IOSR Journal of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 11, Issue 2 Ser. I (Mar. – Apr. 2019), PP 40-49
www.iosrjournals.org

Dielectric Behavior of Acetonitrile + N-Butyl Alcohol Binary Mixtures at Microwave Frequency at 10°C

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Abstract: *Values of dielectric constant (ε') and dielectric loss (ε") have been experimentally determined for binary liquid mixtures of acetonitrile with n-butyl alcohol at 10.75 GHz microwave frequencies at 10^0C 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}) and a.c. conductivity (σ_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 10^0C. The values of viscosity have been used to evaluate the activation energy (E_a).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.*

Date of Submission: 26-02-2019 Date of acceptance: 12-03-2019

I. Introduction

Acetonitrile is the chemical compound with formula CH_3CN. It is colorless liquid and is simplest organic nitrile. It is used as a polar aprotic solvent in organic synthesis and in the purification of butadiene. It is used in battery applications because of its relatively high dielectric constant. n-butyl alcohol is a primary alcohol with 4-carbon structure as well as molecular formula C_4H_9OH. It is used as direct solvent and as an intermediate in the manufacture of other organic chemicals (e.g. butyl a crylate, methacrylate and glycol ethers) etc. Alcohols are strongly associated in solutions because of dipole-dipole interaction and hydrogen bonding[1, 2] Therefore; it is seemed important to examine the dielectric behavior of n-butyl alcohol with acetonitrile.

A dielectric investigation of solutions having changeable amounts of interacting molecules helps to detect the formation and composition of complexes in them. According to survey of the literature a few workers have tried to investigate some binary systems taking nitriles as one of the components in the binary mixtures. P. J. Singh et.al [3] have studied dielectric behavior of ketone-amine binary mixture at microwave frequencies and suggests that the strong interaction between the ketone and amine molecule and there is a formation of 1:1 complex in the binary system. shantilal oswal [4] has studied studies on density, viscosity, dielectric constant, and refractive index of binary mixtures of esters in benzene and carbon tetrachloride and suggest that in binary mixtures (EBT+ C_6H_6, EA+ CCL_4) The forces between pairs of different molecules are little less than the forces between pairs of like molecules and that is why the mixture is less viscous. The nature of complex formation in binary mixtures is still far from clear. With this in view, the present paper reports new experimental data, which are the values of dissipation factor (D), dielectric constant (ε′) and dielectric loss (ε″) using surber's technique at 10ºC temperature and at 10.75GHz microwave frequency. Loss tangent (tanδ), molar polarization (P_{12}), a. c. conductivity (σ_p), is obtained by using the above values. These parameters have been explained in terms of molecular interaction between unlike molecules of binary. The microwave frequency 10.75GHz helps to study weak or very weak molecular interaction in dilute solutions.

Knowledge of frequency dependent dielectric properties of binary liquid mixtures is essential both in fundamental studies of determination of solvent structure and it's dynamics as well as in the practical uses.

At fundamental level the x-band microwave frequency dependent dielectric behavior of liquid mixtures gives information on molecular interactions and mechanisms of molecules process. For the prediction of solubility and chemical stability of the drug in analytical sciences the dielectric constant of binary mixed solvent is needed. The research work on this system has not been carried out in the past, by using FDR technique.

Hence, the present investigation which may provide the useful experimental data about the formation of complexes in the acetonitrile +n-butyl alcohol binary system at 10ºC temperature and at 10.75 GHz microwave frequency.

II. Experimental Details

The dissipation factor (D), dielectric constant (ε') and dielectric loss (ε'') were measured using Surber's technique [3,5-6] 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 10^0C temperature. The experimental setup is shown in figure1 was used to measure wavelength (λd). 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 (λd) of the microwave inside the medium.

The measurement of reflected power at resonance gives the attenuation coefficient of the sample[1].Surber has derived the following relations for the dielectric parameters D, ε' and ε''

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

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

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

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′, and ε″ 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 10°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^3 measuring range density 0 to 3 g/cm^3.Temperature 0 to 40^0C.

