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AC/DC conductivity and dielectric relaxation behavior of aqueous solutions of 1-butyl-3-methylimidazolium chloride

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The complex relative dielectric function $\varepsilon^*(f) = \varepsilon' - j\varepsilon''$ of aqueous solutions of 1-butyl-3-methylimidazolium chloride [BMiM][Cl] of varying concentrations, has been measured using precision LCR meter in the frequency range 20 Hz to 2 MHz at four different temperatures 293.15, 303.15, 313.15 and 323.15 K. Complex ac conductivity $\sigma^*(f)$ of the liquid samples have been determined from the measured $\varepsilon^*(f)$. DC conductivity σ_{dc} of the samples have also been determined. Values of σ_{dc} at different concentrations have been fitted to the empirical Casteel-Amis (CA) equation. The influence of concentration and temperature variation on the complex permittivity and electrical conductivity of the solutions of [BMiM][Cl] in distilled water has been discussed. The molar conductivities and the infinite dilution conductance of these ionic liquids have also been determined. Orientational relaxation behavior of the aqueous solutions of [BMiM][Cl] has also been studied by measuring complex permittivity in the frequency range 1 GHz to 20 GHz using vector network analyzer. Various processes contributed to the electrical/dielectric properties of the solutions of [BMiM][Cl] in distilled water have been explored.

Keywords: Ionic liquids, Dielectric properties, Electrical conductivity, Precision LCR meter, Casteel-amis model, Vector network analyzer

1 Introduction

Ionic liquids (ILs) have unique and fascinating properties like high thermal stability, high electrical conductivity, non flammability and high heat capacity, that make them a suitable candidate for their applications to modern science and technology[1,2]. Modern time application areas of ILs are as electrolytes (for fuel cells, sensors, super capacitors and batteries), solvents (in bio-catalysis, organic reactions, nanoparticles, polymerization), heat storage, as lubricants and additive, etc.[3,4]. One of the most important property of ILs is large value of ionic conductivity due to the presence of free cations, anions or both[5,6]. ILs are regarded as designer solvents because of the vast number of possible anions and cations to form an IL. ILs of desired conductivity can be designed via mixing[7,8]. Vila *et al.* [9] studied electrical conductivity behaviors of mixtures of some ILs with distilled water over the entire range of concentration and found that the electrical conductivity of the mixtures is higher than the ionic conductivity of the components of the mixtures. Electrical conductivity of the distilled water[10] (0.0092 S/m) at 303 K is comparatively much less than that of the [BMiM][Cl] (0.046 S/m) [11]. So, it will be interesting to study electrical conductivity behavior of diluted aqueous solution of [BMiM][Cl] over a range of concentration. In the present investigation we have taken 1-butyl -3-methylimidazolium chloride as solute and distilled water as solvent and solutions of [BMiM][Cl] of different concentrations were prepared. DC ionic conductivity of the aqueous solutions of [BMiM][Cl] were determined at four different temperatures from measurements of complex permittivity $\varepsilon^*(f)$ of the samples in the frequency range of 20 Hz to 2 MHz. The quantities, molar conductivity ($\wedge_m$) and molar conductivity at infinite dilution ($\wedge^0$) of ionic liquids has received cumulative attention of many researchers in recent past[12–17], because using these properties information on the effect of solvent structure on ionic association and solvation of ionic liquids can be obtained. We have determined molar conductivity ($\wedge_m$) and molar conductivity at infinite dilution ($\wedge^0$) from dc conductivity of the [BMiM][Cl] solutions. Orientational relaxation process of water is studied by many researchers[18–20] and it is observed that dipolar relaxation peak in $\varepsilon''(f)$ spectra falls in the microwave

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*Corresponding author (E-mail: varana@gujaratuniversity.ac.in) range of frequency. To examine the effect of adding ILs in pure water on its orientational relaxation behavior complex permittivity measurements were also carried out in the frequency range of 1 GHz to 20 GHz using VNA and open ended co-axial probe technique.

Aim of the present investigation is to understand various processes contributed to electrical/dielectric properties and underlying physical mechanisms and to understand ionic conductivity and dipolar relaxation behavior of the solutions of [BMiM][Cl] in water.

2 Materials and Methods

HPLC grade [BMiM][Cl] with purity of 99.0% (assay) and distilled water were procured from Otto Chemie Pvt. Ltd. (India) and High Purity Laboratory Chemicals Pvt. Ltd. (India) respectively, and were used without further purification. The aqueous solution of [BMiM][Cl] with water were prepared at nine different concentrations by weight using digital balance with a precision of 1mg.

