

Dielectric behavior of acetonitrile + n-Butyl alcohol binary mixtures at microwave frequency

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Abstract: Values of dielectric constant (ϵ') and dielectric loss (ϵ'') have been experimentally determined for binary liquid mixtures of acetonitrile + n-butyl alcohol at 10.75 GHz microwave frequencies at 30°C and over the complete mole fraction range. The values of ϵ' and ϵ'' have been used to evaluate the loss tangent ($\tan \delta$), molar polarization (P_{12}), apparent polarization (P_2) and a.c. conductivity (σ_p). The results are discussed in terms of intermolecular interactions. The results are positive over the entire range of composition.

Viscosity, density and refractive index measurement of pure liquid and binary liquid mixtures were carried out at 30°C. The values of viscosity have been used to evaluate the activation energy (E_a). The surface tension has been experimentally determined for pure liquid and binary liquid mixtures at 30°C. These parameters have been used to explain the formation of hydrogen bonding and formation of complex in the binary liquid mixtures.

Keywords: Binary mixture, Dielectric constant, dielectric loss, Molar polarization, Activation energy, Molecular interaction.

I. Introduction

Alcohols are industrially and scientifically important organic compounds and their physical and chemical properties are largely determined by -OH group. Alcohols are strongly associated in solutions because of dipole-dipole interaction and hydrogen bonding^{1,2,3}.

N-butyl alcohol is used as a direct solvent and as an intermediate in the manufacture of other organic chemicals (e.g. Butyl acrylate, methacrylate, glycol ethers and butyl acetate). Therefore it seemed important to examine the dielectric behavior of n-butyl alcohol with acetonitrile.

A dielectric investigation of solutions containing varying amounts of interacting molecules helps to detect the formation and composition of complexes in them⁴. A survey of the literature shows that a few workers have tried to investigate some binary systems taking nitriles as one of the constituent components in the binary mixtures. Though the information in this field is steadily being enlarged by a number of workers.⁵⁻⁹ the nature of complex formation in binary mixtures is still far from clear. With this in view, we report new experimental data dielectric constant (ϵ'), dielectric loss (ϵ''), a.c. conductivity (σ_p), loss tangent ($\tan \delta$), molar polarization (P_{12}), apparent polarization (P_2), activation energy (E_a). In this paper we also report the new experimental data of physical properties includes viscosity, density, surface tension and refractive index.

Knowledge of frequency dependent dielectric properties of binary liquid mixtures is important both in fundamental studies of solvent structure determination and its dynamics as well as in the practical applications. At fundamental level, the frequency dependent dielectric behavior of liquid mixtures provides information on molecular interactions and mechanisms of molecular process. In pharmaceutical and analytical sciences, the dielectric constant of mixed solvents is required to predict the solubility and chemical stability of the drug. The dielectric study of acetonitrile and n-butyl alcohol binary liquid mixtures using frequency domain reflectometry (FDR) have not been carried out in the past.

Hence, we felt that the present investigation which may provide useful information about the formation of complexes in the acetonitrile + n-butyl alcohol binary liquid mixtures at 30°C.

II. Experimental Details

The dissipation factor (D), dielectric constant (ϵ') and dielectric loss (ϵ'') were measured using Surber's technique^{10, 12} of measuring the reflection coefficient from the air-dielectric boundary of the liquid in the microwave X-band at 10.75 GHz frequency and at 30°C temperature.

The experimental setup is shown in figure 1. The dielectric closed cell has a movable short. To hold the liquid in the cell, a thin mica window, whose VSWR and attenuation were neglected, was introduced between the cell and the rest of the microwave bench. Here source of reflex klystron 2 K 25 (USSR) was used.

A plunger wave guide is converted into a cavity by introducing a coupling hole in the entrance and shorting the other end with the calibrated plunger. The sample occupies the entire volume of the cavity the frequency is kept constant and the length of the plunger cavity is changed. Hence, several nodes appear as one increase the length

of the cavity plunger, whenever the length of the cavity equals the half integral multiples of the guide wave length inside the medium. The plunger wave guide resonates the distance through which the plunger is moved between the successive cavity nodes gives half of the wave length (λ_d) of the microwave inside the medium.



