



# THE DIELECTRIC RESPONSE OF AMIDE FUNCTIONALISED BENZIMIDAZOLIUM DICATIONIC IONIC LIQUIDS: EFFECT OF ANION VARIATION

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## Abstract

This paper is focused on the synthesis of a new class of amide functionalized dicationic benzimidazolium based ionic liquids (DBimILs) using dialkylation reaction of N-propyl benzimidazole followed by anion exchange reaction. The important requisite for the application of ionic liquids is to understand their structure and properties. The structures of DBimILs, based on benzimidazolium combined with chloride, tetrafluoroborate, hexafluorophosphate and trifluoromethane sulphonate anion, were identified using <sup>1</sup>H, <sup>13</sup>C, <sup>19</sup>F, <sup>31</sup>P NMR, Mass and FTIR spectroscopy. The thermal stability of synthesized ionic liquids was investigated using thermogravimetric analysis (TGA). The dielectric properties of synthesized ionic liquids were investigated in the frequency range 50 Hz – 20 MHz and temperature in the range 30-150°C. The correlation between the structure and the dielectric properties of synthesized ionic liquids was evaluated. It was found that the values of dielectric constant were significantly changed simply by varying the anionic moieties of ionic liquids. Thus, taking into account the dielectric constant and the dielectric loss values, it can be concluded that these ionic liquids have the great potential to be designed for energy storage applications.

**Keywords:** Dicationic Ionic Liquids, benzimidazolium, dielectric properties

## Introduction

From last few decades, ionic liquids (ILs) have gripped much more in green chemistry due to designer medium for chemical reactions. Ionic liquids (ILs) are consists of organic pair of soft cationic or anionic species in the form of molten

salts [1]. The huge numbers of potential ionic liquids are known due to their combination of cations and anions. However, the chemical and physical properties vary tremendously by changing composition. Ionic liquids gained enormous advantages over commercial organic solvents since they are liquids at room temperature [2], non flammable [3], non combustible, easy to recycle [4], non reactive in various industrial applications [5], negligible vapor pressure [6], high inherent conductivity [7], wide electrochemical window [8], high thermal and electrochemical stability [9, 10], also these RTILs have potential in catalytic processes [11]. The ionic liquids are used in many areas of chemistry, biochemistry, engineering and material science [12-16].

ILs are environmentally benign and designable due to their nonvolatile, recyclability and good thermal stability, ionic liquids are categorized as green solvents [17-19]. Ionic liquids could be design by changing different ions (cations or anions) combinations for variety of applications. As a result many researchers and scientists have designed and developed task specific ionic liquids for many application fields [20-23].

A new class of ionic liquids i.e. germinal dicationic ionic liquids (DILs) [24, 25] which consists of two singly charged cations linked with spacer (aliphatic or aromatic chain) and paired with two singly charged anions. From the literature it is found that the germinal dicationic ILs posses higher thermal stability than mono cationic [26, 27]. An applications of dicationic ILs have been investigated, such as dyes sensitized solar cells [28 29], electrolytes in secondary batteries [30, 31], stationary phase for gas chromatography [32, 33], high temperature lubricants [34, 35], and solvent

cum catalyst for high temperature reactions [25].

The investigation of the dielectric properties of ionic liquids generally characterized by the dielectric constant ( $\epsilon$ ) and by the dielectric loss ( $\tan\delta$ ) which determines important facets of their properties. Recently, the dielectric constant of ionic liquids has been measured using dielectric spectroscopy with high accuracy [36-39]. It was reported that the ionic liquids have modest dielectric constants which reflects contribution from electronic and orientation polarizations caused by the molecular polarizability and the permanent dipole moments of ions respectively [40]. Most of the ionic liquids reported exhibits dielectric constant in the range 10 to  $16^{1-7}$ . In the present study, a new class of thermally stable amide functionalized dicationic benzimidazolium based ionic liquids (DBimILs) were synthesized using dialkylation reaction of N-propyl benzimidazole followed by anion exchange reaction [41]. The influence of anion variation on the dielectric properties of these ionic liquids is investigated to understand the contributions from different polarizations.

## Experimental

### Materials

Benzimidazole, 1-bromopropane, 1, 6-hexamethylene diamine were obtained from Sigma-Aldrich (India). Sodium hydride (60%), tetrahydrofuran (specially dried), toluene (specially dried), ethyl acetate, diethyl ether, sodium sulphate (anhydrous), and potassium bicarbonate were purchased from S.D. Fine Chemicals, Mumbai. Chloroacetyl chloride was purchased from Merck & Co Ltd. All the chemicals were used as received unless it is specified.

### Characterizations

NMR spectra of synthesized ionic liquid were recorded in DMSO- $d_6$ ,  $CDCl_3$  on a Bruker spectrometer operating at 400 MHz and chemical shifts are given in ppm downfield from TMS ( $\delta = 0.00$ ).

FTIR spectra of synthesized products were obtained using Fourier Transform Infrared Spectrophotometer (Shimadzu, IRAffinity-1, Japan) in the wavenumber range  $400-4000\text{ cm}^{-1}$  in the transmittance mode using KBr pellets.

Mass spectra of all catalysts were recorded on a JEOL GC MATE II HRMS (EI) and ESI-MS spectrometer.

The thermal stability of synthesized ionic liquids was evaluated using thermogravimetric analyzer (TGA Q5000 V3.13 Build 261) in the temperature range  $50-700^\circ\text{C}$  at the heating rate of  $20^\circ\text{C}/\text{min}$  under  $N_2$  atmosphere.

The dielectric properties of all IL samples were obtained using measured using Wayne Kerr 6500B (Chichester, West Sussex, UK) Precision Impedance Analyzer in the frequency range from 50 Hz to 20 MHz and temperature in the range  $40-150^\circ\text{C}$  with an accuracy of about  $\pm 0.1^\circ\text{C}$ . (The dielectric measurements were performed on pellets processed by applying uniaxial pressure on powder.).

### Synthesis of DBimILs

The bischloroacetyl of hexamethylene diamine and propyl benzimidazole prepared by known procedure and the spectral data are matching with the literature.

#### Synthesis of 3,3'-(2,2'-(hexane-1,6-diylbis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(1-propyl-1H-benzo[d]imidazol-3-ium) chloride. (DBimIL-3a)

The synthesis of ionic liquid DBimIL-3a was carried out based on the literature procedure (Scheme 1) [42]. 1-Propylbenzimidazole (**1**) (2.1 equivalents) and N,N'-(hexane-1,6-diyl) bis (2-chloroacetamide) (**2**) (1 equivalent) was charged with toluene in a 100 mL round bottom flask and the reaction mixture was stirred at  $90^\circ\text{C}$  for 48 h. The white viscous solid precipitate was washed repeatedly with ethyl acetate and diethyl ether to remove un-reacted residues and finally dried in vacuum at  $60^\circ\text{C}$  for 2 h, to get pure white solid.

Yield = 83.45%.  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ ):  $\delta = 0.92$  (t,  $J = 7.40$  Hz, 6H), 1.28 (bs, 4H), 1.43 (bs, 4H), 1.95 (m, 4H), 3.11 (t,  $J = 6.80$  Hz, 4H), 4.55 (t,  $J = 7.0$  Hz, 4H), 5.38 (s, 4H), 7.68 (d,  $J = 8.60$  Hz), 7.99 (m, 2H), 8.13 (m, 2H), 9.02 (s, 2H), 9.94 (s, 2H).  $^{13}\text{C}$  NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  10.00, 22.03, 25.93, 28.7, 42.69, 48.11, 48.49, 113.61, 113.75, 126.53, 126.71, 130.75, 131.6, 143.3, 164.4. FT-IR (KBr,  $\nu/\text{cm}^{-1}$ ): 3371, 3178, 3037, 2929, 2856, 1660, 1558, 1438, 1359, 1263, 748, 655. TOF MS (EI+) calculated for  $C_{30}H_{42}Cl_2N_6O_2$ ; 553  $[\text{M}-\text{Cl}]^+$ ; found 553  $[\text{M}-\text{Cl}]^+$ .