3 Experimental Details

The complex relative dielectric function $\varepsilon^*(f)$ of liquid samples in the frequency range 20 Hz to 2 MHz were determined by using Agilent E4980A precision LCR meter. A four terminal liquid dielectric test fixture (Agilent 16452 A) was used for capacitance and resistance measurement. The short circuit compensation of the cell and its correction coefficient were considered to eliminate the effect of stray capacitance during the evaluation of the frequency dependent values of complex dielectric function[21]. Details of the experimental method used to determine complex dielectric permittivity and its measurement accuracy are described elsewhere[22]. The low frequency LCR measurements were carried out at four different temperatures (293.15 to 323.15 K). The microwave dielectric measurement of the liquid samples in the frequency range (1 GHz to 20 GHz) were carried out using a Vector network analyzer (VNA) [MS46322A, Anritsu] and DAK 3.5 (Speag) probe[23]. Before performing actual dielectric measurements of the samples, the dielectric probe was calibrated using open, short and dielectric load and the procedure as described in reference[23]. Refractive index of mixtures were measured by using Abbe's refractometer with an accuracy of ± 0.001. VNA and refractive index measurements were carried out at three different temperatures (293.15, 303.15 and 313.15 K). In all the measurements, the temperature was maintained at constant level using a thermostat (MIC Fourtech; India) with standard uncertainty of 0.1 K.

4 Results and Discussion

4.1 Dielectric behaviors in the frequency range 20 Hz to 2 MHz

Figure 1(a and b) shows spectra of $\varepsilon'(f)$ and $\varepsilon''(f)$ for aqueous solutions of different weight fractions of [BMiM][Cl] at 293.15 K temperature, respectively. The spectral features observed in the spectra are caused by the contribution of the charge transport in the bulk and the electrode polarization effect due to formation of interfacial layer at the surface of the metal electrodes of the measuring dielectric cell. With increasing concentration of [BMiM][Cl] in water, $\varepsilon'(f)$ spectra are shifted to right (Fig.1(a)). These spectra are dominated by a large increase of $\varepsilon'(f)$ with decrease in frequency which is typically found in many organic polar liquids[24–26] including ionic liquids[27–29]. This is due to the electrode polarization effect which dominates over

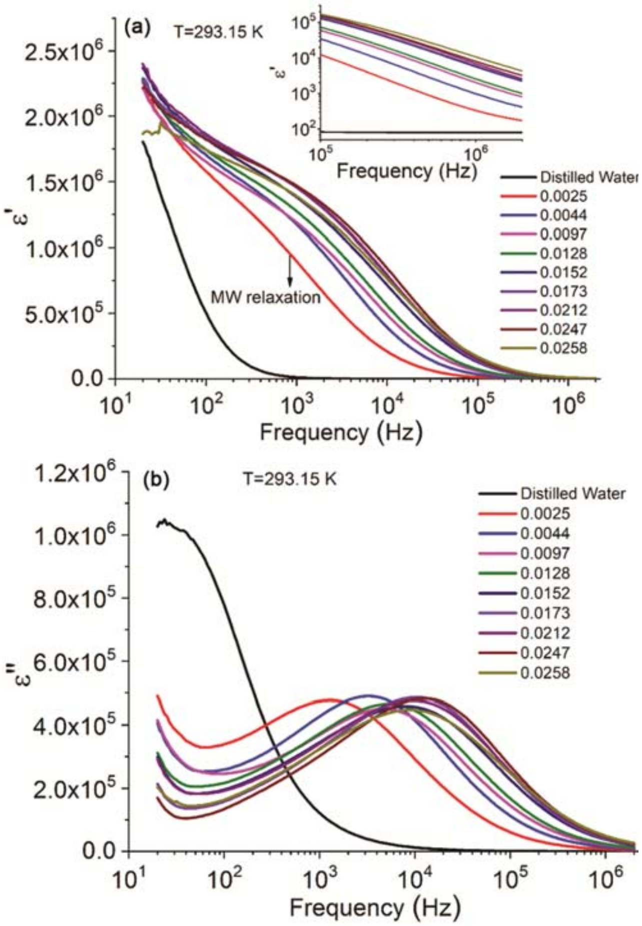

Fig. 1 – Plots of (a) dielectric constant ε' and (b) dielectric loss ε'' against frequency for different weight fractions of [BMiM][Cl] in distilled water at 293.15 K