Figure 1. The experimental setup of microwave X-band bench for the measurement of ϵ' and ϵ''

The measurement of reflected power at resonance gives the attenuation coefficient of the sample¹¹. Surber has derived the following relations for the dielectric parameters D, ϵ', ϵ''

$$D = \tan \left[2 \tan^{-1} \left(\frac{\alpha_d \lambda_d}{2\pi} \right) \right] \quad \dots\dots\dots (1)$$

$$\epsilon' = \left(\frac{\lambda_0}{\lambda_c} \right)^2 + \left(\frac{\lambda_0}{\lambda_d} \right)^2 \left[1 - \tan^2 \left(\frac{1}{2} \tan^{-1} D \right) \right] \quad \dots\dots\dots (2)$$

$$\epsilon'' = \frac{1}{\pi} \left(\frac{\lambda_0}{\lambda_d} \right)^2 \alpha_d \lambda_d \quad \dots\dots\dots (3)$$

Where D is the dissipation factor, α_d is the attenuation constant due to dielectric, λ_d is the wave length of the e.m. wave in the wave guide filled with the dielectric λ_0 is the free space wavelength, $\lambda_c = 2a$ is the cut off wavelength for the wave guide. $\alpha_d \lambda_d$ is the attenuation per wavelength. Having determined $\alpha_d \lambda_d, \lambda_0 \lambda_c$ and λ_d the values of D, ϵ', ϵ'' may be calculated by using the equations (1), (2) and (3) respectively.

The density of pure components and their mixtures were measured by using DMA 35 portable vibrating density meter, AntonPaar, Austria (Europe). The parts of enhanced ULA adapter : ULA-49 EAY water jacket, sample chamber, tube end cap ULA-34, ULA-31 EY, ULA-31 EYZ, clamping collar ULA-OZE of Brook field engineering laboratories USA and low temperature circulating water bath, Nivtech Instruments & Engineers, Thane, India at 30°C. Specification of density meter, AntonPaar Austria is accuracy 0.001 g/cm³ measuring range density 0 to 3 g/cm³. Temperature 0 to 40°C.

The refractive index of the pure components and their mixtures were measured by using Abbe's refractometer (with Glass scale) Mittal Enterprises, New Delhi, India, having an accuracy 0.001 by reading and 0.0001 by estimation. Measuring range extends from 1.300 to 1.700 with the help of sodium D line surface tension of pure components and their mixtures were measured by using Stalagmometer at room temperature 30°C.

Viscosity of pure components and their mixtures were measured by using viscometer Brook field DV-II + Pro model LVDV – II + P Brook field engineering laboratories, INC, USA, calibration of this instrument will be accurate to within $\pm 1\%$ of its full scale range.

Acetonitrile (AR grade) purity (GC) ≤ 10 . Identity IR supplied by Merck KGaA, Darm Stadt, Germany and n-Butyl alcohol (AR Grade) minimum assay (GC) 99.9%, Refractive index 1.399 to 1.400 boiling range 95% Supplied by SDFCL s d fine chem. Limited Mumbai-30, India were used without further purification. Acetone was used for rinsing laboratory glass ware and liquid cell

The solutions were prepared by mixing acetonitrile + n-butyl alcohol in volume. These binary liquid mixtures according to their proportions were mixed well and kept for 6 hours in a well stopper bottle to ensure

good thermal equilibrium. Microwave input and output power measured by Pm-437 (Attest) power meter, Chennai, India. Rectangular wave guide working Γ_{E10} mode 10dB, VidyutyantraUdyog, India.

Low temperature water circulating bath was used for maintaining temperature of pure components and their binary liquid mixtures for measurement of refractive index, density, viscosity and plunger reading using X-band microwave bench.

III. Results And Discussion

In recent years there has been considerable advancement in the theoretical and experimental investigation of the dielectric properties of binary liquid mixtures. Liquid mixtures exhibit various phenomenon's, which cannot be found in pure substances. The most interesting of these are perhaps the new types of phase equilibrium. Which arise from the extra degrees of freedom introduced by the possibility of varying the properties of the components. A limited number of studies have been reported for mixtures.