The refractive index of the pure components and their mixtures were measured at 10°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 10°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) $\leq$ 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.

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.

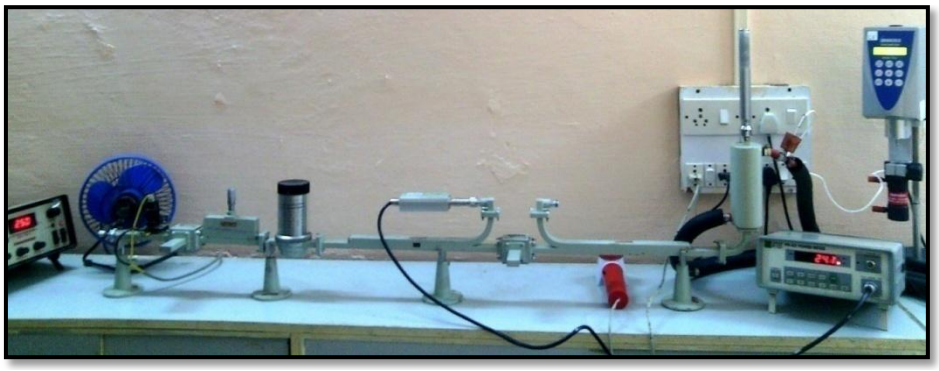

Figure1. The experimental setup of microwave X-band bench for the measurement of ε' and ε''

III. Result and Discussion

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_A) of acetonitrile for the binary mixture (C_2H_3N and C_4H_9OH) are presented in Table (1).The values of viscosity (η), refractive index (n_D), density, mole fraction of solute and solvent are listed in Table (2) at 10°C temperature and at 10.75GHz microwave frequency.

Dielectric constant

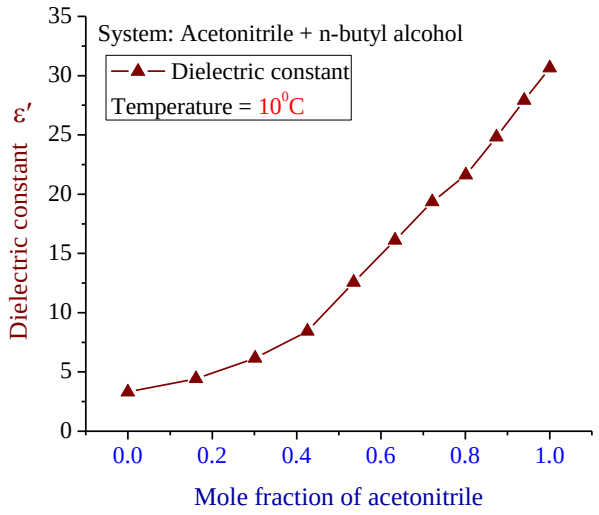

FIGURE 2 Variation of dielectric constant (ε′) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

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 at 10°C temperature. 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 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 10°C

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

Hydrogen bonding interaction

H-bonding was formed between the H of alcoholic group of n-butyl alcohol and nitrogen of acetonitrile at temperature 10ºC.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 intermolecular interaction takes place between solute and solvent [6].Same behavior obtained [7,8]. It is observed that the dielectric constant (ε′) increases with increasing mole fraction of acetonitrile in the binary mixtures of acetonitrile + n-butyl alcohol. Similar value of ε′ for pure n-butyl alcohol is obtained [9,10]

Loss tangent (tanδ)

From figure 3, that the microwave energy absorption in the mixture is greater than that in pure liquids, a maxima in tan δ curve taking place at X_A=0.301699 mole fraction of acetonitrile at 10°C temperature 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 [10].