the bulk property of the sample. The electrode polarization shows up as a so-called Maxwell-Wagner (MW) relaxation step in ε'(f) that should lead to well-defined plateaus at low frequencies[30]. However, in the present case no such distinct plateau is observed in the low frequency region of the spectra. This is due to the presence of several MW- relaxations or by a distribution of MW relaxation[31,32]. The MW relaxation appears as a step in ε' at around 1 kHz frequency which is also observed as a local maxima in the dielectric loss spectra (Fig.1 (b)) at around 1 kHz frequency for 0.0025 weight fraction solution of [BMiM][Cl] and the loss maxima is shifting towards higher frequencies with increasing concentration. The high frequency dielectric loss peaks arise from the dc conductivity which becomes more obvious in the σ'_{ac} against frequency plots by the corresponding frequency independent plateaus as shown in Fig. 2 (a). At lower frequencies, the electrode polarization leads to decrease in conductivity causing decrease in σ'_{ac} values with decrease in frequency. The density of accumulated ions and charges at the interface decreases gradually with increase in frequency of the simulating electric field, and at higher frequencies, it completely diminishes, thereby, the ε'(f) values tend to approach a steady state (static permittivity ε_0). The inset of Fig. 1 (a) shows the enlarged view of ε'(f) spectra in log-log scale in frequency window of 0.1 MHz to 2 MHz. From this inset it can be seen that steady state in ε'(f) value of pure water is reached. But when IL is added into the water ε'(f) spectra is enormously affected due to large contribution of electrode polarization and ionic conductivity of the dissociated cations and anions in the water. Therefore, steady state in the ε'(f) value is not attained up to 2 MHz frequency in [BMiM][Cl] solutions. Static dielectric constant of conductive liquids cannot be determined using low frequency dielectric spectroscopic measurements. It is easier to measure static dielectric constant at microwave frequencies where the conductivity effect is proportionally smaller[33–35]. Therefore, static dielectric constant of aqueous solutions of [BMiM][Cl] were determined by complex permittivity measurements in the 1 GHz to 20 GHz frequency range and they are discussed in the later part of this paper.

The most prominent feature of electrode polarization is observed in ε' and σ''_{ac} versus frequency plots as shown in Fig. 1 (a) and Fig. 2 (b), respectively. Electrode polarization effect comes into play at a certain frequency f_{on}, where a steep increase in ε' corresponding to a minimum in σ''_{ac} which is around 1.41 MHz for 0.0025 weight fraction of [BMiM][Cl]. Frequency at which electrode polarization effect takes place for higher concentration of [BMiM][Cl] seems to fall above 2 MHz frequency and the nature of the curves (Fig. 1(a)) suggests that it increases with increase in the weight fraction of [BMiM][Cl]. At lower frequencies, a plateau in ε' (Fig. 1(a)) starts to develop corresponding to a peak in σ''_{ac} (Fig. 2(b)).

In Fig. 2 (b), for 0.0025 weight fraction of [BMiM][Cl] f_{on} is around 1.41 MHz and f_{on} is found to increase with increase in concentration. Despite these peculiar frequency dependencies, a perfect scaling is observed in σ''_{ac} upon varying concentration. The scaling of σ''_{ac} at 293.15 K temperature for different concentration of [BMiM][Cl] solutions is shown in Fig. 3. The overlap of the spectra for different concentration on a single master curve suggests that the