Dissipation factor, dielectric constant, dielectric loss, a.c.conductivity, loss tangent ($\tan \delta$), molar polarization (p_{12}), apparent polarization (P_2) and activation energy (E_a) for the viscous flow with increasing mole fraction (X_A) of Acetonitrile for the binary mixture. Acetonitrile + n-butyl alcohol are listed in Table 1. The values of viscosity (η), refractive index (n_D), density (ρ), surface tension (T), square of refractive index, mole fraction of solute and solvent are listed in Table 2.

The figure 2 shows the variation of the dielectric constant (ϵ') versus mole fraction of acetonitrile in the system of acetonitrile + n-butyl alcohol binary mixtures. There is an increasing trend according to P. Job if dielectric constant (ϵ') is plotted against the mole fraction for one of the components of a mixture the nature of the graph gives information regarding the occurrence of complex. If the relationship observed is linear, then there is no occurrence of complex.

On the other hand, if the two species are mixed and if complex form between the two species the value by additive property will pass through a maxima or minima the complex is at its greatest concentration at a point where the species are combined in the ratio in which they occurs in the complex. The curve of dielectric constant against mole fraction therefore shows a change in slope at the mole fraction corresponding to the complex. If the change in slope occurs at a mole fraction of 0.5 then it indicates a complex of the 1:1 type and if the change in slope occurs at a mole fraction of 0.7 then it indicates a complex of the 2:1 type. In figure 2. The graph shows deviation from linearity, indicating complex formation in the mixture as suggested by P. Job. The deviation is maximum at about 0.535343 mole fraction of acetonitrile.

Hence, complex is at its greatest concentration at point $X_A=0.535343$ indicating the formation of 1:1 complex in the binary. This curve suggest that there is occurrence of intermolecular interaction between solute and solvent¹² same behavior obtained^{1,15, 16}. It is observed that the dielectric constant (ϵ') increases with increasing mole fraction of acetonitrile in the binary liquid mixtures of Acetonitrile+ n-butyl alcohol. Similar values of ϵ' for pure n-butyl alcohol is obtained^{13,14}.

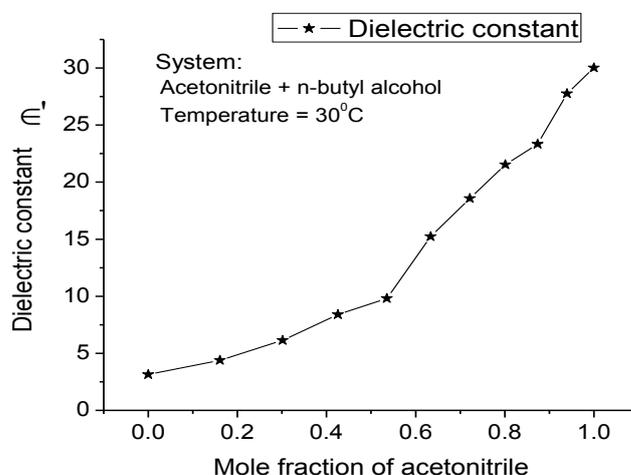


Figure 2: Variation of ϵ' versus mole fraction of Acetonitrile in the mixture

Loss tangent ($\tan \delta$):

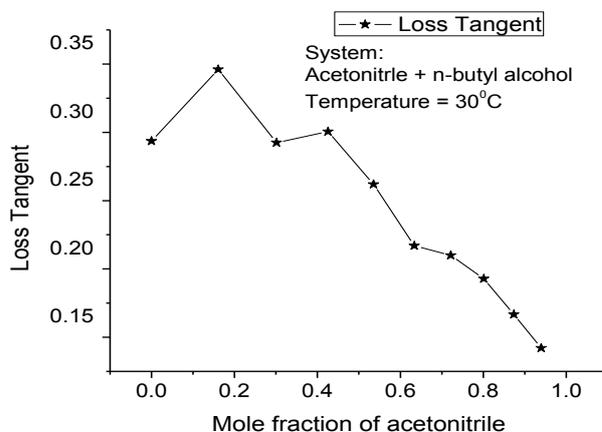


Figure3. Variation of Loss Tangent ($\tan \delta$) versus Mole fraction of acetonitrile in the mixture