The synthesis of DBimIL-3b, 3c and 3d were carried out according to literature [43]. The DBimIL-3a (1 equivalent) was dissolved in distilled water and aqueous solution of  $\text{NaBF}_4$  (2.1 equivalent) or  $\text{NaPF}_6$  (2.1 equivalent) or  $\text{CF}_3\text{SO}_3\text{Li}$  (2.1 equivalent) were added drop wise with constant stirring for 15 min at room temperature. The reaction mixture was allowed to stir for 5 hr, after which the white solid DBimIL-3b, 3c and 3d precipitated out were collected by filtration and washed several times with ethyl acetate over filter paper, finally dried in oven at 60 °C for 10 h.

**Synthesis of 3,3'-(2,2'-(hexane-1,6-diylbis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(1-propyl-1H-benzo [d] imidazol-3-ium) tetrafluoro borate. (DBimIL-3b)**

Yield = 89.23%,  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  = 0.93 (t,  $J$  = 7.14 Hz, 6H), 1.28 (bs, 4H), 1.43 (bs, 4H), 1.96 (m, 4H), 3.14 (t,  $J$  = 6.14 Hz, 4H), 4.55 (t,  $J$  = 6.93 Hz, 4H), 5.38 (s, 4H), 7.69 (t,  $J$  = 8.60 Hz, 4H), 7.90 (q, 2H), 8.13 (t, 2H), 8.52 (t,  $J$  = 5.03 Hz, 2H), 9.75 (s, 2H).  $^{13}\text{C}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  10.60, 22.04, 26.0, 28.85, 48.15, 48.43, 113.53, 113.78, 126.57, 126.75, 130.75, 131.67, 143.3, 164.36.  $^{19}\text{F}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  = 148.23. FT-IR (KBr,  $\text{v/cm}^{-1}$ ): 3388, 2937, 1695, 1552, 1435, 1112, 1060, 1041, 758, 653.

**Synthesis of 3, 3'-(2, 2'-(hexane-1,6-diylbis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(1-propyl-1H-benzo [d] imidazol-3-ium) hexafluoro phosphate. (DBimIL-3c)**

Yield = 98.12%,  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  = 0.93 (t,  $J$  = 7.35 Hz, 6H), 1.28 (bs, 4H), 1.44 (d, 4H), 1.97 (m, 4H), 3.14 (q,  $J$  = 6.52 Hz, 4H), 4.54 (t,  $J$  = 6.97 Hz, 4H), 5.26 (s, 4H), 7.70 (q,  $J$  = 8.60 Hz), 7.91 (q, 2H), 8.13 (t, 2H), 8.51 (s, 2H), 9.75 (s, 2H).  $^{13}\text{C}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  10.60, 22.04, 26.0, 28.84, 38.95, 48.15, 48.44, 113.54, 113.77, 126.57, 126.75, 130.76, 131.68, 143.32, 164.37.  $^{19}\text{F}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  = -69.19, -71.07.  $^{31}\text{P}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  = -157.36, -152.98, -148.59, -144.20, -139.81, -135.41, -131.03. FT-IR (KBr,  $\text{v/cm}^{-1}$ ): 3423, 3099, 2939, 1691, 1527, 1467, 1197, 823, 759, 740, 555.

**Synthesis of 3,3'-(2,2'-(hexane-1,6-diylbis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(1-propyl-1H-benzo [d] imidazol-3-ium) trifluoro methane sulphonate. (DBimIL-3d)**

Yield = 95.87%,  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  = 0.93 (t,  $J$  = 7.22 Hz, 6H), 1.29 (bs, 4H), 1.44 (bs, 4H), 1.94 (m, 4H), 3.13 (d,  $J$  = 5.88 Hz, 4H), 4.54 (t,  $J$  = 6.80 Hz, 4H), 5.27 (s, 4H), 7.69 (q,  $J$  = 8.60 Hz, 4H), 7.91 (t, 2H), 8.12 (d, 2H), 8.51 (s, 2H), 9.76 (s, 2H).  $^{13}\text{C}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  10.58, 22.04, 26.0, 28.83, 38.95, 48.17, 48.45, 113.53, 113.76, 119.09, 122.30, 126.56, 126.75, 130.77, 131.69, 143.33, 164.37.  $^{19}\text{F}$  NMR (400 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  = -77.77. FT-IR (KBr,  $\text{v/cm}^{-1}$ ): 3311, 3088, 2943, 1685, 1564, 1435, 1246, 1224, 1197, 1153, 1028, 759, 634.

## Results and Discussion

### Thermal analysis:

Thermogravimetric analysis (TGA) was employed to study the thermal stability of synthesized ionic liquid samples. **Figure 1 (a-d)** shows TGA thermograms of four ionic liquid samples namely DBimIL-3a, DBimILs-3b, DBimILs-3c and DBimILs-3d. It can be seen that DBimIL-3a shows initial weight loss below 100°C which could be due to removal of adsorbed water. With further increase in the temperature up to 300°C, DBimIL-3a sample showed very stable weight loss. However, above 300°C, DBimIL-3a starts decomposing rapidly up to 420°C and with further increase in temperature up to 700°C, DBimIL-3a decomposes completely. On the other hand, DBimIL-3b showed better thermal stability than DBimIL-3a upto 300°C. Above 300°C, DBimIL-3b also starts to decompose. Similarly, DBimIL-3c and DBimIL-3d also showed better thermal stability than DBimIL-3a. All ionic liquid samples demonstrate stable weight loss up to 300°C. This indicates that the thermal stability of synthesized ionic liquid is up to 300°C. Therefore, these ionic liquids are promising candidate for energy storage applications where operating temperature required is upto 300°C.

### Infrared spectroscopy

The obtained characteristic IR bands of ILs showed that the strong peaks at 3371, 3388, 3423 and 3311  $\text{cm}^{-1}$  can be attributed to sec. N-H stretching for DBimIL-3a, 3b, 3c and 3d resp. Whereas peaks 2929, 2937, 2939 and 2943  $\text{cm}^{-1}$  indicated to C-H stretching vibrations in the alkyl chains of the cations. The strong peak observed at 1660, 1695, 1691 and 1685  $\text{cm}^{-1}$  stretching vibration shows C=O group of amide bond of all four ILs resp. Moreover strong peak

is observed in the range of 1435-1467  $\text{cm}^{-1}$  which readily assigned to the  $\text{CH}_3$  bending vibrations. The C-N stretching vibrations of amide linkage are evidenced in the range of 1197-1263  $\text{cm}^{-1}$  region. The  $\text{PF}_6$  anion in DBimIL-3c exhibits a peak at 823  $\text{cm}^{-1}$ .

Nevertheless, it should be noticed that some bands were located at 1558, 1552, 1527 and 1564  $\text{cm}^{-1}$  indicated that aromatic stretching.