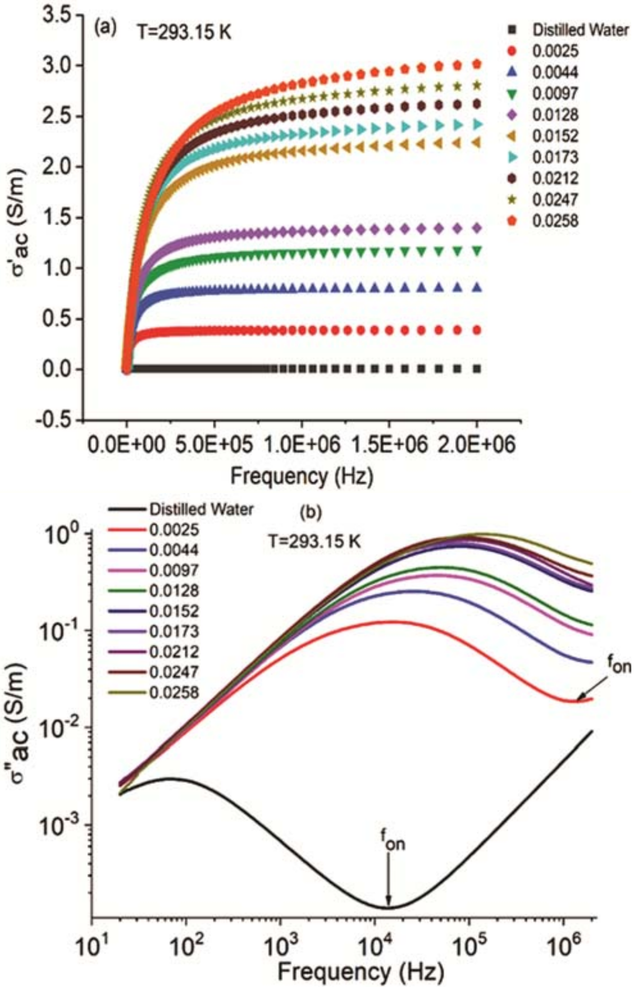

Fig. 2 – Plots of (a) real part σ'_{ac} and (b) imaginary part σ''_{ac} of complex ac conductivity against frequency for different weight fractions of [BMiM][Cl] in distilled water at 293.15 K.

dynamical processes occurring are independent of concentration, the relaxation dynamics of charge carriers follows a common mechanism. Similar scaling behavior in σ''_{ac} with respect to concentration is noticed at all temperatures.

The σ'_{ac} spectra of aqua solution of [BMiM][Cl] have frequency independent plateau in high frequency regime (Fig. 2(a)), which correspond to dc electrical conductivity σ'_{ac}. DC ionic conductivity σ'_{ac} of aqua system evaluated at all temperatures from the extrapolation of high frequency independent plateau of σ'_{ac} spectra to zero frequency[36], are reported in Table 1. DC ionic conductivity of the aqueous solutions is found to increase non-linearly with the concentration of [BMiM][Cl]. To describe the relationship between dc ionic conductivity and concentration, we fitted values of σ_{dc} at different concentrations using the empirical Casteel-Amis[37,38] equation in the mole-fraction scale.

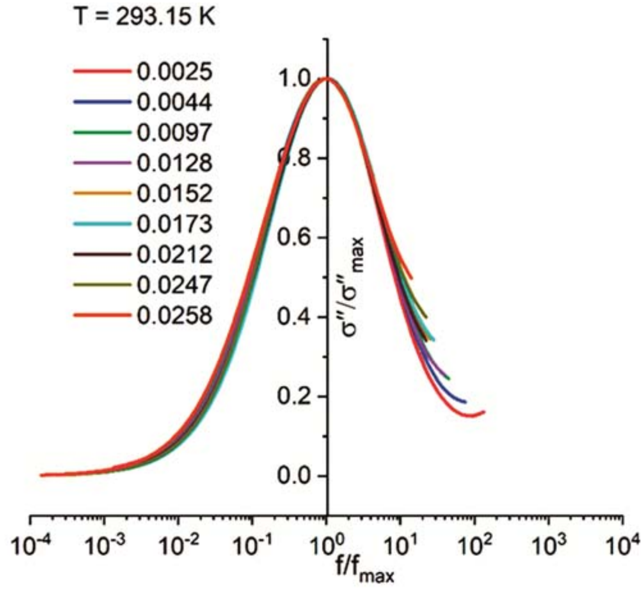

Fig. 3 – Normalized curves of σ''_{ac} against frequency for different weight fraction of [BMiM][Cl] in distilled water at 293.15 K temperature.

$$\sigma_{dc} = \sigma_{max}\left(\frac{X}{X_{max}}\right)^2 \exp\left[b(X - X_{max})^2 - \frac{a}{X_{max}}(X - X_{max})\right] \quad \ldots(1)$$

Where, σ_{max} is the maximum value of the electrical conductivity on the mole fraction scale X_{max}. a and b are the empirical parameters. The fitted values of empirical parameters and the standard deviation (s) are listed in Table 2. From Fig. 4 it can be seen that the electrical conductivity values calculated using Casteel-Amis equation are in good agreement with experimentally determined values.

The conductivity of a sample depends on the charge density (which is high in pure IL) and on the mobility of the ions, which is coupled to viscosity via the Stokes-Einstein relation[39]. As a consequence of their large viscosity pure IL exhibits moderate value of conductivity compared to their binary mixtures with polar solvents. DC ionic conductivity shows a rapid rise at lower concentration of IL due to the increasing number of charge carriers, then after it exhibits a saturation phase due to counter balancing effect of the rapidly rising viscosity on ionic mobility. The other reason for the saturation in the high concentration range could be the formation of aggregate. The aggregation formation becomes dominant factor reducing the number of charge carriers[40,41]. From Table 1, it can be seen that σ_{dc} increases with increase in temperature which suggests

Table 1 – Determined values of dc conductivity (σ_{dc}) at different temperatures and activation energy (E_σ) for different weight fraction of [BMiM][Cl] in distilled water.