It is seen from figure 3, that the microwave energy absorption in the mixture is greater than that in pure liquids, a maxima in $\tan \delta$ cure occurring at $X_A=0.301699$ mole fraction of Acetonitrile in the Acetonitrile + n-butyl alcohol mixtures An interaction causing association between two types of molecules may be responsible for nonlinear behavior. Similar result was obtained.¹⁴

Molar polarization:

The values of molar polarization P_{12} of the mixtures were obtained by the formula.

$$P_{12} = \frac{(\epsilon' - 1)(X_A M_1 + X_B M_2)}{(\epsilon' + 2)\rho} \dots\dots\dots (4)$$

Where M_1 and M_2 are the molecular weights. X_A and X_B are the mole fraction of solute (Acetonitrile) and solvent (n-butyl alcohol) respectively and ρ is the density of the mixture. (ϵ') dielectric constant of the binary liquid mixtures.

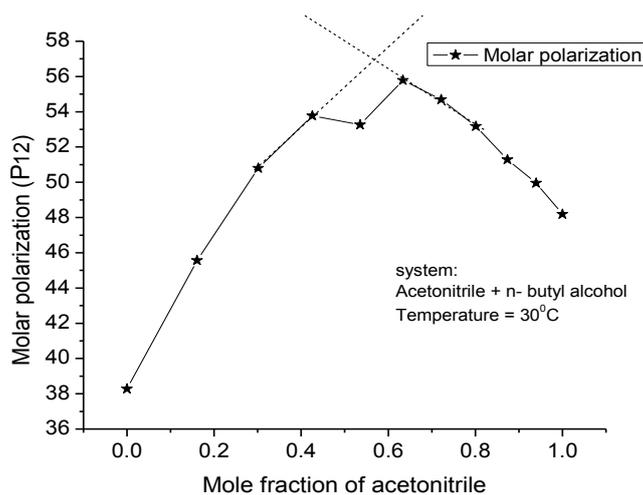


Figure 4. Variation of Molar Polarization (P_{12}) versus Mole fraction of acetonitrile in the mixture

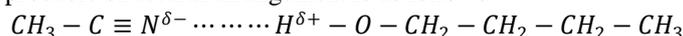
Solvent – Solvent interaction between types of polar protic – polar aprotic solvent is due to hydrogen bonding formation as a result of amphiprotic hydrogen bond acceptor-donor (HBA-D). These solvent-solvent interaction property is depends mainly on different physical properties of solvent such as dielectric constant, dipole moment, molar polarization (P_{12}), donor number, chemical structure solvatochromic quantitative values of Kamlet Taft hydrogen bond acidity, basicity and dipolarity – Polarizability⁸.

In present investigation the concentration of acetonitrile in the system increases the dielectric constant of the system(acetonitrile + n-butyl alcohol) increases with molar polarization. It is due to the formation of KamletTalfhydrogen bonding between Acetonitrile and n-butyl alcohol binary system.

We may interpret figure 4 as, the intersection of the dotted straight line ideally representing the point of maximum concentration of complex. The point of intersection for the system occurs at about $X_A = 0.55$ mole fraction of Acetonitrile which corresponds to 1:1 complex for the system.

Thus this result regarding the formation of complex is supported by our earlier conclusion made from the ϵ' versus mole fraction of acetonitrile curve for the same system. Similar result have been obtained.¹⁴

This is due to formation of hydrogen bonding and this occurred due to increasing the concentration of more polar solvent. The probable structural arrangement is as follows:



Apparent polarization (P_2):

Apparent polarization (P_2) of the mixtures were obtained by the formula

$$P_2 = \frac{P_{12} - X_A P_1}{X_B} \dots \dots \dots (5)$$

Where P_{12} is the molar polarization of the mixtures, P_1 is molar polarization of pure solvent, X_A and X_B are mole fraction of solute and solvent respectively.