### Dielectric Properties:

It is important to gain information about the dielectric properties of ionic liquids in order to predict their specific application. In the present study, the dielectric properties of synthesized ionic liquids intended to be used for energy storage applications were determined in the frequency range 50 Hz-20 MHz and temperature in the range 30°C-150°C. **Table 1** depicts comparative values of dielectric constant and the dielectric loss as a function of frequency and temperature. It can be seen that the DBimIL-3c demonstrates lower dielectric constant (76.79, 50 Hz, 150°C) and DBimIL-3d demonstrates ultra high dielectric constant (24059.37, 50 Hz, 150°C) as shown in **Figure 2(a-d)**. All the samples demonstrated high dielectric constant at lower frequencies. In addition, the dielectric constant decreases with increase in the frequency which could be attributed to the decrease in the dipole polarizations at higher frequencies [44, 45]. The dielectric loss plots of synthesized ionic liquids are given in **Figure 3(a-d)**. It can be seen that the DBimIL-3b demonstrates lower dielectric loss (7.48, 50 Hz, 150°C) and DBimIL-3a demonstrates high dielectric loss (24059.37, 50 Hz, 150°C). Also, it is worthy to note that the dielectric loss values of all ionic liquid samples are high at lower frequency and as the frequency increases the dielectric loss decreases. The high values of dielectric loss at lower frequencies are due to the increased mobility of charge carriers giving birth to interfacial or Maxwell Wagner polarizations. The values of dielectric loss are significantly high for all samples which indicate that the conductivity is accelerated due to mobility of free charges [46]. Thus, it can be concluded that the structure of synthesized ionic liquids have significant influence on their dielectric properties.

### Conclusions:

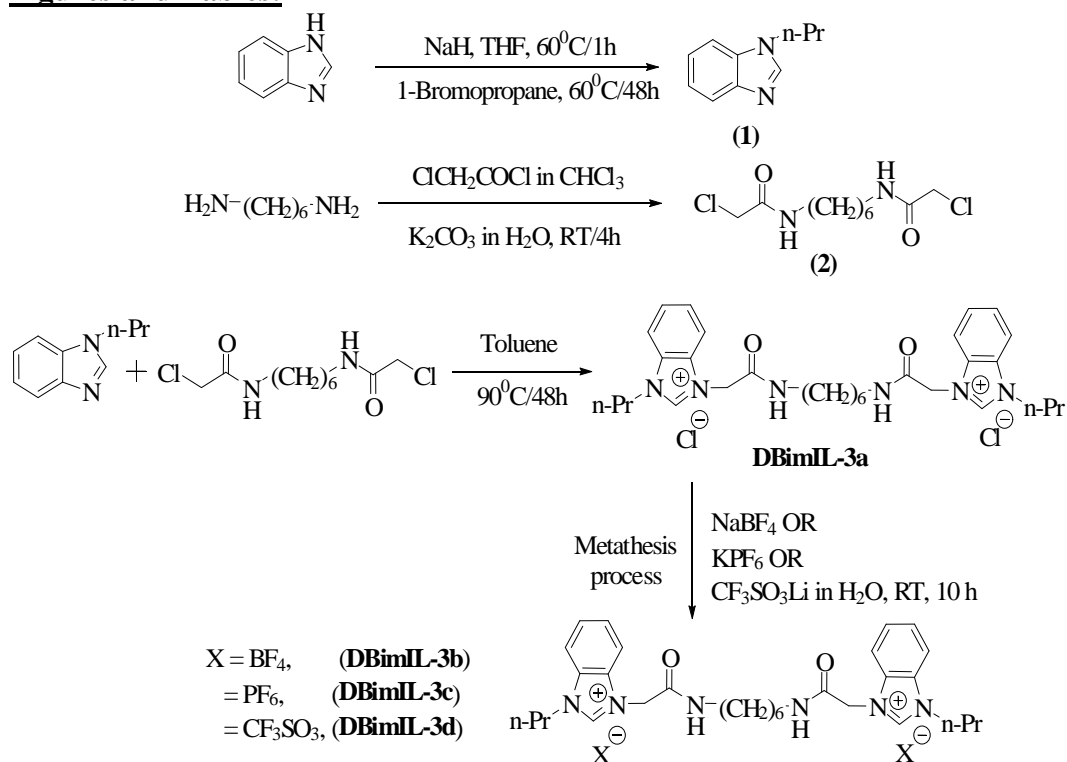
In the present study, new class of amide functionalized dicationic benzimidazolium

based ionic liquids (DBimILs) was prepared by using dialkylation reaction of N-propyl benzimidazole followed by anion exchange reaction. The structures of synthesized DBimILs were identified using  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{19}\text{F}$ ,  $^{31}\text{P}$  NMR, Mass and FTIR spectroscopy and the thermal stability was evaluated using thermogravimetric analysis (TGA). The dielectric properties of synthesized ionic liquids were investigated in the frequency range 50 Hz – 20 MHz and temperature in the range 30-150°C and correlated to their structure. TGA analysis indicates that all the ionic liquids are thermally stable up to 300°C. The dielectric properties indicate that the structure of ionic liquids have significant influence on the dielectric constant values. Thus, taking into account the values of dielectric constant and dielectric loss, it can be concluded that the structure of ionic liquids have strong influence on their dielectric properties. The highest dielectric constant value is exhibited by DBimIL-3d. Hence, it is the most suitable candidate for energy storage applications.

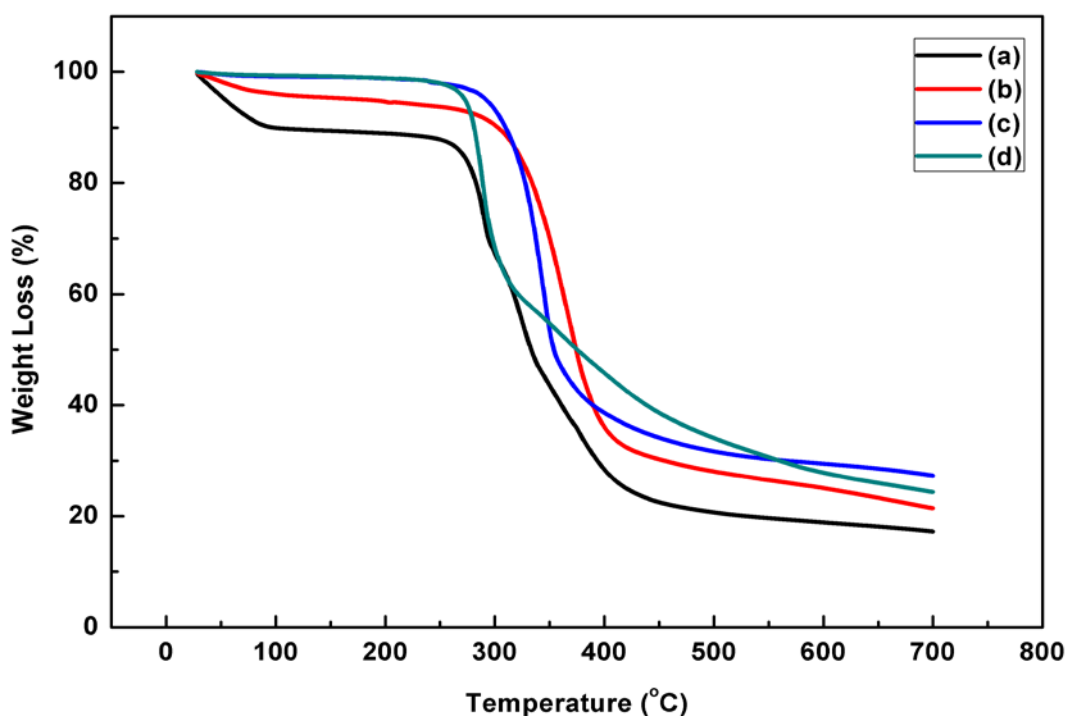
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**Figures and Tables:**