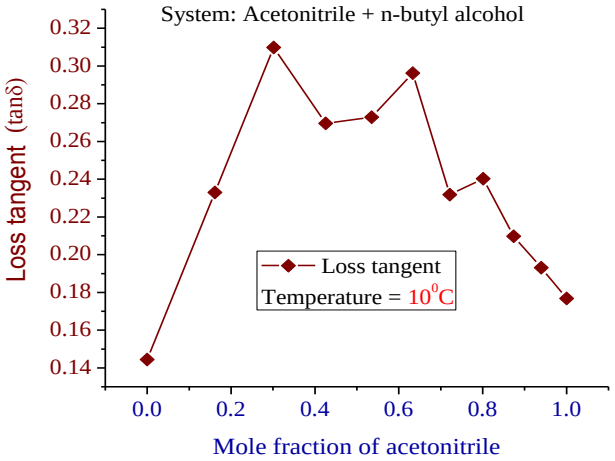

FIGURE 3 Variation of loss tangent (tanδ) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

Molar polarization

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

$$P_{12}=\frac{(\varepsilon'-1)}{(\varepsilon'+2)}\frac{(X_AM_1+X_BM_2)}{d}$$

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. (ε′) is the 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(ε′),dipole moment, molar polarization, donor number, chemical structure solvatochromic quantitative values of Kamlet Taft hydrogen bond acidity, basicity and dipolarity – Polarizability [11].

Current investigation the concentration of acetonitrile in the system increases the dielectric constant (ε′) of the acetonitrile and n-butyl alcohol system increases with molar polarization. It is due to the formation of hydrogen bonding between acetonitrile and n-butyl alcohol molecules.

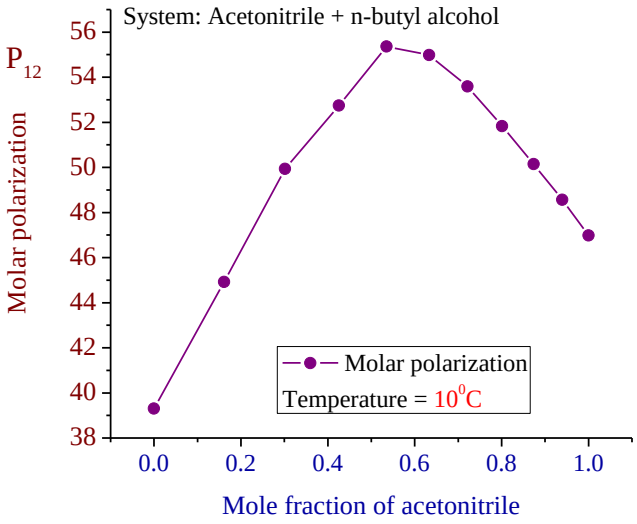

FIGURE 4 Variation of molar polarization versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

In figure 4, The Molar polarization is positive and maximum at X_A= 0.535343 mole fraction of acetonitrile in the binary mixtures of acetonitrile and n-butyl alcohol at temperature 10°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 results have been obtained [10]. 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:

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

Hydrogen bonding interaction

A.c. Conductivity (σ_p)

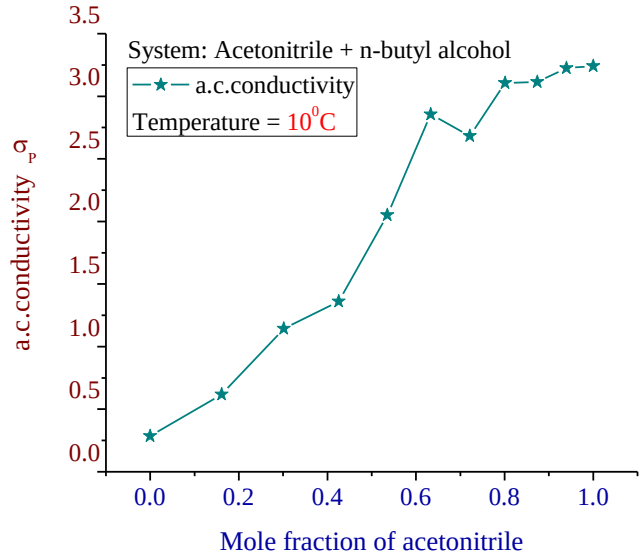

FIGURE 5 Variation of a.c.conductivity (σ_p) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