Weight fraction of [BMiM][Cl]	Dc conductivity σ_{dc} (S/m)				Activation Energy E_σ (kJ/mol)
	293.15 K	303.15 K	313.15 K	323.15 K	
0	0.007	0.009	0.012	0.012	14.64
0.0025	0.343	0.436	0.469	0.599	13.73
0.0044	0.685	0.849	0.920	1.123	12.32
0.0097	0.982	1.238	1.324	1.661	12.95
0.0128	1.310	1.591	1.720	2.092	11.67
0.0152	1.646	1.998	2.163	2.538	10.87
0.0173	1.943	2.311	2.508	2.769	9.05
0.0212	2.180	2.626	2.862	3.166	9.53
0.0247	2.467	2.833	3.077	3.502	8.93
0.0258	2.491	2.853	3.087	3.520	8.68

Table 2 – Fitted values of the empirical parameters a, b and standard deviation (s) of Casteel-Amis model and empirical constants of molar conductivity for solutions of [BMiM][Cl] in distilled water at different temperatures.

Temperature	Empirical parameters of Casteel-Amis model					Empirical constants of molar conductivity		
(K)	a	b	s	σ_{max} (S/m)	X_{max}	$\wedge^0$ ($\Omega^{-1}cm^2mol^{-1}$)	p	q (10^2)
293.15	0.282	-2883.40	0.052	2.371	0.026	128.82	4.297	-5.971
303.15	0.769	-1681.60	0.093	2.633	0.026	169.46	4.486	-8.488
313.15	0.618	-2014.40	0.099	2.917	0.026	182.81	4.698	-9.544
323.15	0.651	-1737.80	0.061	3.300	0.026	243.90	4.695	-1.333

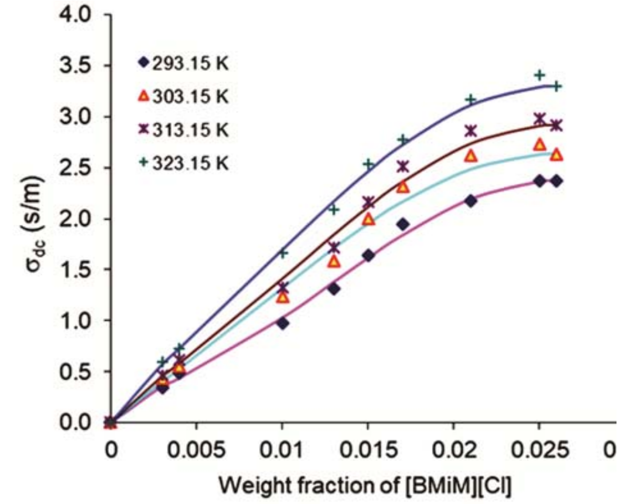

Fig. 4 – Plot of Casteel-Amis model of electric conductivity (σ') against different weight fraction of [BMiM][Cl] in distilled water at different temperatures.

the thermally activated behavior of induced ions in the solutions of [BMiM][Cl] in water and this behavior of σ_{dc} is exactly same as that of the diluted electrolyte solution[42].

On the basis of electrical conductivity studies, the activation energy E_σ of the charge conduction process for aqueous solutions of [BMiM][Cl] can be determined according to the following Arrhenius formula [10];

$$\sigma_{dc}(T) = \sigma_0 \exp\left(\frac{E_\sigma}{RT}\right) \quad \ldots(2)$$

Where, σ stands for electrical conductivity, T the temperature, and R is the gas constant. Calculations were performed based on the linearity of the following relationship;

$$\ln\sigma_{dc} = \ln\sigma_0 + \left(\frac{E_\sigma}{RT}\right) \quad \ldots(3)$$

The Arrhenius plot of the electrical conductivity and the temperature is shown in Fig. 5. The activation energy (E_σ) of solution of different weight fraction of [BMiM][Cl] in water are reported in Table 1.

