# Dielectric behavior of acetonitrile + n-Butyl alcohol binary mixtures at microwave frequency at 20°C

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#### Abstract:

Values of dielectric constant ( $\epsilon'$ ) and dielectric loss ( $\epsilon''$ ) have been experimentally determined for binary liquid mixtures of acetonitrile with n-butyl alcohol at 10.75 GHz microwave frequencies at 20°C and over the complete mole fraction range. The values of  $(\epsilon')$  and  $(\epsilon'')$  have been used to evaluate the loss tangent (tan  $\delta$ ), molar polarization (P<sub>12</sub>), and a.c. conductivity ( $\sigma p$ ). The results are discussed in terms of intermolecular interactions. It has been shown that the results are positive over the whole range of composition.

Viscosity, density and refractive index measurement of pure liquid and binary liquid mixtures were carried out at 20°C. The values of viscosity have been used to evaluate the activation energy (Ea). 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, a.c.conductivity, Molar polarization, Activation Energy, viscosity, density, refractive index.

## 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<sup>1, 2</sup>

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 them<sup>1</sup>. 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. 1, 3-7

The nature of complex formation in binary mixtures is still far from clear. With this in view, we report new experimental data dielectric constant ( $\epsilon'$ ), dielectric loss ( $\epsilon''$ ), a.c. conductivity ( $\sigma_D$ ), loss tangent ( $\tan \delta$ ), molar polarization (P<sub>12</sub>), activation energy (Ea). In this paper we also report the new experimental data of physical properties includes viscosity, density 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 nature of 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 at 20°C 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  $20^{\circ}$ C.

#### II. EXPERIMENTAL DETAILS

The dissipation factor (D), dielectric constant ( $\varepsilon'$ ) and dielectric loss ( $\varepsilon''$ ) were measured using Surber's technique<sup>8-10</sup> 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 20°C temperature. The experimental setup is shown in figure 1. was used to measure wavelength ( $\lambda d$ ). 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 (  $\lambda d$ ) of the microwave inside the medium.

The measurement of reflected power at resonance gives the attenuation coefficient of the sample 11. Surber has derived the following relations for the dielectric parameters D,  $\varepsilon'$ ,  $\varepsilon''$ 

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

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

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

Where D is the dissipation factor,  $\alpha_d$  is the attenuation constant due to dielectric,  $\lambda_d$  is the wave length of the e.m. wave in the wave guide filled with the dielectric  $\lambda_0$  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  $\lambda_d$  the values of D,  $\epsilon'$ ,  $\epsilon''$  may be calculated by using the equations (1), (2) and (3) respectively.

The densities of binary mixtures of acetonitrile with n-butyl alcohol as well as pure compound were measured at 20°C 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. Specification of density meter, AntonPaar Austria is accuracy 0.001 g/cm<sup>3</sup> measuring range density 0 to 3 g/cm<sup>3</sup>. Temperature 0 to  $40^{0}$ C.

The refractive index of the pure components and their mixtures were measured at 20°C 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.

Viscosity of pure components and their mixtures were measured at 20°C 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 was purchased from Merck KGaA, Darn Stadt, Germany and n-Butyl alcohol (AR Grade) minimum assay (GC) 99.9%, Refractive index 1.399 to 1.400 boiling range 95% was purchased from 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.

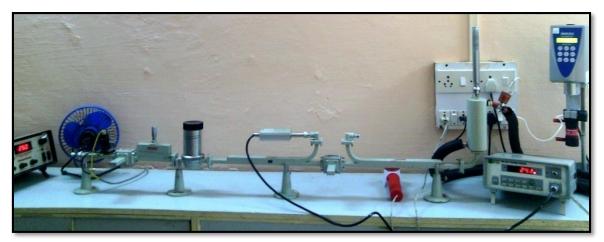


Figure 1. The experimental setup of microwave X-band bench for the measurement of  $\mathcal{E}'$  and  $\mathcal{E}''$ 

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  $\Gamma_{E10}$  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 RESULT AND DISCUSSIONS

Currently, there has been considerable advancement in the theoretical and experimental investigation of the dielectric properties of binary system. Liquid mixtures exhibit various phenomenons', which cannot be found in pure substances. The most interesting of the new types of phase equilibrium that arises from the additional quantity of freedom introduced by the possibility of varying the properties of the components. A limited number of studies have been reported for mixtures.