The graph of apparent polarization (P_2) versus mole fraction (X_A) of acetonitrile is depicted in figure 5. The graph indicates that apparent polarization decreases with increasing mole fraction (X_A) of acetonitrile in the mixture.

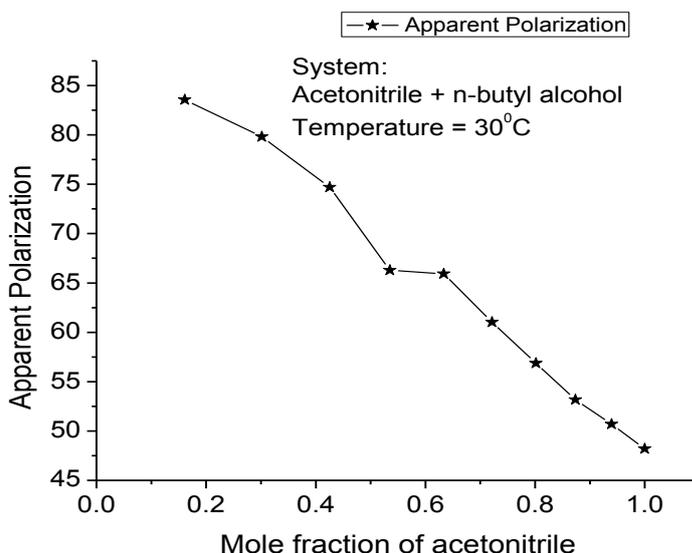


Figure5. Variation of apparent polarization (P_2) versus Mole fraction of acetonitrile in the mixture

a.c. Conductivity (σ_p):

a.c. conductivity of the mixtures were obtained by using the formula

$$\sigma_p = \omega \epsilon_0 \epsilon'' \dots \dots \dots (6)$$

$$\sigma_p = 2\pi f \epsilon_0 \epsilon'' \dots \dots \dots (7)$$

Where $\omega = 2\pi f$, $f = 10.75$ GHz , $\epsilon_0 = 8.854187816 \times 10^{-12} C^2 / Jm$

ϵ'' = Dielectric loss of binary liquid mixture

In our present work, it is found that a.c. conductivity depends on dielectric loss (ϵ''). Dielectric loss increases a.c. conductivity increases and dielectric loss decreases a.c. conductivity decreases.

Dielectric loss is proportional to the a.c. conductivity. Thus a.c. conductivity depends upon the dielectric loss¹⁷.a.c conductivity and dielectric loss is presented in figure (6) and (7) respectively.

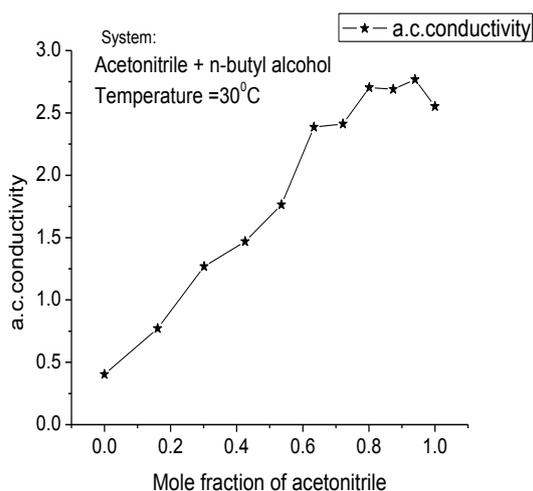


Figure6. Variation of a.c. conductivity (σ) Versus Mole fraction of acetonitrile in the mixture

