

# Thermoacoustical Study of [BMim][Br] in Acetonitrile Solvation at 293.15 K by Ultrasonic Experimental Technique

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**Abstract:** The [BMim][Br] ionic liquid is the material of significant interest in various field including pharmaceutical, biomedical, and environmental applications. The thermoacoustic studies of [BMim][Br] as a solute in Acetonitrile solvent is yet to have scope to uncover scenario at near room temperature. In the present paper, the study on liquid mixture of small molar fraction of [BMim][Br] in Acetonitrile has been conducted at temperature of 293.15 K for finding their thermoacoustical properties. It further explored the changes in their molecular interactions. The 2 MHz frequency probe ultrasonic velocity experimental technique has been utilized for the minute concentration of ionic liquid from 0 to 0.1 molar [BMim][Br] in the acetonitrile solvent of at temperature of 293.15 K. Thermoacoustic study covered adiabatic compressibility, acoustic impedance, relaxation time, free length, free volume, internal pressure, Gibbs free energy and enthalpy etc. parameters to reveal perspective of molecular interactions.

**Keywords:** [BMim][Br]+Acetonitrile, Ultrasonic technique, thermoacoustic parameters, Ionic liquid, molecular interaction.

## I. INTRODUCTION

The Ionic Liquid (IL) '1-Butyl-3-methylimidazolium bromide' ([BMim][Br]) is the material of significant interest in various field including pharmaceutical, biomedical, and environmental applications.<sup>[i, ii, iii, iv, v]</sup> The [BMim][Br] IL is a widely studied room-temperature ionic liquid (RTIL). In most of the time in such application it was often needed to mix with different solvents. <sup>[vi, vii, viii]</sup> Thus, how it interacts with the other molecules in solvation is become a crucial need for information and can be extracted in terms of molecular interactions. Ultrasonic technique is usually the best selection method to uncover these interactions through ultrasonic sound wave as a probe.<sup>[ix, x, xi]</sup> Many solvents already study with [BMim][Br] through thermoacoustic parameters against the concentration of [BMim][Br] at different temperatures e.g., in water methanol and ethanol.<sup>[xii]</sup>

Across the survey of literature, we have come to know that the thermoacoustic studies of [BMim][Br] as a solute in Acetonitrile solvent is not yet found and thus this mixture has a scope to uncover scenario near room temperature. Acetonitrile (CH<sub>3</sub>CN) is volatile at room temperature, as well as it is a good solvent for the [BMim][Br] IL and used; already it's ternary mixture study with aspirin molecules for interactions is reported but binary with said IL is not found in literature yet. <sup>[xiii]</sup>. The understanding of molecular interactions and thermoacoustic trend for the concentration of [BMim][Br] in Acetonitrile solvation is required for different applications and for objectives.



Therefore, in the present paper, the study on liquid mixture of small molar fraction of [BMim][Br] in Acetonitrile has been proposed to be conducted at temperature of 293.15 K for finding the thermoacoustical properties of liquid mixture of the [BMim][Br] and acetonitrile solution. The 2 MHz frequency probe based ultrasonic velocity experimental technique has been selected to conduct the present experimental study. Research methodology is given in second section, and outcome is given in third section.

#### Materials and Methods

The research grade 98.% pure [BMim][Br], 99 % pure acetonitrile purchased from Merck India. Double distilled water (laboratory made) and acetone (purchased from Merck, India) were used for cleaning and washing purpose. The digital ultrasonic velocity meter Model VCT-70 (Vi Microsystems Pvt. Ltd., Chennai) with 2MHz ultrasonic probe and  $\pm 0.01 \text{ ms}^{-1}$  accuracy to measure Ultrasonic velocity (U). This instrument was also standardized using double distilled water (density at 293.15 K). The pycnometer (specific gravity bottle) with a precision of  $\pm 0.1 \text{ kg/m}^3$  is used to determined density ( $\rho$ ) of binary mixture of liquid and the Ostwald viscometer with accuracy of  $\pm 0.001 \text{ N}\cdot\text{s}\cdot\text{m}^{-2}$  is used to measure Viscosity ( $\eta$ ). Standard procedures were employed to determine these primary parameters  $\rho$  and  $\eta$  in the in these experiments after standardization and calibration of Pycnometer and Ostwald viscometers using double distilled water [9, 10]. For the Thermostatically regulated water bath with accuracy of  $\pm 0.01 \text{ K}$  is used to maintain constant temperature for the experiment.