Dielectric constant, tangent of the loss angle (dissipation factor (D)), dielectric loss, a.c. conductivity, loss tangent, molar polarization ( $P_{12}$ ) and activation energy ( $E_a$ ) for the viscous flow with increasing mole fraction (X<sub>A</sub>) of acetonitrile for the binary mixture (C<sub>2</sub>H<sub>3</sub>N and C<sub>4</sub>H<sub>9</sub>OH) are presented in Table (1).The values of viscosity  $(\eta)$ , refractive index  $(n_D)$ , density, mole fraction of solute and solvent are listed in Table (2) at 20°C temperature and at 10.75GHz microwave frequency.

#### **Dielectric constant**

The figure 2 shows the variation of the dielectric constant  $\mathcal{E}'$  versus mole fraction of acetonitrile in the system of acetonitrile + n-butyl alcohol binary mixtures at 20°C temperature. There is an increasing trend according to P. Job, if dielectric constant  $\mathcal{E}'$  is plotted against the mole fraction for one of the components of a mixture the nature of the graph provides information about the occurrence of complexion. If the relationship observed is linear, then there is no occurrence of complexion.

On other way, in case of two species are mixed and if complex form between the two species the value by additive property will go through a minima or maxima the complex is at its greatest concentration at a point where the species are joined in the ratio in which they take place in the complex. The curve of dielectric constant against mole fraction so, gives 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 shows a complex of the 1:1 type and if the change in slope occurs at a mole fraction of 0.7 then it shows a complex of the 2:1 type. In figure 2 the graph shows deviation from linearity, indicating complex formation in the mixture as said by P.Job. The deviation is maximum at about  $X_A$ = 0.425502 mole fraction of acetonitrile at 20°C temperature.

**Table 1.** Mole fraction of solute  $(X_A)$ , dissipation factor (D), dielectric constant  $(\varepsilon')$ , dielectric loss  $(\varepsilon'')$ , loss tangent (tan  $\delta$ ), molar polarization ( $P_{12}$ ), a.c. conductivity ( $\sigma_p$ ) and activation energy ( $E_a$ ) of binary liquid mixtures of acetonitrile and n-butyl alcohol at 20°C

Sr.	X <sub>A</sub>	D	ε'	ε"	tan δ	P <sub>12</sub>	$\sigma_{p}$	Ea
1	0	0.194961	3.238147	0.558712	0.172541	39.051243	0.3341373	6.18351
2	0.161088	0.25469	4.424912	1.032138	0.233256	45.313153	0.61727	5.899606
3	0.301699	0.304134	6.222704	1.779282	0.285934	50.604193	1.064099	5.731059
4	0.425502	0.317809	8.41326	2.555465	0.303743	53.297138	1.528294	5.614199
5	0.535343	0.328236	12.373534	3.93921	0.318358	55.886051	2.355842	5.56217
6	0.633456	0.251885	15.807519	3.887885	0.245952	55.476003	2.325147	5.551182
7	0.721625	0.236269	19.23619	4.456934	0.231695	54.319301	2.665467	5.511004
8	0.801289	0.270431	21.556857	5.728929	0.265759	52.570524	3.426183	5.474212
9	0.873622	0.23741	24.30377	5.681557	0.233773	50.851524	3.397852	5.461413
10	0.93959	0.167912	27.891053	4.620702	0.16567	49.233892	2.763408	5.407202
11	1	0.154823	30.594717	4.679107	0.152938	47.619497	2.798337	5.355239

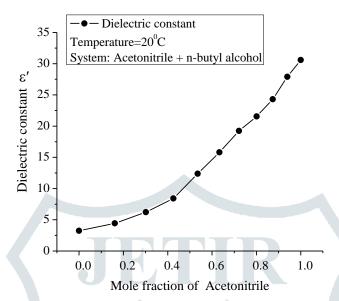
**Table 2.**Mole fraction of solute  $(X_A)$ , mole factor of solvent  $(X_B)$ , density (d), viscosity  $(\eta)$ , refractive index  $(n_D)$ , of binary liquid mixture of acetonitrile and n-butyl alcohol at 20°C