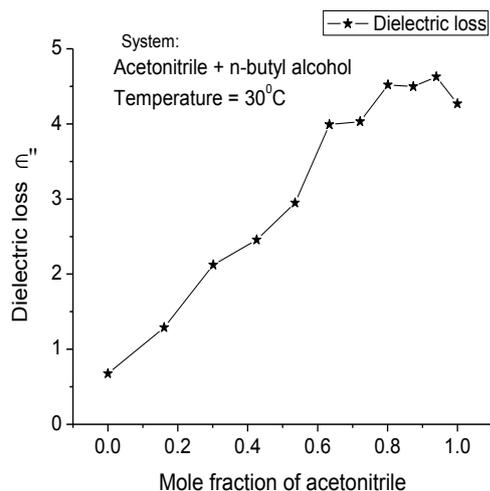


Figure7. Variation of Dielectric loss (ϵ'') versus Mole fraction of acetonitrile in the mixture

Refractive index (n_D):

In our present work it is clear from figure (8) that the refractive index values are decreasing with increasing concentration of acetonitrile in the binary liquid mixture of acetonitrile + n-butyl alcohol. Similar value of refractive index for acetonitrile was obtained⁶. Similar value of refractive index of n-butyl alcohol was obtained.¹⁴

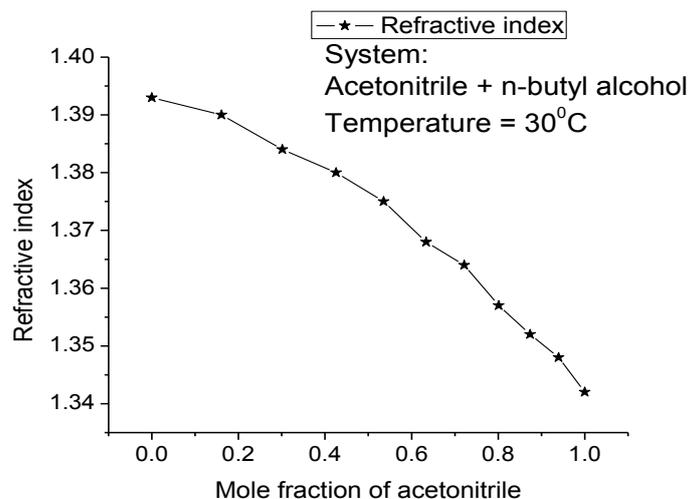


Figure8. Variation of Refractive index (n_D) versus Mole fraction of acetonitrile in the mixture

Viscosity (η):

In figure (9) the graph shows the viscosity decreases nonlinearly with increasing mole fraction of acetonitrile. It was decreased rapidly up to 0.31699 mole fraction of acetonitrile in the system of acetonitrile + n-butyl alcohol at 30°C temperature.

Kenneth Hickey et.al.¹⁸ reported similar result. PatricNgoyTshibangu et.al.¹⁹ reported ionic liquid viscosity is ordinarily influenced by other interactions such as hydrogen bonding and the symmetry of the ions. Similar decreasing trend is obtained^{20,21} weak types of dipole induced dipole of interactions are not sufficient to product bulky or less mobile entities in system and hence decreased trend of viscosity is observed in the present binary liquid mixtures of acetonitrile + n-butyl alcohol.

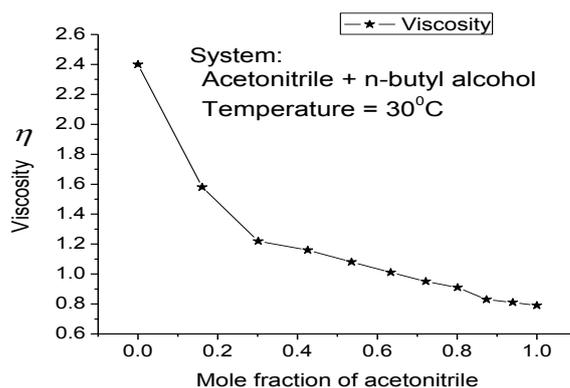


Figure9. Variation of Viscosity (η) versus Mole fraction of acetonitrile in the mixture

Density (ρ):

Figure (10) shows the density measurement curve. The values of density of binary liquid mixtures decreases with increasing mole fraction of acetonitrile in the binary liquid mixture of acetonitrile + n-butyl alcohol. Similar decreased trends of density is obtained.^{1,21,22,23,24} Similar value of density of pure acetonitrile is obtained.^{6,7}