Experimental Procedure to obtain primary parameters 'U,  $\rho$  and  $\eta$ ' is as follows: 1) Prepare the liquid mixture solutions of 0 to 0.1 M (with step of 0.02 M) [BMim][Br] ionic liquid by dissolving it in the acetonitrile solvent at temperature of 298.15 K. 2) Obtain U from velocity meter directly on the screen and determine  $\eta$  and  $\rho$  at 293.15 K temperatures from the time of flow method and the specific gravity-metric method respectively. Further these U,  $\eta$ ,  $\rho$  as primary data were used to compute few selected thermoacoustical properties. These coveradiabatic compressibility  $\beta_{ad}$ , intermolecular free length  $L_f$ , specific acoustic impedance Z, Classical absorption coefficient (attenuation)  $\alpha/f^2$  free volume  $V_f$ , internal pressure  $\pi_{in}$ , relaxation time  $\tau$ , Gibbs free energy  $\Delta G^*$  of the liquid mixture; and theses are calculated using the standard thermodynamic relations mentioned in following equations from eq.(1) to eq.(9).

$$\beta_{ad} = \frac{1}{\rho U^2} \quad (1)$$

$$L_f = K_T \sqrt{\beta_{ad}} \quad (2)$$

where  $K_T$  is a Jacobsen's constant  $(93.875 + 0.375T) \times 10^{-8}$  and T being the absolute temperature.

$$V_f = \left( \frac{M_{eff} U}{K \eta} \right)^{3/2} \quad (3)$$

where,  $M_{eff}$  is an effective molecular weight of mixture ( $M_{eff} = \sum M_i x_i$ ) &  $K = 4.28 \times 10^9$ .

$$b = V \left[ 1 - \left( \frac{RT}{MU^2} \right) \left( 1 + \frac{MU^2}{3RT} \right)^{\frac{1}{2}} \right] \quad (4)$$

where, R is gas constant =  $8.314 \text{ J/mol}\cdot\text{K}$ .

$$Z = U\rho \quad (5)$$

$$\frac{\alpha}{f^2} = \frac{8\eta\pi_i^2}{3\rho U^2} \quad (6)$$

$$\therefore \pi_i = bRT \left( \frac{K_j \eta}{U} \right)^{\frac{1}{2}} \left( \frac{\rho^{\frac{2}{3}}}{M_e^{\frac{2}{3}}} \right) \quad (7)$$

where, b (=2) is Cubic Packing factor;  $K_j$  - temperature independent constant =  $4.28 \times 10^9$ ; and  $M_{eff}$ - resultant/ effective molecular weight of mixture.

$$\tau = \frac{4}{3} (\beta_{ad} \cdot \eta) \quad (8)$$



$$\Delta G^* = RT \ln \left( \frac{k_B T \tau}{h} \right) \quad (9)$$

where, R is gas constant = 8.314 J/mol.K, h be the Plank's constant (its value is  $6.63 \times 10^{-34}$  Js) and N is the Avogadro's number i.e.  $6.022 \times 10^{23}$  mole<sup>-1</sup>.

### III. RESULTS AND DISCUSSION

#### A. Experimental Primary Parameters

At **293.15 K (20 °C)**, the experimental physical properties of pure **Acetonitrile** under atmospheric pressure density 776.94 kg/m<sup>3</sup> (however intercept on Y axis came out approximately ~784.5 kg/m<sup>3</sup> in linear fitting shown in fig.1(a); supports strong linear property) And ultrasonic velocity 1288 m/s are matched with literature values of: **Density**= 782.2 kg/m<sup>3</sup>[<sup>xiv</sup>] and **Ultrasonic Velocity**:1299 m/s [<sup>14</sup>], 1283 [<sup>xv</sup>]. Whereas at **20 °C (293.15 K)**, pure **1-butyl-3-methylimidazolium bromide ([BMim][Br])** exhibits: **Density is 1292 Kg/m<sup>3</sup>**. Depending on the trace water content and purification levels common to this highly hygroscopic ionic liquid, literature values generally range between **1290-1300 Kg/m<sup>3</sup>** and **Ultrasonic Velocity is approximately 1580 to 1610 m/s**. [<sup>12</sup>]. Since largest amount dissolved in present study is very small i.e., 0.1 M, this velocity could not be achieved for all binary mixtures prepared here.