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 , $\varepsilon_0 = 8.854187816 \times 10^{-12} C^2 / Jm$

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

In this work, it is found that (1) Efficiency of transfer of electric charge is increases as mole fraction of acetonitrile in the binary mixtures increases (2) a.c. conductivity depends on dielectric loss (ε″)
(3) Dielectric loss gets increases as a.c. conductivity increases (4) 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 [11]. a.c conductivity and dielectric loss is presented in figure (5) and (6) respectively.

Dielectric loss

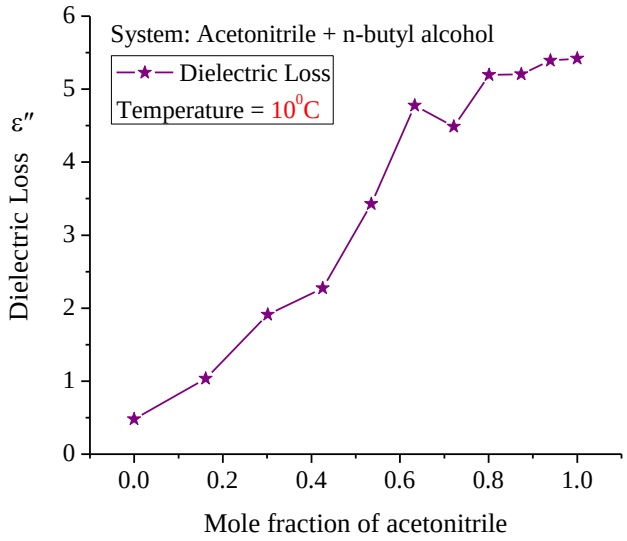

FIGURE 6 Variation of dielectric loss (ε″) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

Refractive Index

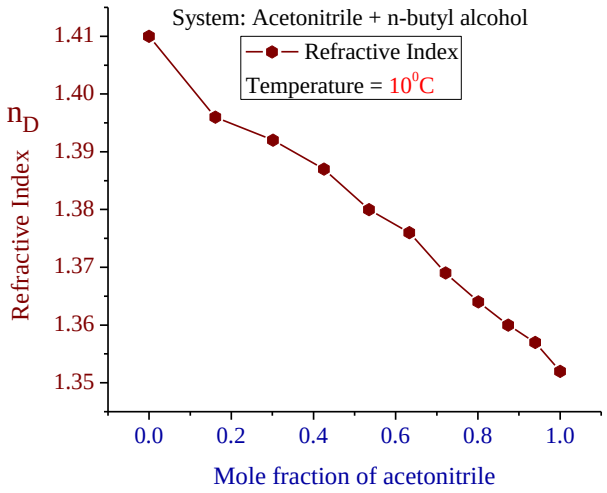

FIGURE 7 Variation of refractive index (n_D) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

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 10°C. Similar value of refractive index for acetonitrile was obtained [12]. Similar value of refractive index of C_4H_9OH was obtained [10].

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 10°C then slowly decreases in the system of acetonitrile +n-butyl alcohol.

Kenneth Hickey et.al [13] reported similar result. Patric Ngoy Tshibangu et al. [14] 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 [15, 16]. 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.