Sr. no.	$X_A$	$X_{B}$	d gm/cm³	η cP	$n_D$
1	0	1	0.811	3.11	1.395
2	0.161088	0.838912	0.8093	1.91	1.394
3	0.301699	0.698301	0.8051	1.43	1.388
4	0.425502	0.574498	0.8021	1.17	1.384
5	0.535343	0.464657	0.7988	1.07	1.377
6	0.633456	0.366544	0.797	1.05	1.37
7	0.721625	0.278375	0.7945	0.98	1.366
8	0.801289	0.198711	0.7905	0.92	1.361
9	0.873622	0.126378	0.788	0.9	1.358
10	0.93959	0.06041	0.7866	0.82	1.351
11	1	0	0.7827	0.75	1.345

$$CH_{3}-CH_{2}-CH_{2}-CH_{2}-O-\overset{\delta^{+}}{H}\cdots\overset{\delta^{-}}{N}\equiv C-CH_{3}$$
 Hydrogen bonding interaction

H-bonding was formed between the H of alcoholic group of n-butyl alcohol and nitrogen of acetonitrile at temperature 20°C

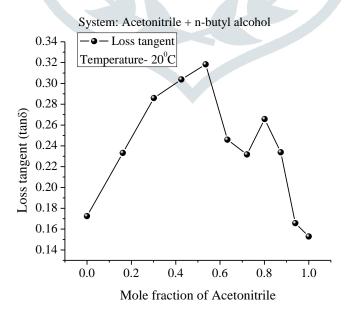
Hence, complex is at its maximum concentration at point  $X_A=0.425502$  indicating the formation of 1:1 complex in the binary. From graph it is clear that, there is strong intermolecular interaction takes place between solute and solvent [10]. Same behavior obtained [11, 12]. It is observed that the dielectric constant  $\varepsilon'$  increases with increasing mole fraction of acetonitrile in the binary system (4). Similar value of  $\varepsilon'$  for pure n-butyl alcohol is obtained [13, 14].



**FIGURE 2** Variation of dielectric constant (ε') versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at 20°C temperature

# Loss tangent $(\tan \delta)$

From figure 3, that the microwave energy absorption in the mixture is greater than that in pure liquids, a maxima in  $\tan \delta$  curve taking place at  $X_A$ = 0.535343 mole fraction of acetonitrile at 20°C in the acetonitrile and n-butyl alcohol mixtures. An interaction causing association between two kinds of molecules is responsible for nonlinear behavior of curve. Similar kind of result was obtained [14].



**FIGURE 3** Variation of loss tangent  $(\tan \delta)$  versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at  $20^{\circ}$ C temperature

## Molar polarization

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

$$P_{12} = \frac{\left(\varepsilon' - 1\right)}{\left(\varepsilon' + 2\right)} \frac{\left(X_A M_1 + X_B M_2\right)}{d}$$

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 d is the density of the mixture.  $\mathcal{E}'$  dielectric constant of the binary liquid mixtures.

Solvent – Solvent interaction between kinds of polar protic – polar aprotic solvent is due to hydrogen bonding formation as a result of amphiprotic hydrogen bond acceptor-donor. These solvent-solvent interaction property is depends mainly on various physical properties of solvent such as dielectric constant(  $\mathcal{E}'$ ), dipole moment, molar polarization, donor number, chemical structure solvatochromic quantitative values of Kamlet Taft hydrogen bond acidity, basicity and dipolarity – Polarizability [15].

Current investigation the concentration of acetonitrile in the system increases the dielectric constant  $(\mathcal{E}')$  of the acetonitrile and n-butyl alcohol system increases with molar polarization. It is due to the formation hydrogen bonding between acetonitrile and n-butyl alcohol molecules.

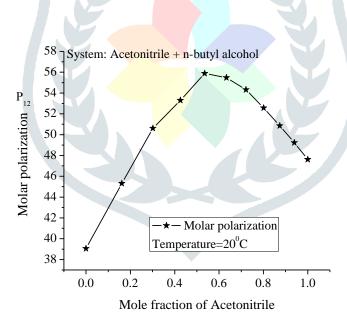


FIGURE 4 Variation of molar polarization versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at 20°C temperature

We may interpret figure 4 as the point of intersection for the system occurs at  $X_A = 0.535343$  mole fraction of acetonitrile at temperature 20°C

This corresponds to 1:1 complex in the system. Thus this result about the formation of complex is supported by our earlier conclusion made from figure 2. Similar result has been obtained [14]. This is due to formation of hydrogen bonding and this taken place due to increasing the more concentration of polar solvent. The probable structural arrangement is as follows:

$$CH_3-C\equiv N^{\delta-}\cdots\cdots H^{\delta+}-O-CH_2-CH_2-CH_2-CH_3$$

Hydrogen bonding interaction

# A.c. Conductivity $(\sigma_p)$

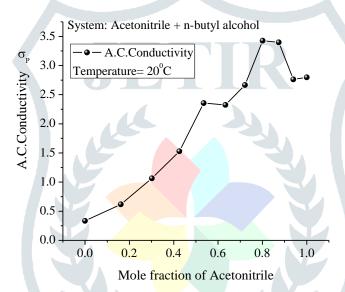
a.c. conductivity of the mixtures were calculated by using the formula

$$\sigma_p = \omega \varepsilon_0 \varepsilon$$
"

$$\sigma_p = 2\pi f \, \varepsilon_0 \varepsilon$$
"