**Scheme 1.** Synthesis of Amide Functionalized Dicationic Benzimidazolium based Ionic Liquid Catalysts (**DBimILs**)



**Figure 1:** TGA thermograms of synthesized ionic liquids (a) DBimIL-a (b) DBimILs-b (c) DBimILs-c (d) DBimILs-d

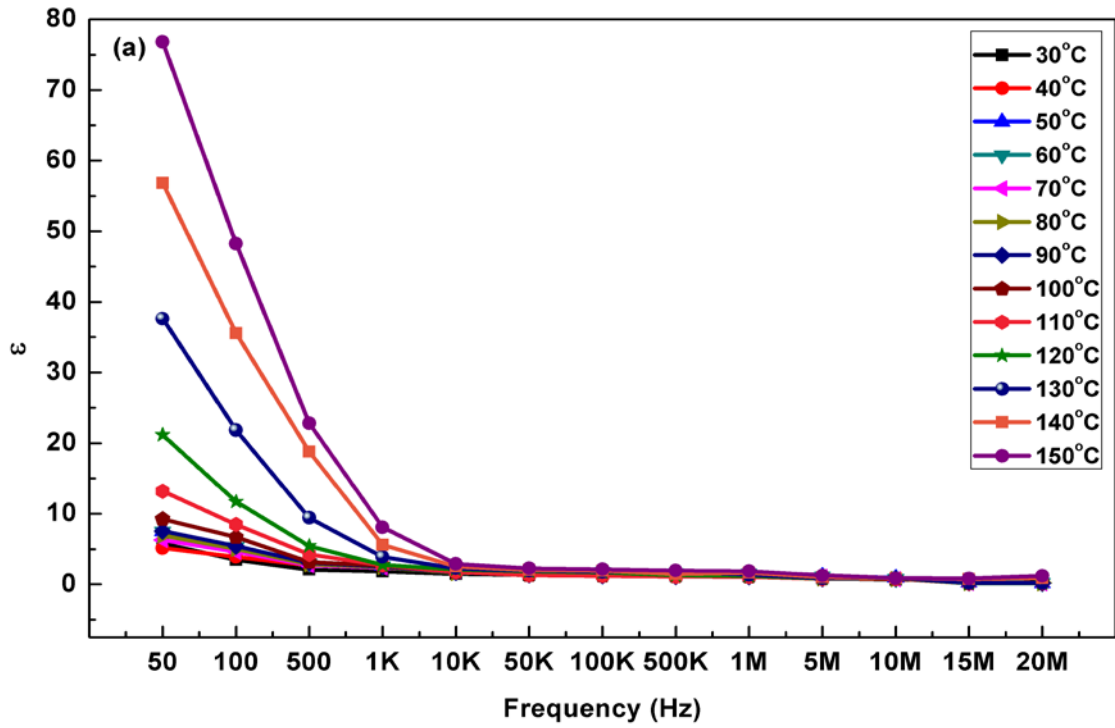


Figure 2 (a): Dielectric constant of DBimIL-3c at various frequencies and temperatures.

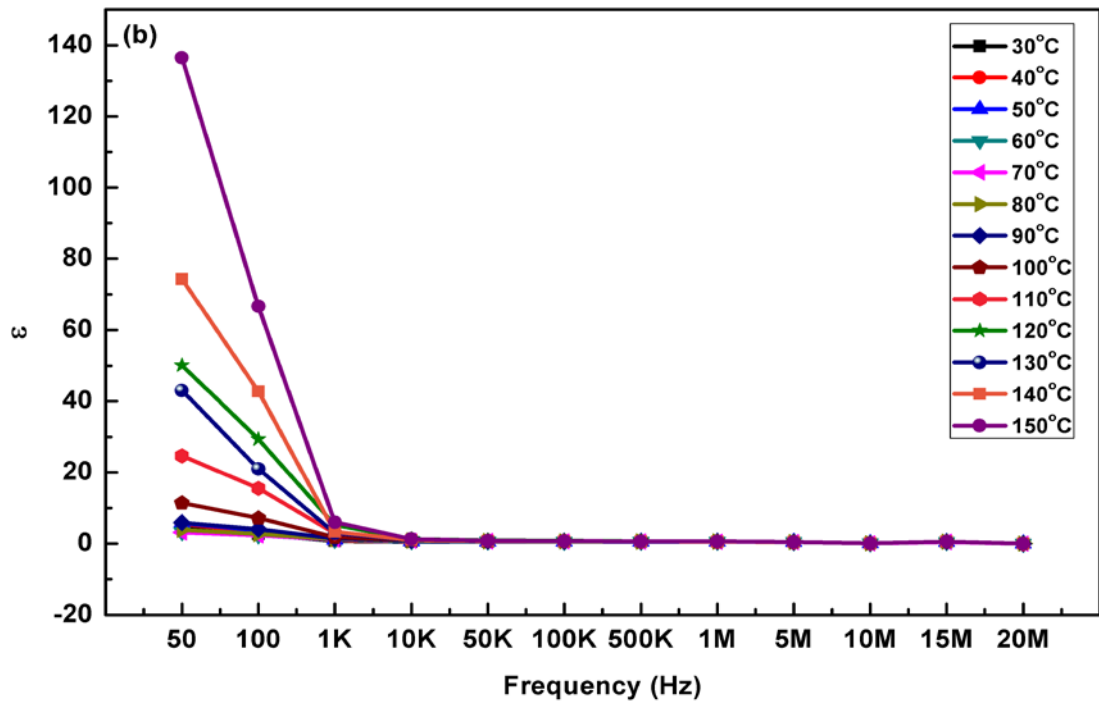


Figure 2 (a): Dielectric constant of DBimIL-3c at various frequencies and temperatures.

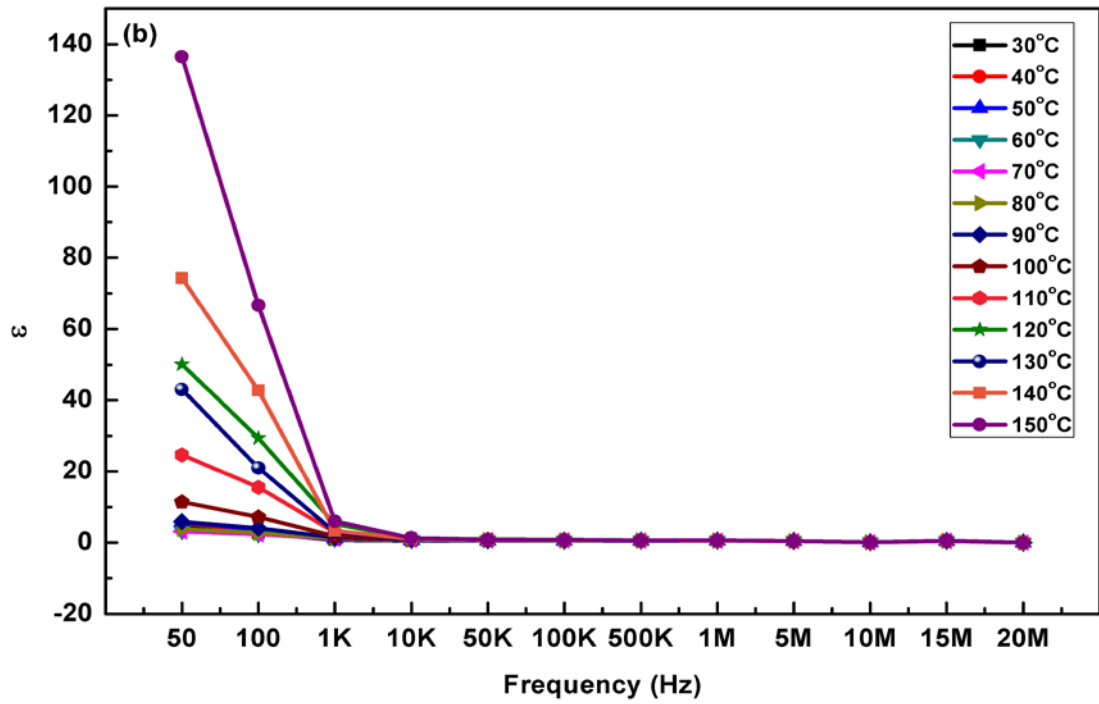


Figure 2(b): Dielectric constant of DBimIL-3a at various frequencies and temperatures