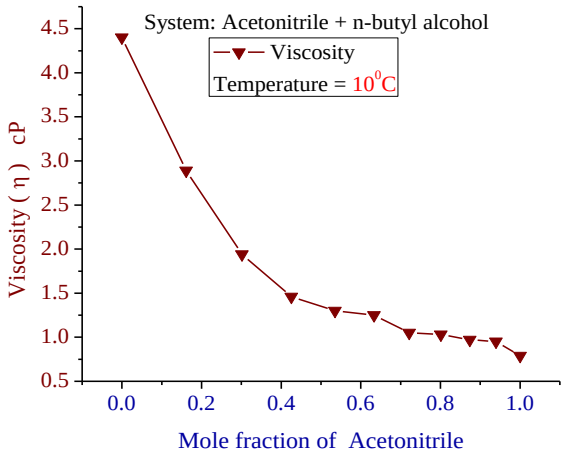

FIGURE 8 Variation of viscosity (η) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

Density (d)

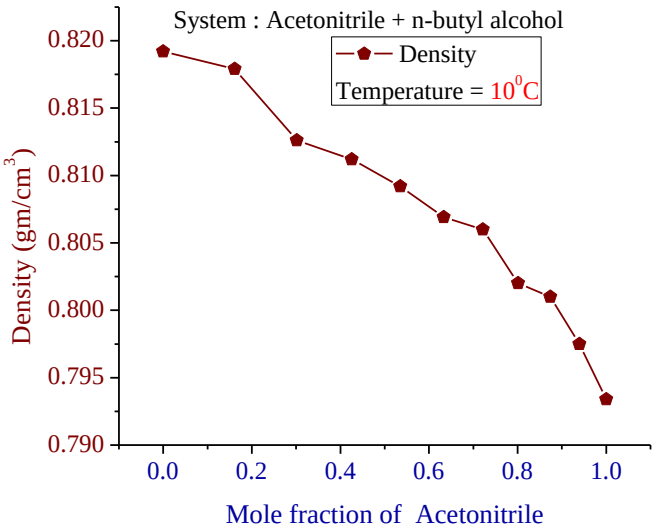

FIGURE 9 Variation of density (d) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

Figure (9) shows the density measurement curve. The values of density of binary liquid mixtures decrease with increasing mole fraction of acetonitrile at 10°C temperature in the binary liquid mixture of acetonitrile + n-butyl alcohol. Similar trends of density are obtained [8, 16, 17, 18, 19]. Similar value of density of pure acetonitrile is obtained [12, 20].

Table 1 Mole fraction of solute (X_A), dissipation factor (D), dielectric constant (ε'), dielectric loss (ε''), loss tangent (tan δ), molar polarization (P_{12}), a.c.conductivity (σ_p) and activation energy (E_a) of binary liquid mixtures of acetonitrile and n-butyl alcohol at 10°C

Sr. no.	X_A	D	ε'	ε''	tan δ	P_{12}	σ_p	E_a
1	0	0.162761	3.304552	0.477243	0.14442	39.309059	0.285414	6.159998
2	0.161088	0.254351	4.438866	1.034311	0.233012	44.92181	0.618569	5.923565
3	0.301699	0.329787	6.166707	1.91089	0.309872	49.939664	1.142807	5.699383
4	0.425502	0.281984	8.437235	2.274155	0.269538	52.748242	1.360057	5.5395
5	0.535343	0.281198	12.566456	3.428942	0.272865	55.360518	2.050677	5.474213
6	0.633456	0.303221	16.118297	4.774496	0.296216	54.985779	2.855384	5.452153
7	0.721625	0.236414	19.354704	4.487686	0.231865	53.593159	2.683858	5.354084
8	0.801289	0.244499	21.610202	5.19262	0.240286	51.833793	3.105443	5.343267
9	0.873622	0.212983	24.813993	5.205644	0.209787	50.148762	3.113232	5.309509
10	0.93959	0.195687	27.927227	5.392119	0.193077	48.567526	3.224754	5.29779
11	1	0.178984	30.654665	5.420051	0.17681	46.98603	3.241458	5.194055

Table 2 Mole fraction of solute (X_A), mole factor of solvent (X_B), density (d), viscosity (η), refractive index (n_D), of binary liquid mixture of acetonitrile and n-butyl alcohol at 10ºC