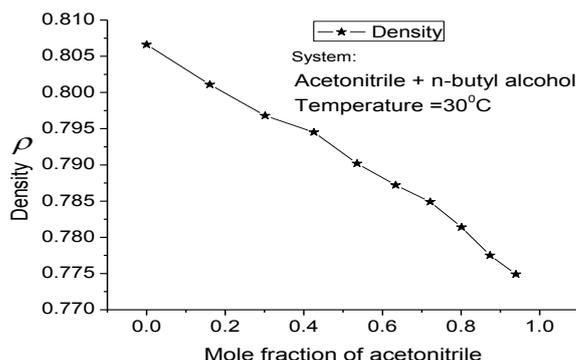


Figure10. Variation of Density (ρ) versus Mole fraction of acetonitrile in the mixture

Surface Tension(T):

The values of mole fraction of acetonitrile versus surface tension of binary liquid mixture of acetonitrile and n-butyl alcohol are presented in figure (11). Surface tension is nonlinearly increases with increasing mole fraction of acetonitrile. Similar nature of graph is obtained.²⁷

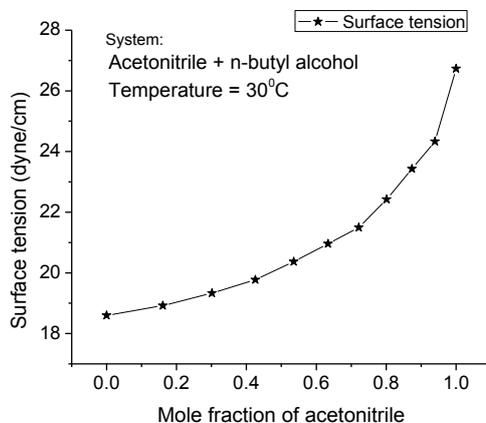


Figure11. Variation of Surface Tension (T) versus Mole fraction of acetonitrile in the mixture

As it can be seen from figure (11), surface tension (T) is positive for the binary mixture of (acetonitrile + n-butyl alcohol) the strong interaction between acetonitrile + n-butyl alcohol give rise to an increase in surface

tension. This is in turn responsible for the positive surface tension (T) for the (Acetonitrile + n-butyl alcohol) system.²³

Table No.1. Mole Fraction of Solute (X_A), Dissipation factor (D), Dielectric Constant (ϵ'), Dielectric loss (ϵ''), Loss Tangent ($\tan \delta$), Molar Polarization (P_{12}), a.c. Conductivity (σ_p), apparent Polarization (P_2) and Activation Energy (Ea) of Binary Liquid Mixtures at 30°C

S R	X_A	D	ϵ'	ϵ''	$\tan \delta$	P_{12}	σ_p	P_2	Ea
1	0	0.243806	3.141077	0.675023	0.214902	38.270554	0.403697	----	6.251771
2	0.161088	0.320796	4.391134	1.289196	0.293591	45.565053	0.771003	83.55325	5.995019
3	0.301699	0.368416	6.130999	2.121565	0.346039	50.800065	1.268801	79.800393	5.839301
4	0.425502	0.306066	8.39396	2.45513	0.292488	53.766534	1.468289	74.688667	5.80893
5	0.535343	0.312338	9.815365	2.949397	0.300488	53.26792	1.763885	66.285053	5.765896
6	0.633456	0.268477	15.238049	3.991094	0.261916	55.79071	2.386871	65.928601	5.725541
7	0.721625	0.221458	18.575775	4.031288	0.217019	54.69335	2.410909	61.028629	5.688659
8	0.801289	0.21361	21.52745	4.518931	0.209915	53.173047	2.702544	56.868704	5.662753
9	0.873622	0.195939	23.325525	4.497414	0.192811	51.281985	2.689676	53.164216	5.607338
10	0.93959	0.168976	27.754148	4.626864	0.166709	49.951607	2.767093	50.702629	5.592649
11	1	0.143943	30.013627	4.266641	0.142157	48.190674	2.551662	48.190674	5.577592