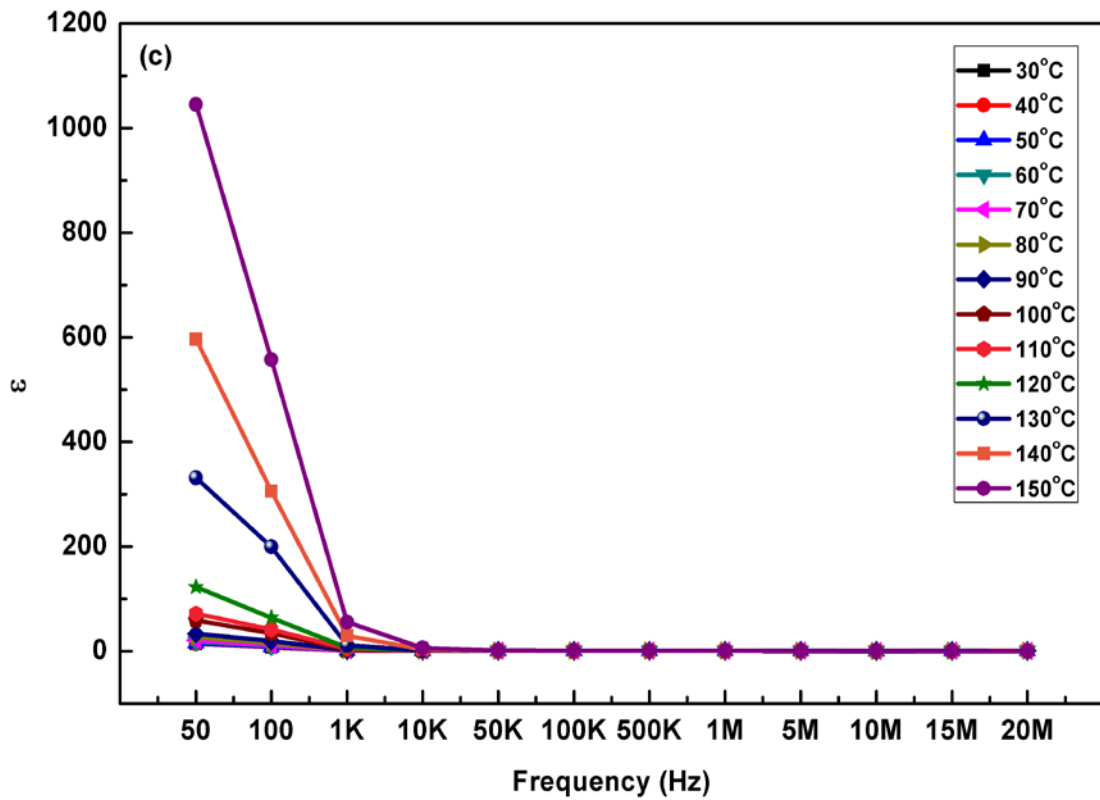


Figure 2(c): Dielectric constant of DBimIL-3b at various frequencies and temperatures



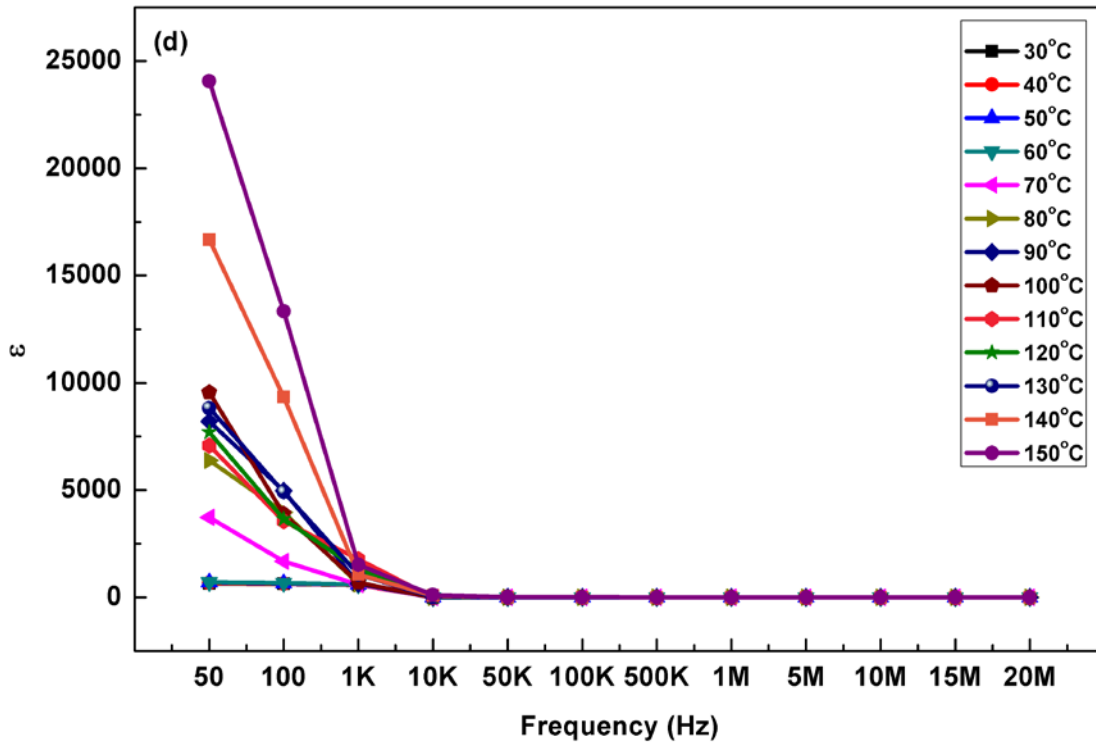


Figure 2(d): Dielectric constant of DBimIL-3d at various frequencies and Temperatures