Table 1: Primary parameters directly obtained from the experiment

| C<br>(M) | U<br>(m/sec) | ρ<br>(Kg/m3) | η<br>(mPaSec) |
|----------|--------------|--------------|---------------|
| 0        | 1288.012     | 776.78       | 0.4447        |
| 0.02     | 1300.406     | 786.45       | 0.4593        |
| 0.04     | 1303.112     | 787.77       | 0.4633        |
| 0.06     | 1305.179     | 788.88       | 0.4660        |
| 0.08     | 1306.084     | 789.87       | 0.4673        |
| 0.1      | 1309.967     | 793.41       | 0.4730        |

The density of liquid, viscosity of liquid and Ultrasonic velocity are provided in the Table1, and their graphical presentations are shown in Fig.1(a), (b) and (c) respectively. Initially, all these primary parameters drastically increase on addition of 0.02 M [BMim][Br] into acetonitrile from the pure acetonitrile solution. But By step increment of 0.02 M from 0.02 M to 0.1 M their rate (values are mentioned within the graphs along with fitting illustration) are reduced and substantially signify toward linearity. For [BMim][Br] mixed liquid's data points, linear fitting revealed density, viscosity and ultrasonic velocity is increased with rate of 80.065 Kg/m3/M, 0.158 mPa-s/M and 110.47 m/s/M (for per molar change in Acetonitrile). These changes are significant. From η, fig.1(b) suggests liquid mixture exhibits *Uniform Frictional Resistance*. [<sup>xvi</sup>] Unfortunately, the η is deviated, might be due to hygroscopic and volatile preferably and yet need some work to be performed on this due to the same result has been repeated for different attempts with change in sources, materials and methods known to us. Thus re-production of same results has been validated the data.



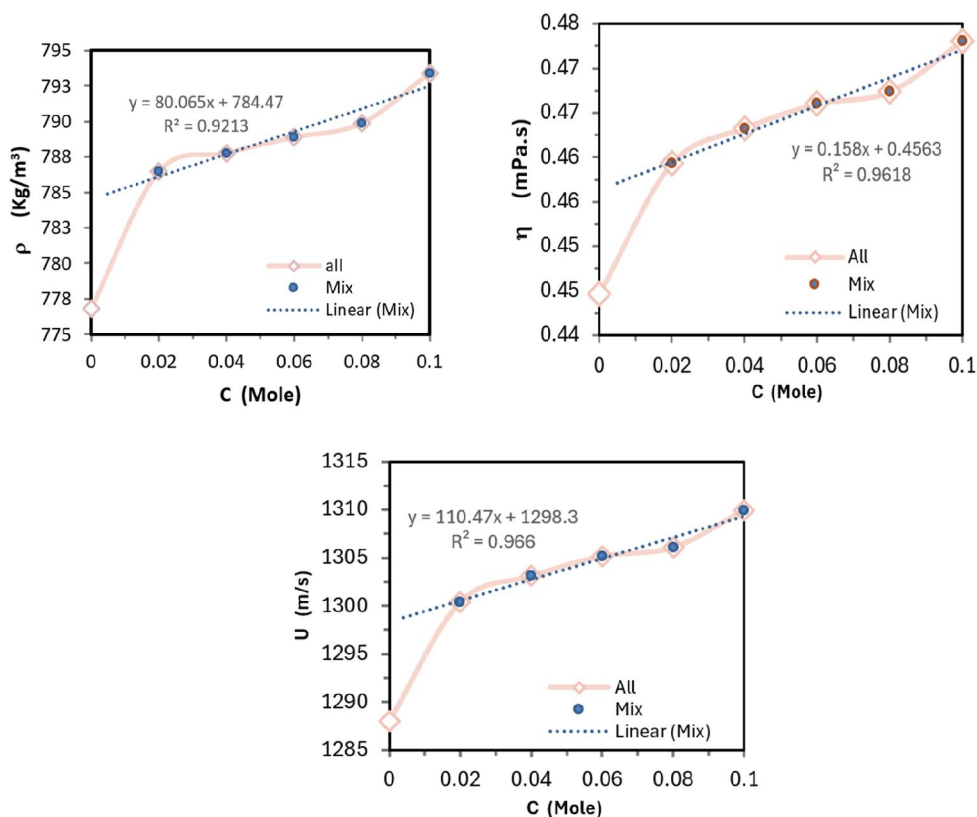


Fig. 1. Graphical illustration of Density ( $\rho$ ), viscosity ( $\eta$ ) and ultrasonic velocity ( $U$ ) as a function of molar concentration of IL in solvent

