rangle (1-V_X)/V_X$ are average number of hydrogen bonds with solute-solute and solute-solvent pairs respectively. Φ_{11} , Φ_{12} and Φ_{21} show angles between neighboring dipoles of unlike molecules.

Average number of hydrogen bonds formed as n_{HB}^{11} and n_{HB}^{12} per solute molecule for $1i$ pairs ($i = 1$ or 2), have been obtained using the relation⁴²⁻⁴³,

$$n_{HB}^{1i} = n_{HB}^{1i} n_{1i} \omega^{1i} / n_1 \quad \dots (14)$$

where, $\omega^{1i} = 1/[1 + \alpha^{1i} \exp(\beta E^{1i})]$ represents probability of forming the bonds between solute and solvent molecule. n_1 is density of solute molecule, $\beta = 1/kT$, and α^{1i} are statistical volume ratios of hydrogen-bonded and non-hydrogen-bonded pairs related to two sub-volumes of phase space. Energy levels for 11 and 12 pairs are E^{11} and E^{12} . Density value causes variation in n_{HB}^{11} and n_{HB}^{12} of solute, solvent and solute-solute pair of molecules, respectively⁴⁴.

For two different binary solutions of IPA+DMSO and IPA+Water, values of g_1 and g_2 increases with increase in V_{IPA} , as represented in Fig. 4 (a) and (b). Increase in V_{IPA} causes decrease in bonding amongst IPA+DMSO and IPA+Water molecules such that n_{HB}^{11} value increases with decrease in n_{HB}^{12} , as shown in Fig. 5 (a) and (b), respectively⁴⁴⁻⁴⁵.

In IPA+DMSO system, ϵ' values obtained theoretically in comparison with experimental values found good as shown in Fig. 6 (a) and that of IPA+Water solutions, the values are nearly equal such that theoretical and experimental values are in better agreement as shown in Fig. 6 (b). The theoretical ϵ' values were obtained using feasible molecular

Table 2 — Comparison of literature values with the present study values for dielectric parameters.

Liquid	Parameters						
	Polarity Index ³⁹⁻⁴⁰	Literature values		Str./Unstr.	Present study		
		$\epsilon'^{18, 41}$	g^{11}		ϵ'	g	Str./Unstr.
IPA	3.9	19.13	3.08	Str.	15.45	2.64	Str.
DMSO	7.2	47.00	1.04	Unstr.	47.84	1.19	Unstr.
Water	10.2	78.36	2.57	Str.	78.78	2.87	Str.

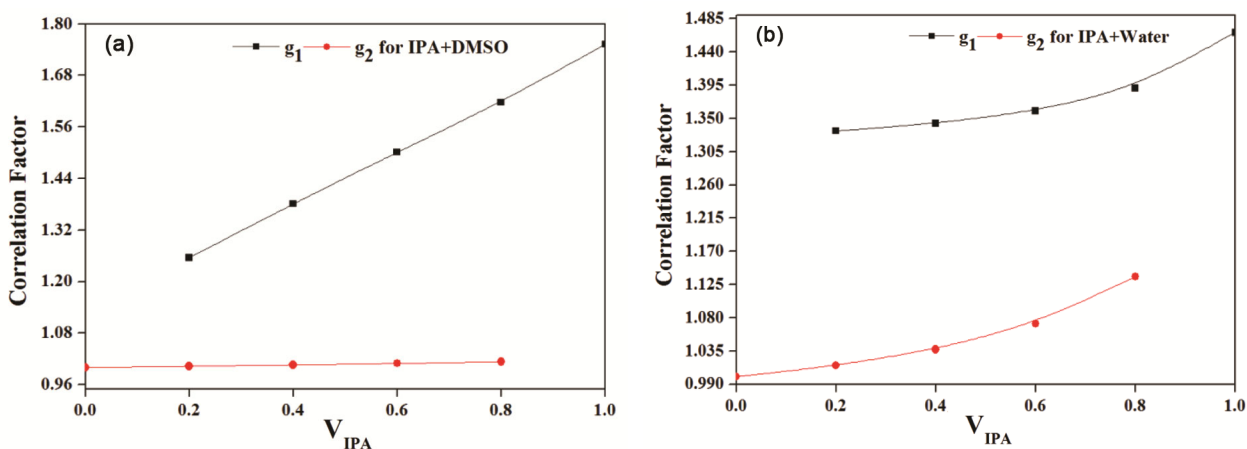


Fig. 4 — Variation of correlation factor (g_1 and g_2) vs. V_{IPA} for (a) IPA+DMSO and (b) IPA+Water solutions at room temperature.