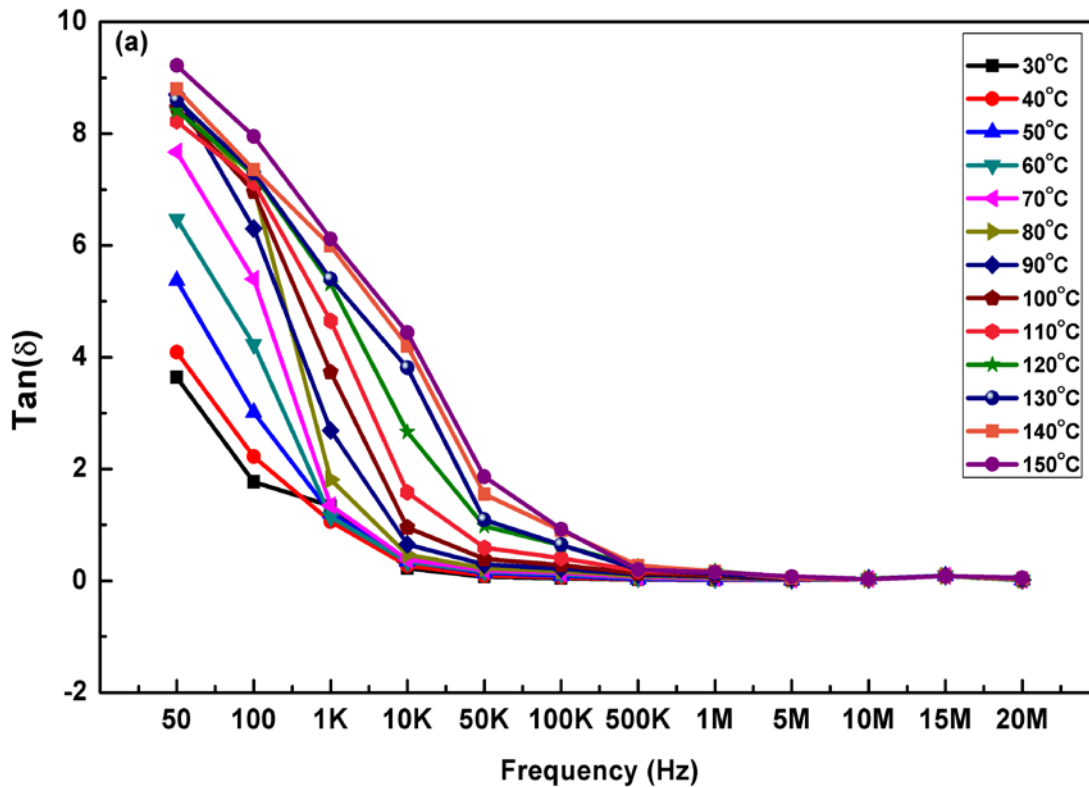


Figure 3(a): Dielectric loss ( $\tan \delta$ ) of DBimIL-3c at various frequencies and Temperatures

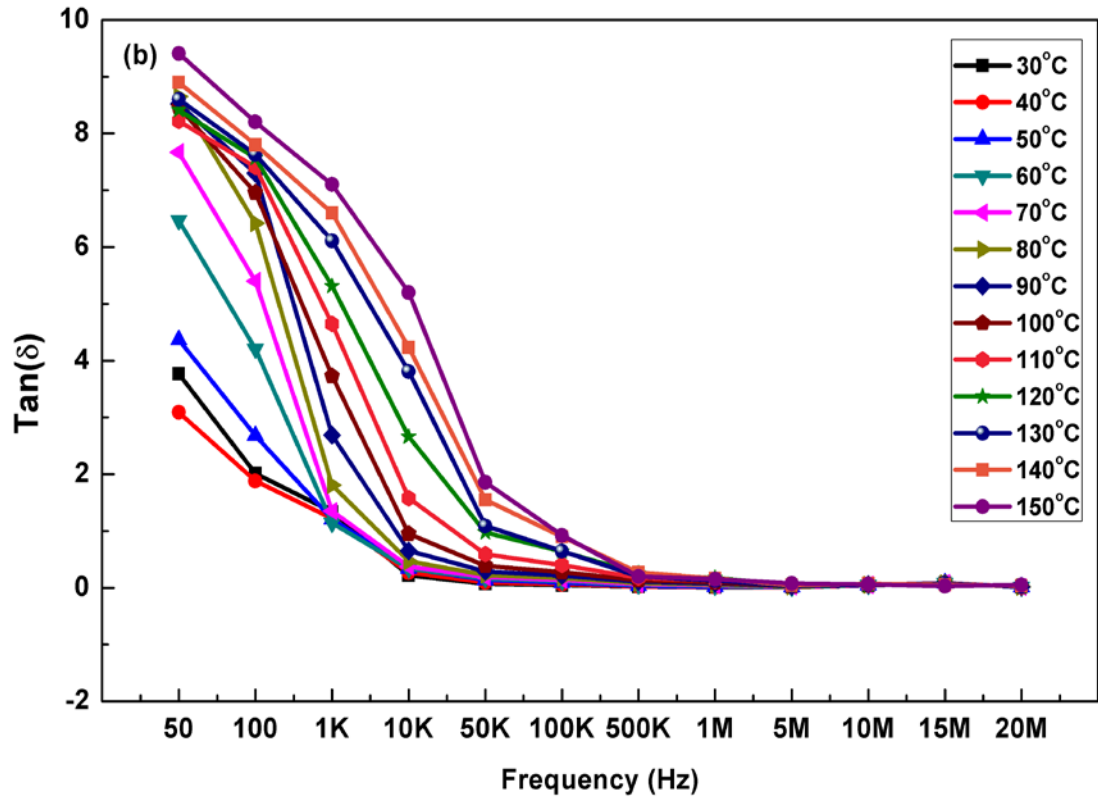


Figure 3(b): Dielectric loss ( $\tan \delta$ ) of DBimIL-3a at various frequencies and temperatures.

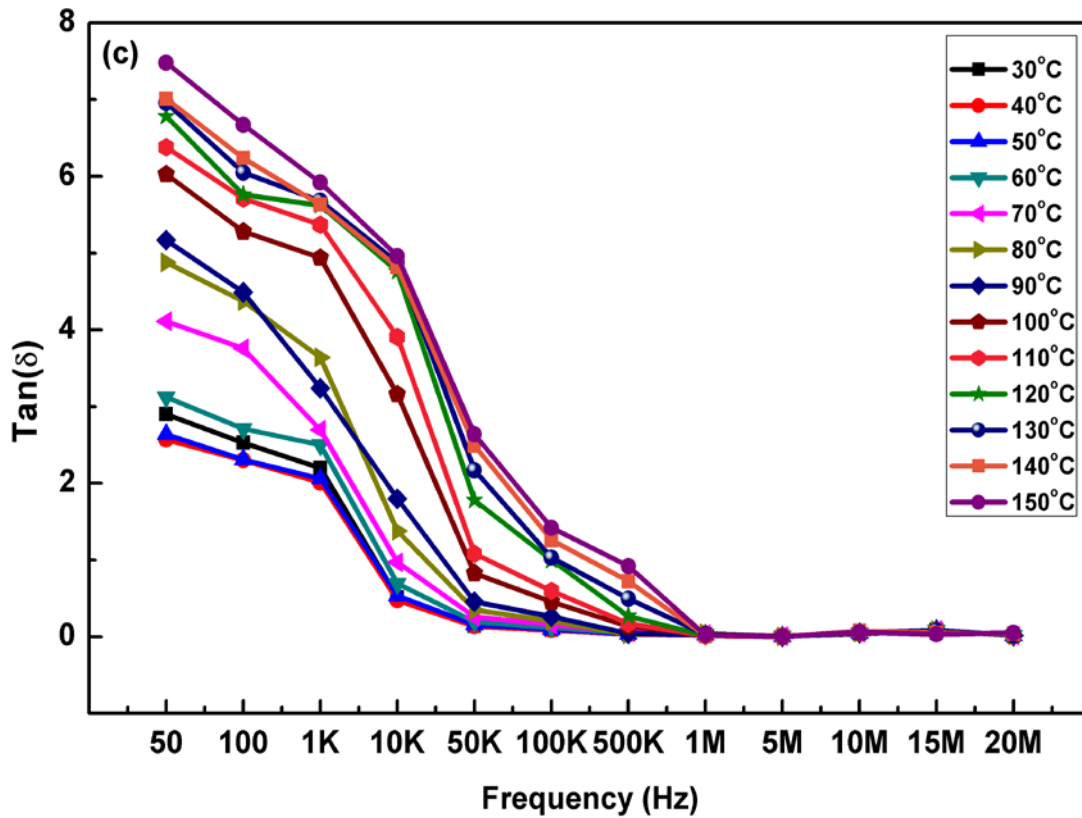


Figure 3(c): Dielectric loss ( $\tan \delta$ ) of DBimIL-3b at various frequencies and temperatures.