Sr. no.	X_A	X_B	d gm/cm³	η cP	n_D
1	0	1	0.8192	4.4	1.41
2	0.161088	0.838912	0.8179	2.89	1.396
3	0.301699	0.698301	0.8126	1.94	1.392
4	0.425502	0.574498	0.8112	1.46	1.387
5	0.535343	0.464657	0.8092	1.3	1.38
6	0.633456	0.366544	0.8069	1.25	1.376
7	0.721625	0.278375	0.806	1.05	1.369
8	0.801289	0.198711	0.802	1.03	1.364
9	0.873622	0.126378	0.801	0.97	1.36
10	0.93959	0.06041	0.7975	0.95	1.357
11	1	0	0.7934	0.79	1.352

Activation Energy (E_a)

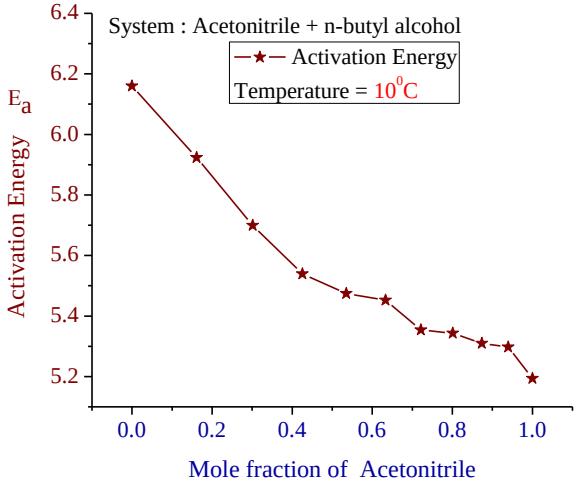

FIGURE 10 Variation of activation of energy (E_a) versus mole fraction of acetonitrile in the mixture of (acetonitrile and n-butyl alcohol) at temperature 10°C

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_a. The graph of Activation energy versus mole fraction (X_A) of Acetonitrile in the system is presented in figure (10). We observed that activation energy decreases with increasing molar concentration of acetonitrile in the binary mixture of acetonitrile + n-butyl alcohol. Same nature of graph is obtained [21]. Figure (10) shows that activation energy of binary mixture verses mole fraction of actonitrile.

IV. Conclusion

The values of dielectric constant, dielectric loss, molar polarization, a. c. conductivity, activation energy for the viscous flow, viscosity, density, refractive index, have been presented for acetonitrile+ n-butyl alcohol binary mixtures at various molar concentrations and at temperature 10ºC

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 10ºC in the binary mixtures of acetonitrile + n-butyl alcohol. These studies suggested that there is the strong interactions between acetonitrile + n-butyl alcohol molecules.

In the present paper it is found that

1) The dielectric constant for the system acetonitrile + n-butyl alcohol gets increases as the molar concentration of acetonitrile in the binary system increases. This is because the molar concentration of acetonitrile in the binary system increases and also due to the dielectric constant of simple acetonitrile is greater than the alcohols.
2) Loss tangent curves indicates large microwave energy absorption takes place at X_A=0.301699 mole fraction of acetonitrile at temperature 10ºC
3) Efficiency of transfer of electric charge is increases as the mole fraction of acetonitrile in the binary mixture increases.
4) Dielectric loss is proportional to a. c. conductivity of the binary liquid mixtures of acetonitrile + n-butyl alcohol.
5) Higher the viscosity lowers the a. c. conductivity in the binary system at temperature 10°C.
6) The macro molecular properties (viscosity, density) indicate the interaction between the molecules of system will depend upon the structure breaking interaction process.
7) Refractive index, viscosity, density, and activation energy got decreases as molar concentration of acetonitrile in the binary mixture increases at temperature 10°C. This is due to at least one of the component in the acetonitrile + n-butyle alcohol binary mixtures exhibits hydrogen bonding.
8) 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 temperature 10ºC. There is an intermolecular interactions among the atoms and molecules of the binary mixtures leading to H-bond formation of the type 1:1 complex formation between acetonitrile and n-butyl alcohol molecules at 10ºC temperature.

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