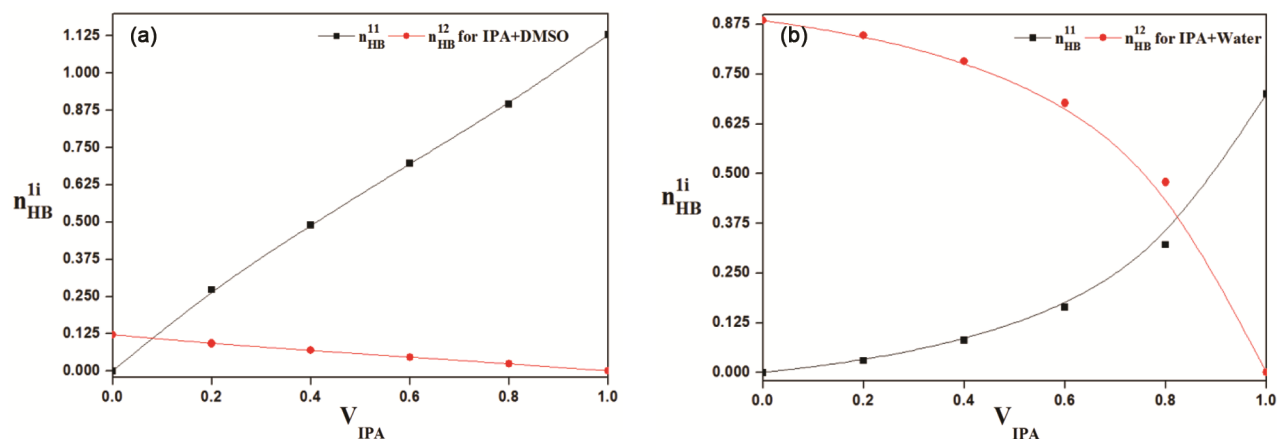


Fig. 5 — n_{HB}^{i1} vs. V_{IPA} for (a) IPA+DMSO and (b) IPA+Water solutions at room temperature.

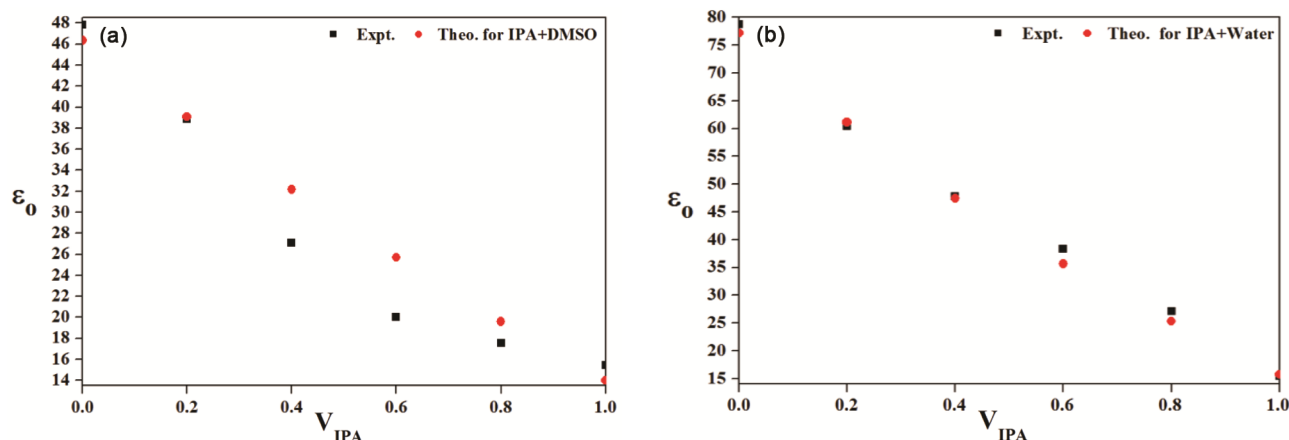


Fig. 6 — Theoretical and experimental ϵ' values vs. V_{IPA} for (a) IPA+DMSO and (b) IPA+Water solutions at room temperature.

Table 3 — Molecular parameters utilized in computation of ϵ' for IPA+DMSO and IPA+Water binary solutions at room temperature

Molecular Parameters for IPA+DMSO		Molecular Parameters for IPA+Water	
Effective Dipole Moment ^a of IPA	1.66	Effective Dipole Moment ^a of IPA	1.66
Effective Dipole Moment ^a of DMSO	3.96	Effective Dipole Moment ^a of Water	1.84
Polarizability ^b of IPA	9.85	Polarizability ^b of IPA	12.5
Polarizability ^b of DMSO	9.55	Polarizability ^b of Water	3.75
Bonding energy ^c of IPA-IPA	-9.6	Bonding energy ^c of IPA-IPA	-7
Bonding energy ^c of IPA-DMSO	-2.7	Bonding energy ^c of IPA-Water	-9
Enthalpy ^c of IPA-IPA	35	Enthalpy ^c of IPA-IPA	35
Enthalpy ^c of IPA-DMSO	45	Enthalpy ^c of IPA-Water	45
No. of Hydrogen Bonds	03	No. of Hydrogen Bonds	02

^aUnit: Debye; ^bUnit: A⁰³; ^cUnit: kJ/mol

parameters, as tabulated in Table 3 for IPA+DMSO and IPA+Water solutions, respectively.

Conclusions

Static dielectric permittivity values of both systems were determined using frequency domain technique at 9.685 GHz. The variation in concentration of IPA causes decrease in dielectric constant thereby confirming the variations in structural properties and

heteromolecular behavior such that presence of strong intermolecular forces cause change in structural arrangement, physical as well as intermolecular interactions, which follows the effect of density, polarity index on dielectric properties of IPA+DMSO and IPA+Water solutions. Kirkwood correlation factor and Bruggeman factor confirms antiparallel orientation, increased effective dipoles with strong intermolecular interactions. The structuredness of

these molecules is in the order as, DMSO < IPA < Water. Luzar model provides decent results such that theoretical static dielectric constant values are well in agreement with experimental values and it provides a worthy computational theoretical basis for prediction of formation of hydrogen bonding. For IPA+Water, increasing V_{IPA} indicate increasing acidic strength with decrease in pH value.

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