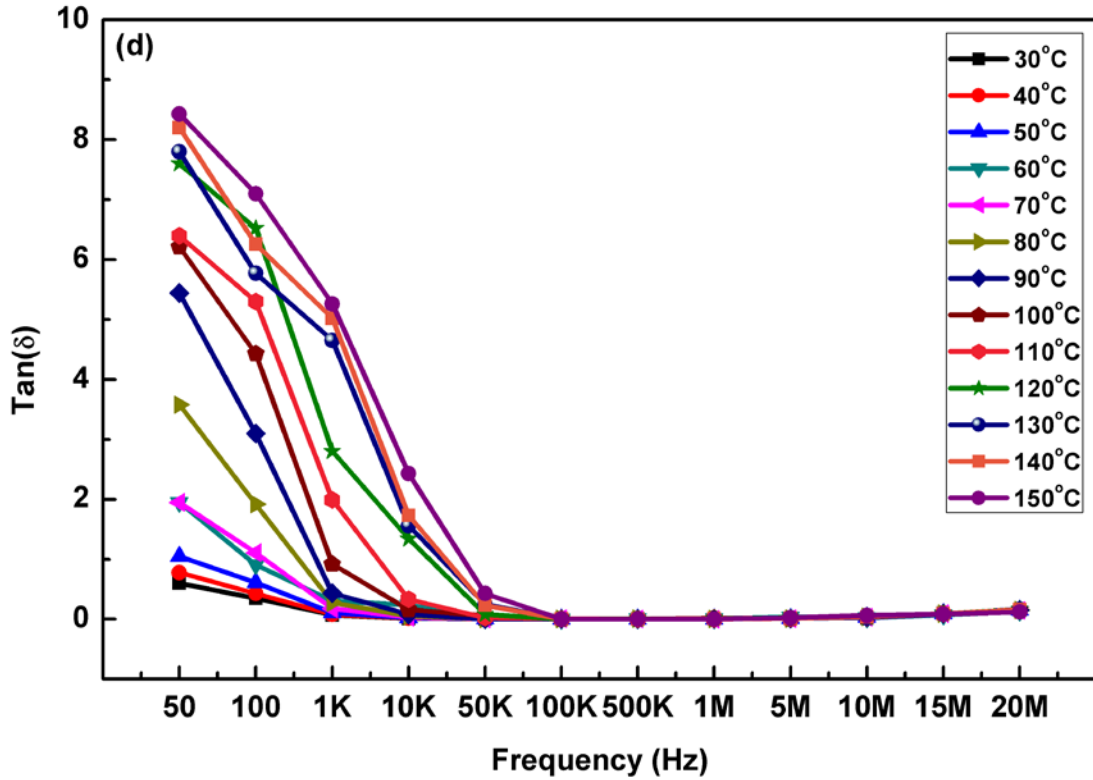


Figure 3(d): Dielectric loss ( $\tan \delta$ ) of DBimIL-3d at various frequencies and temperatures.

Table 1: Comparative values of dielectric constant and dielectric loss of synthesized ionic Liquids

Samples	Dielectric Constant ( $\epsilon$ )	Dielectric Loss ( $\tan\delta$ )
DBimIL-3a	136.47, 50Hz, 150°C	9.41, 50Hz, 150°C
DBimIL-3b	1045.20, 50Hz, 150°C	7.48, 50Hz, 150°C
DBimIL-3c	76.79, 50Hz, 150°C	9.22, 50Hz, 150°C
DBimIL-3b	24059.37, 50Hz, 150°C	8.43, 50Hz, 150°C