Where  $\omega = 2\pi f$ , f = 10.75 GHz,  $\varepsilon_0 = 8.854187816 \times 10^{-12} \text{ C}^2 / \text{Jm}$ 

 $\varepsilon$ " = Dielectric loss of binary liquid mixture



**FIGURE 5** Variation of a.c. conductivity ( $\sigma_p$ ) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at 20°C temperature

In this work, it is found that a.c. conductivity depends on dielectric loss ( $\epsilon''$ ). Dielectric loss gets increases as a.c. conductivity increases. Dielectric loss decreases as a.c. conductivity also get decreases. Hence, Dielectric loss is proportional to the a.c. conductivity. Therefore, a.c. conductivity depends upon the dielectric loss [16]. a.c conductivity and dielectric loss is presented in figure (5) and (6) respectively.

#### **Dielectric loss**

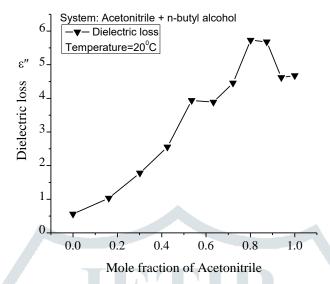


FIGURE 6 Variation of dielectric loss (ε") versus mole fraction of acetonitrile in the mixture at 20°C temperature **Refractive index (nD)** 

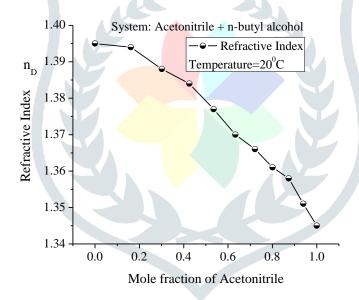


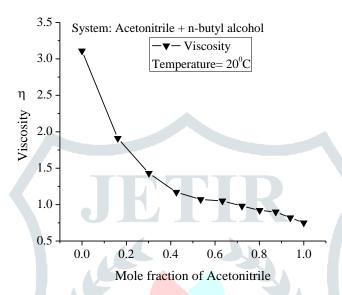
FIGURE 7 Variation of refractive index (n<sub>D</sub>) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at 20°C temperature

In this work, from figure (7) it is clear that, the refractive index values are decreasing with increasing concentration of acetonitrile in the binary liquid mixture at temperature 20°C. Similar value of refractive index for acetonitrile was obtained [17]. Similar value of refractive index of n-butyl alcohol was obtained [14].

## Viscosity (η)

In figure (8) the graph shows the viscosity decreases nonlinearly with increasing mole fraction of acetonitrile. It was decreased quickly up to 0.31699 mole fraction of acetonitrile at temperature 20°C then slowly decreases in the system of acetonitrile +n-butyl alcohol.

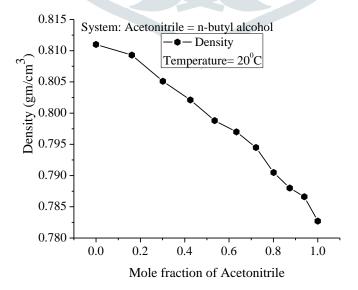
Kenneth Hickey et.al [18] reported similar result. Patric Ngoy Tshibangu et al. [19] reported ionic liquid viscosity is ordinarily influenced by another interaction such as hydrogen bonding and the symmetry of the ions. Similar decreasing trend is obtained [20, 21]. Weak types of dipole induced dipole of interactions are not sufficient to product bulky or less movable entities in system and hence decreased trend of viscosity is observed in the present binary liquid mixtures of acetonitrile + n-butyl alcohol.