### B.. Calculated thermoacoustical parameters

The calculated thermoacoustic parameters of the '[BMim][Br]+Ac' binary mixture from their primary data is provided in Table 2 and illustrated graphically in fig.(2) as a function of molar concentration of IL in Acetonitrile solvent. Alike primary parameters all other parameters in fig.2 also reflect drastic change from pure solvent Acetonitrile to addition of first minimum dose of IL of 0.02 M in molar concentration. Present adulterated solution of mixture showed more close linear trending properties. In addition to primary parameters with adiabatic compressibility, their combination signifies presence of strong **association rather than dissociation**. The components of mixture are actively interacting to form a more stable, cohesive, and tightly bound molecular network and indicated the occurrence of Kosmotropic Effect (means Structure-Making Behavior).

Acoustical impedance is elevated (increases) as shown in fig.2(b), because both density and velocity increase, that must be mathematically happen. Physically, a higher acoustic impedance means **the medium is offering more resistance to the propagation of sound waves**. The classical absorption coefficient characterizes sound energy loss due to viscous friction. Even though  $\eta$  increases,  **$U$  increases much more rapidly** and it is cubed in the denominator. The sharp, nonlinear drop in absorption shows that the medium has become a highly efficient transmitter of acoustic energy specifically at the no-addition to addition of IL part in fig.2(b). The nonlinearity is pointing to the fact that stable **ion-pair formations** may occur, or the **complex solvation shells may be formed**.



Table 2.

Thermoacoustic parameters calculated from primary parameters.

| C<br>(M) | $\tau \times 10^{-13}$<br>(Sec) | Z<br>(Kg/m <sup>2</sup> sec <sup>1</sup> ) | $(\alpha/\rho^2)$<br>$\times 10^{-15}$ cla | $L_f \times 10^{-11}$<br>(m) | $V_f \times 10^{-7}$<br>(m <sup>3</sup> /mole) | $b \times 10^{-6}$<br>(m <sup>3</sup> /mole) | $P_i \times 10^{-8}$<br>(N/m <sup>2</sup> ) | $\Delta G^*$<br>(KJ/mol.K) |
|----------|---------------------------------|--------------------------------------------|--------------------------------------------|------------------------------|------------------------------------------------|----------------------------------------------|---------------------------------------------|----------------------------|
| 0        | 4.6008                          | 1.0005                                     | 7.0438                                     | 5.4310                       | 1.4643                                         | 46.774                                       | 6.5607                                      | 2.5137                     |
| 0.02     | 4.6046                          | 1.0227                                     | 6.9823                                     | 5.3460                       | 1.4248                                         | 46.486                                       | 6.6556                                      | 2.5157                     |
| 0.04     | 4.6175                          | 1.0265                                     | 6.9873                                     | 5.3305                       | 1.4204                                         | 46.645                                       | 6.6500                                      | 2.5225                     |
| 0.06     | 4.6234                          | 1.0296                                     | 6.9852                                     | 5.3183                       | 1.4208                                         | 46.812                                       | 6.6359                                      | 2.5256                     |
| 0.08     | 4.6245                          | 1.0316                                     | 6.9821                                     | 5.3113                       | 1.4255                                         | 46.980                                       | 6.6145                                      | 2.5262                     |
| 0.10     | 4.6326                          | 1.0393                                     | 6.9736                                     | 5.2837                       | 1.4153                                         | 47.010                                       | 6.6306                                      | 2.5305                     |

According to Jacobson's Free Length Theory,  $U$  is inversely proportional to the intermolecular free length  $L_f$ . A **decrease in  $L_f$**  means the empty space between the molecules is shrinking. The solute [BMim][Br] and solvent Acetonitrile molecules are being pulled tightly together due to **strong attractive forces as seen in previous discussion**. The Van der Waals constant i.e., '**b**' is **increasing in** means that the effective hydrodynamic size or volume of the interacting molecular clusters is increasing as shown in fig.2(f).