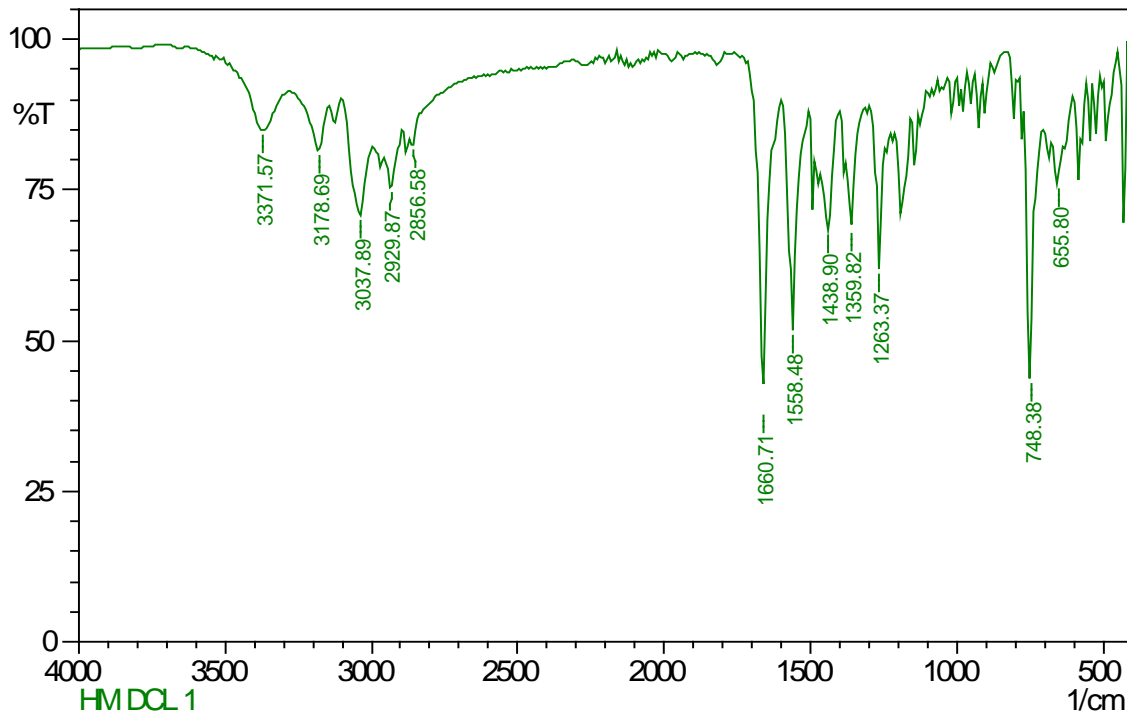


Figure 4a. FT-IR spectra of DBimIL-3a

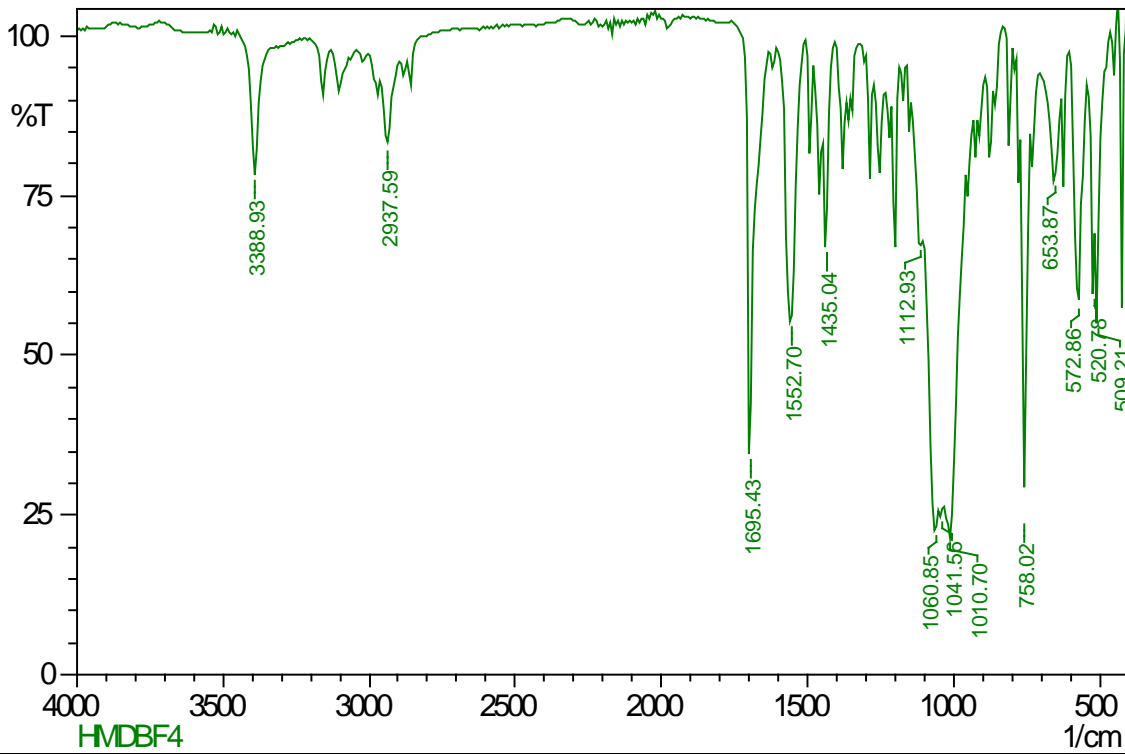


Figure 4b. FT-IR spectra of DBimIL-3b

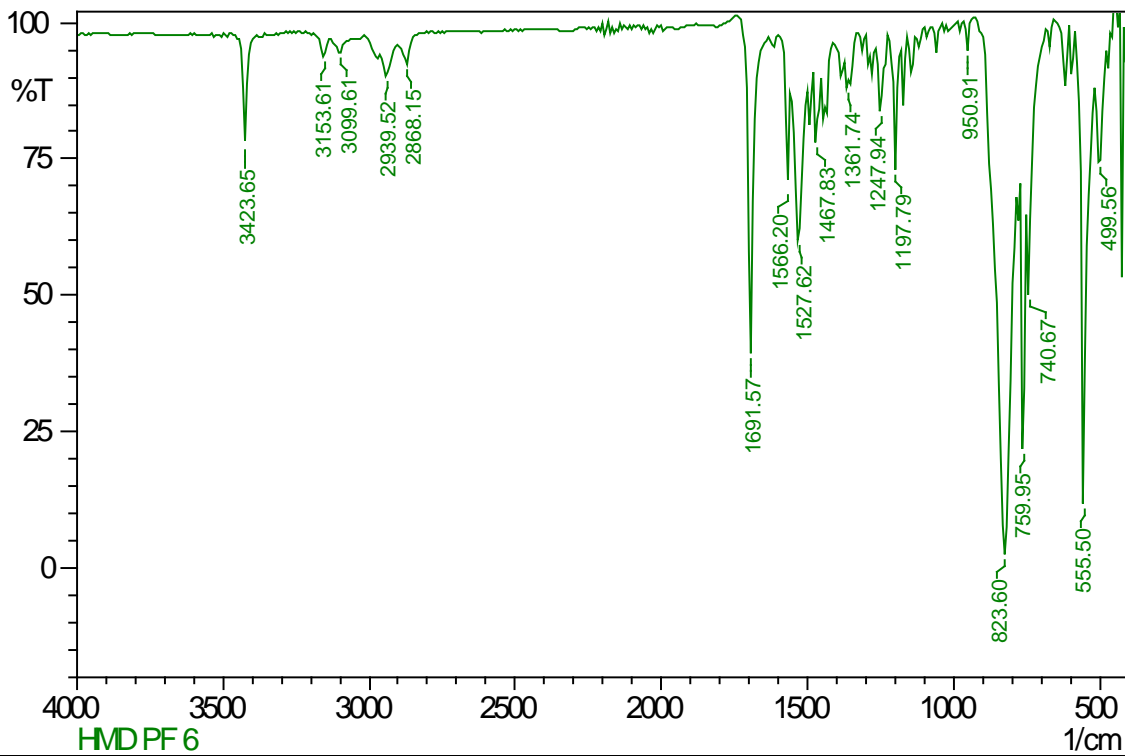


Figure 4c. FT-IR spectra of DBimIL-3c

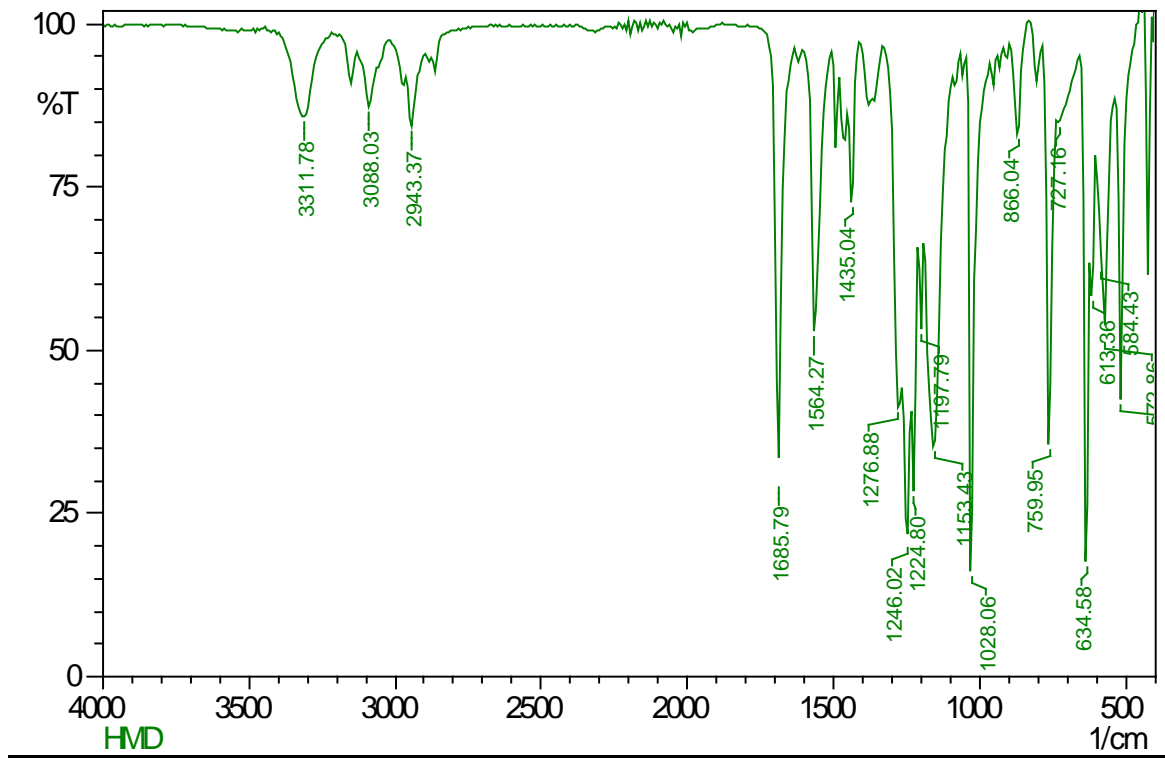


Figure 4d. FT-IR spectra of DBimIL-3d