**FIGURE 8** Variation of viscosity  $(\eta)$  versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at 20°C temperature

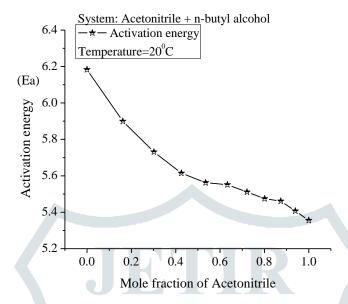
#### Density (d)

Figure (9) shows the density measurement curve. The values of density of binary liquid mixtures decrease with increasing mole fraction of acetonitrile at 20°C temperature in the binary liquid mixture of acetonitrile + n-butyl alcohol. Similar trends of density are obtained [12, 21, 22, 23, 24]. Similar value of density of pure acetonitrile is obtained [14, 22].



**FIGURE 9** Variation of density (d) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at 20°C temperature

## Activation Energy $(E_a)$



**FIGURE 10** Variation of activation of energy  $(E_a)$  versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at 20°C temperature

In chemistry the term activation energy is introduced by the Swedish scientist Arrhenius. It is defined as the minimum energy that must be given to a chemical system, containing potential reactants, in order for a chemical reaction to take place, in other way, Activation energy is defined as minimum energy required to start a chemical reaction. The activation energy is generally denoted by  $E_a$ . Activation energy can be thought of as the height of the energy barrier separating 2 minima of potential energy of the reactants and products of a reaction. For a chemical reaction to continue at a reasonable rate, there should exit an appreciable number of molecules with energy greater than or equal to the activation energy. Activation energy depends upon the nature of the reaction fast reaction generally represent by a small  $E_a$  and slow reaction with a large E<sub>a</sub>. The graph of Activation energy versus mole fraction (X<sub>A</sub>) of Acetonitrile in the system is presented in figure (10). We observed that activation energy decreases with increasing molar concentration of acetonitrile in the mixture. Same nature of graph is obtained [25] at 20°C temperature.

## **Conclusions**

In this paper the values of dielectric constant, dielectric loss, molar polarization, a. c. conductivity, activation energy for the viscous flow, viscosity, density and refractive index, have been presented for acetonitrile+ n-butyl alcohol binary mixtures at various molar concentrations and at temperature 20°C

The values of dielectric constant, dielectric loss, loss tangent, molar polarization, activation energy, viscosity, refractive index, density are all positive values for different molar concentrations at temperature 20°C in binary mixtures of acetonitrile + n-butyl alcohol. These studies suggested that the very strong interactions take place between acetonitrile and n-butyl alcohol molecules. It is found that

- As the molar concentration of acetonitrile in the binary mixture of acetonitrile + n-butyl alcohol increases, the 1) dielectric constant of binary mixtures goes on increases, but refractive indices of the binary system goes on decreases at temperature 20°C.
- As the mole fraction of acetonitrile in the binary mixtures of acetonitrile + n-butyl alcohol increases, viscosities, densities, and activation energy goes on decreases at temperature 20°C. This is due to at least one of the component in the corresponding systems exhibits hydrogen bonding.
- Dielectric loss is proportional to a. c. conductivity of the binary liquid mixtures of acetonitrile + n-butyl alcohol. In this system dielectric loss increases may be due to increasing rotational or translational motion of the molecules.
- In the present work loss tangent curves indicates large microwave energy absorption takes place at X<sub>A</sub>=0.535343 mole fractions of acetonitrile at 20°C temperature.
- It is also observed that in the system acetonitrile + n-butyl alcohol higher the viscosity, lower the a. c. conductivity in the binary system at temperature 20°C.
- The macro molecular properties (viscosity, density) indicate the interaction between the molecules of system will depend upon the structure breaking interaction process.
- The dielectric constant of the present system at temperature 20°C gets increases as the molar concentration of acetonitrile in the binary system increases at 10.75 GHz microwave frequency. The increase in the dielectric constant it is due to the ions pairing and dielectric constant decreases due to the ions dissociation, specifically, the dielectric constant of the binary system acetonitrile + n-butyl alcohol got increased as the concentration of solute (Acetonitrile) in the corresponding binary system increases. This is because the molar concentration of acetonitrile in the binary systems increases and also due to the dielectric constant of simple acetonitrile is greater than the n-butyl alcohol.
- At 1:1 ratio of binary mixtures in the system acetonitrile + n-butyl alcohol shows that there is an involvement of intermolecular interaction between solute and solvent is more.
- H-bonding was formed between the H of (Primary alcoholic hydrogen) and nitrogen of acetonitrile at 9) temperature 20°C.
- There is an intermolecular interaction among the atoms and molecules of the binary mixtures leading to Hbond formation of the type 1:1 complex formation between acetonitrile and n-butyl alcohol molecules at 20°C temperature.

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