Activation Energy (Ea):

In chemistry activation energy is term introduced in 1889 by the Swedish scientist Arrhenius that is defined as the minimum energy that must be in put to a chemical system, containing potential reactants, in order for a chemical reaction to occur, Activation energy may also be defined as minimum energy required to start a chemical reaction. The activation energy of a reaction is usually denoted by Ea. Activation energy can be thought of as the height of the potential barrier (sometimes called the energy barrier) separating two minima of potential energy of the reactants and products of a reaction. For a chemical reaction to proceed at a reasonable rate, there should exit an appreciable number of molecules with energy equal to or greater than the activation energy. Activation energy depends upon the nature of the reaction fast reaction usually have a small Ea those with a large Ea usually proceed slowly²⁶. The graph of Activation energy (Ea) versus mole fraction (X_A) of Acetonitrile is presented in figure (12). We observed that activation energy decreases with increasing mole fraction of Acetonitrile in the mixture. Same nature of graph is obtained.²⁵

Table: 2. Mole fraction of solute (X_A), Mole Fraction of Solvent (X_B), Density (ρ), Viscosity (η), Refractive Index (n_D), Square of Refractive Index ($(n_D)^2$) and Surface Tension (T) of Binary Liquid Mixtures at 30°C

Sr. no.	X_A	X_B	ρ gm/cm ³	η cP	n_D	$(n_D)^2$	T dyne/cm
1	0	1	0.8066	2.4	1.393	1.940449	18.599529
2	0.161088	0.838912	0.8011	1.58	1.39	1.9321	18.924089
3	0.301699	0.698301	0.7968	1.22	1.384	1.915456	19.332894
4	0.425502	0.574498	0.7945	1.16	1.38	1.9044	19.770798
5	0.535343	0.464657	0.7902	1.08	1.375	1.890625	20.367215
6	0.633456	0.366544	0.7872	1.01	1.368	1.871424	20.963967
7	0.721625	0.278375	0.7849	0.95	1.364	1.860496	21.4979
8	0.801289	0.198711	0.7814	0.91	1.357	1.841449	22.420257
9	0.873622	0.126378	0.7775	0.83	1.352	1.827904	23.430082
10	0.93959	0.06041	0.7749	0.81	1.348	1.817104	24.330418
11	1	0	0.772	0.79	1.342	1.800964	26.731381

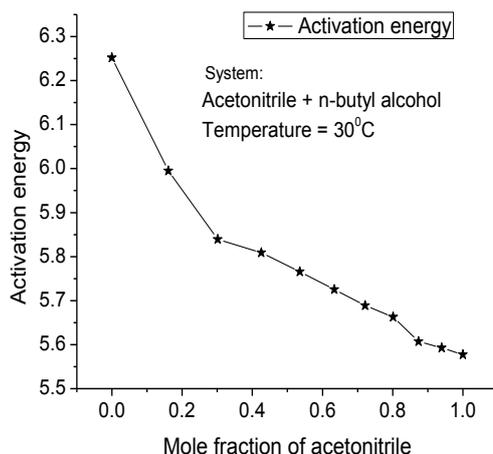


Figure12. Variation of Activation Energy (E_a) versus Mole fraction of acetonitrile in the mixture

IV. Conclusions

The values of dielectric constant (ϵ'), Dielectric loss (ϵ''), loss tangent ($\tan \delta$), molar polarization (P_{12}), Apparent polarization (P_2), activation energy (E_a) and viscosity, refractive index, square of refractive index, surface tension and density are all positive values indicating strong interactions between acetonitrile + n-butyl alcohol molecules. Loss tangent curve indicates large microwave energy absorption occurred at $X_A=0.301699$ mole fraction of acetonitrile. We also investigated dielectric loss is proportional to a.c. conductivity of binary liquid mixtures.

There is intermolecular interaction among the components of the binary mixtures leading to hydrogen bond formation. Dielectric constant curve indicates 1:1 complex and molar polarization curve indicates 1:1 complex in the binary liquid mixtures (Acetonitrile + n-butyl alcohol).

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