Internal pressure ( $\pi_i$ ) measures the cohesive forces holding the liquid together, whereas free volume ( $V_f$ ) represents the empty space available for a molecule to move and both share an inverse relationship as illustrated in 'fig.2(h) and (g)'. The three-stage non-linear trend signifies the complex, concentration-dependent structural rearrangements using  $\pi_i$ ; these are: 1) **Initial Phase Low Concentration** - At low concentrations, the introduction of the large ionic liquid ions ([BMim]<sup>+</sup> and [Br]<sup>-</sup>) breaks apart the self-associated or existing dipole-dipole structures of pure Acetonitrile (solvent-solvent interactions). 2) **Intermediate Concentration i.e. Linear Phase** - The steady linear phase shows the stabilization and ion-dipole interactions and the dominance of **solute-solvent complexation**. The system becomes systematically structured as more ions, from 0.02 M to 0.08 M, are solvated by the solvent. 3) **Final Phase at the End**: The slight reversal or increase at the end at 0.08 M to 0.1 M indicates **ion-ion self-association (solute-solute interactions)** is suspected (not sure).

Nonlinear increase in relaxation time ( $\tau$ ) is shown in fig.2(h); the increase suggests newly formed ion-solvent structures of [BMim][Br]+Acetonitrile are stable and take longer to rearrange and the **non-linear** progression proved the interaction is not a simple dilution effect, but rather a dynamic, **concentration-dependent complex formation** or structural clustering ( **Structural Rigidity**) for **IL's increment**. Nonlinear increase in change in Gibbs Free Energy ( $\Delta G^*$ ) of activation is observed in fig.2(i). Increment **and nonlinearity** have represented thermodynamic **stability and complex formation respectively**. It means a larger amount of energy is required for the cooperative rearrangement of the liquid molecules during the viscous flow and acoustic propagation. The non-linear rise portion implies the system undergoes specific structural transformations and building a closely and tight bounded molecular network.

All thermoacoustic properties plotted in fig.2 reveal the interaction of type dipole-ion in between solute and solvent along with the dipole-dipole interaction between solvent molecules. No ionic (ion-ion) interaction observed from the keen investigations of the value of all these parameters except the ending trend of  $\pi_i$  (fig.2(g)). The ion-ion interaction reflected in from 0.08 M to 0.1 M range in  $\pi_i$  but  $\Delta G^*$  does not support in sum-up for the overall prominent interactions. In addition to this the dipole-dipole or dipole induced interactions presence are also supported by this study. But largely, the hydrogen bond presence is explicitly indicated by fig.2 plots. The Gibbs free energy of activation is approximately ~2.5 KJ/mole which is correlated to require to form a single hydrogen bond per mole of substance of liquid solution (from the value). Thus naturally hydrogen bond inherently present and indicated increment with addition of IL [BMim][Br].



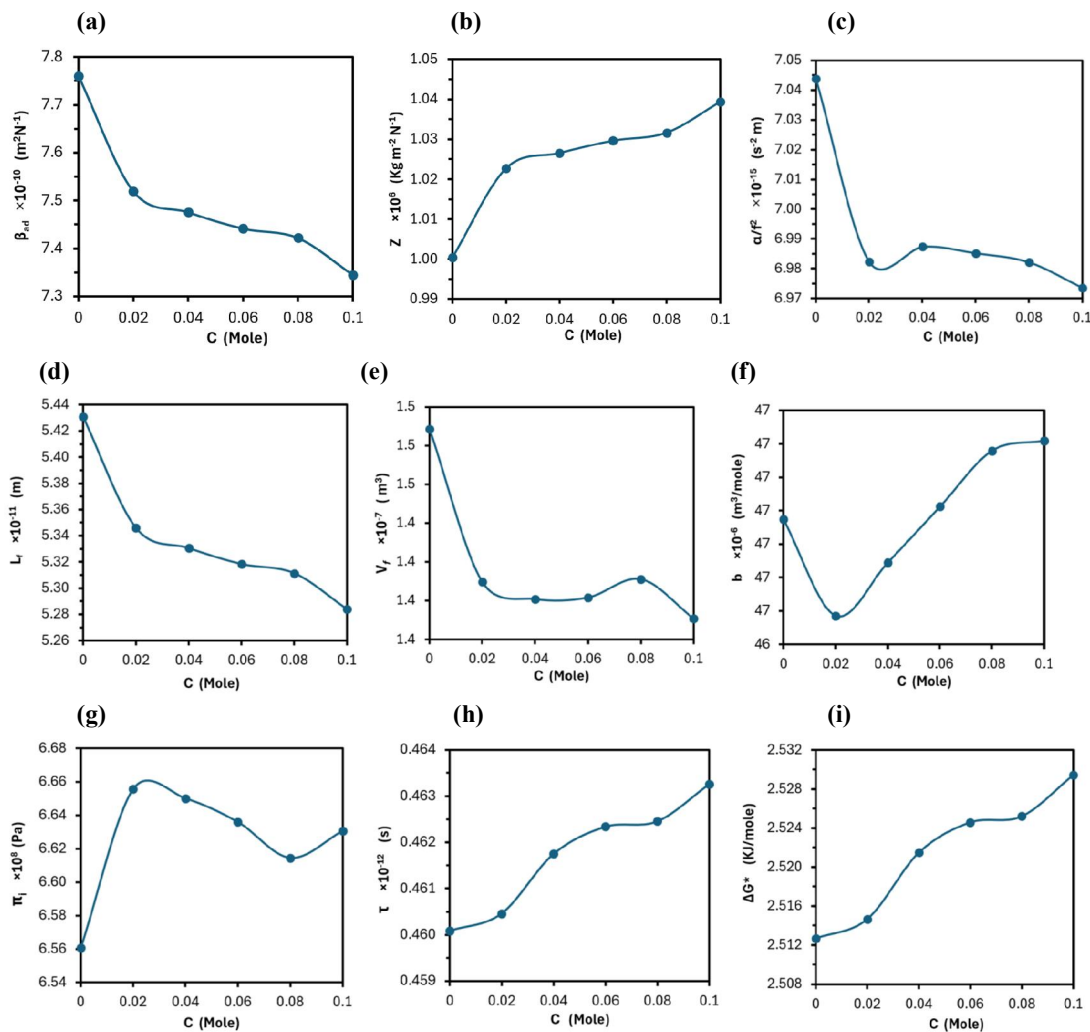


Fig.2. Illustration of graphs of thermoacoustic parameters plotted as a function of molar concentration of IL in Acetonitrile solvent: (a) adiabatic compressibility ( $\beta_{ad}$ ), (b) acoustical impedance ( $Z$ ), (c) absorption coefficient, (d) free length ( $L_f$ ), (e) free volume ( $V_f$ ), (f) Van der Waal's 'b' constant ( $b$ ), (g) internal pressure ( $\pi_i$ ), (h) relaxation time ( $\tau$ ), (i) Gibbs free energy of activation barrier ( $\Delta G^*$ ). [Note: C is molar concentration of the solute in 100ml solvent]

### C. Perspective and final verdict

Thus,  $U$ ,  $\eta$  and  $\rho$  indicates **strong molecular interactions** and the formation of a **highly structured, cohesive liquid medium**. This trend associated with following characteristics of solution: Structural Compactness and Molecular Association, Enhanced Intermolecular Forces, Near-Ideal Homogeneous Behavior etc. **The** medium becoming uniformly more rigid and less compressible. These are also suggested the High Stability and Structural Uniformity. The reason is lied on the e strictly linear plots for mixture, it signified that the mixture was behaving as a homogeneous, stable system without sudden complex transitions or phase disruptions at 293.15 K. The  $U$ ,  $\rho$  and  $\eta$  trends also suggested *Strong Cation-Solvent and Anion-Solvent Solvation*, which is due to an active, robust **ion-dipole interaction** taking place between the ionic components of [BMim][Br] and the highly polar Acetonitrile molecules. [13]. All



Calculated Thermoacoustical parameters also confirmed and validated these perspective and provide more insight in how this would happen; details are covered already in section 3.3. Therefore it is confirmed that there is a tightly bound, well-structured molecular arrangement within liquid of this binary mixture that leaves less "free volume" (see fig.(e)) for the atoms to move.

#### IV. CONCLUSION

In present study, the ultrasonic characterization of properly dissolved [BMim][Br] in Acetonitrile solvent has been successful done using 2 MHz frequency probe at 293.15 K with all precautions. For step increments of molarity of IL, thermoacoustical properties are discussed with molecular interactions. The overall data interprets that strong, strong, non-ideal ion-dipole (solute-solvent) interactions leading to cooperative complexation and high structural ordering between [BMim]<sup>+</sup>[Br]<sup>-</sup> and Acetonitrile and it govern the system through most concentrations, triggering tight molecular packing. The system undergoes structural transitions progressing from a **solvent-breaking phase** at very low concentrations to **solute-solvent complexation** and possibly leading to **ion-pair self-association** at higher concentrated levels at 293.15 K. But one thing is sure that these associations are due to prominent involvement of hydrogen bonding and its enhancement for IL addition.

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#### Conflicts of Interest:

There are no conflicts of interest to declare.

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