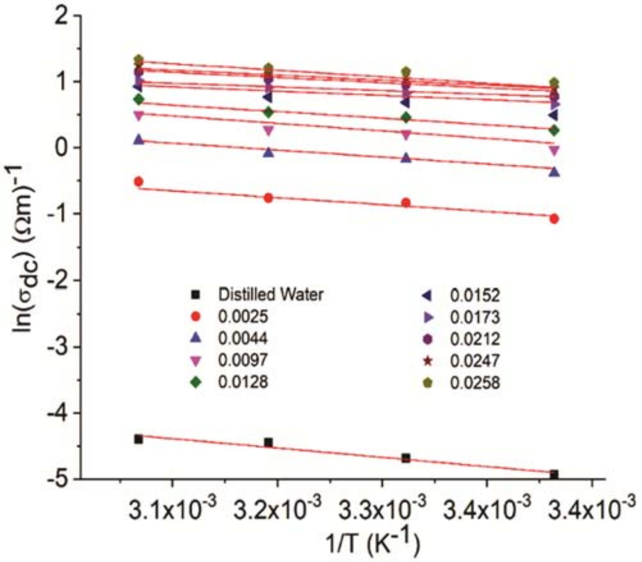

Fig. 5 – Plot of σ_{dc} against inverse of temperature (1/T) for different weight fraction of [BMiM][Cl] in distilled water.

The molar conductivity of the system can be found with the help of dc conductivity by relation $\wedge_m = \frac{\sigma_{dc}}{c}$, where $\wedge_m$ is molar conductivity and c is molar concentration[13]. Figure 6 is the plot of molar conductivity versus square root of molar concentration at different isotherms. The molar conductivity shows linearly decreasing behavior with square root of molar concentration. With increasing amount of ionic liquid, ion association, relaxation and electrophoretic effects arise among anion and cation of ionic liquid, consequently more aggregates of the ionic liquid dispersed in water[14]. The evaluated data of molar conductivity were fitted to following equation proposed by Robinson and Stokes[43]:

$$\wedge_m = \wedge^0 + (p\wedge^0 + q)c^{\frac{1}{2}} \quad \ldots(4)$$

where $\wedge^0$ is molar conductivity at infinite dilution, p and q are empirical constant. Fitted results are shown in Table 2. Molar conductivity at infinite dilution increases with increase in temperature since the mobility of free ions is increased and similar behavior was observed by M. Rogac *et al.*[13] (aqua solution of [Bmim][Cl]), C. Wong *et al.*[16] (aqua solution of [Bmim][X]) and Wang *et al.*[17] ([C$_n$mim][X] in different solvents). However, our values of ^{0} differ from those reported by M. Rogac *et al.*[13] for similar aqua system. The discrepancies in the value of ^{0} may be owing to different technique used to determine dc conductivity by M. Rogac *et al.*[13]. The temperature dependence of molar conductivity at infinite dilution yields Eyring's activation enthalpy of the charge transport[44]:

$$\ln\wedge^0 + \frac{2}{3}\ln d_s = -\frac{\Delta H}{RT} + B \qquad \ldots (5)$$

where d$_s$ is density of solvent, ΔH is activation enthalpy and B is constant. The obtained value of Eyring activation enthalpy is 15.5 kJmol^{-1} and is very close to 15.8 kJmol^{-1} which is reported by M. Rogac *et al.*[13] for the same system.

4.2 Dielectric behavior in the frequency range 1 GHz to 20 GHz

Figure 7(a) and (b) shows logarithmic frequency dependence of ε'(f) and ε"(f) of water and aqueous solutions of [BMiM][Cl] in the frequency range of 1 GHz to 20 GHz at 293.15 K, respectively. With increase in concentration of [BMiM][Cl] ε'(f) spectra are shifted downwards. Static dielectric constants of water and aqueous solution of [BMiM][Cl] were determined by extrapolating the frequency independent region of ε'(f) plots to zero frequency and they are listed in Table 3. Static dielectric constant of water is 80.28 at 293.15 K and it decreases with concentration of [BMiM][Cl]. Variation in static dielectric constant with concentration of [BMiM][Cl] at 293.15 K is shown in Fig. 8. Dielectric constant decrement is due to the formation of ionic aggregates by dipolar interaction between isolated ion pairs and water molecules. This reduces the ability of those water molecules to reorient. With increase in concentration of [BMiM][Cl] more and more water molecules are attached to isolated ion pairs which contribute to the reduction in dielectric constant.

Dipolar relaxation process of water is studied many times and it is well known that the relaxation process is of Debye type[18–20,45]. Dielectric loss spectra of pure water in Fig. 7(b) is in confirmation with these studies exhibiting one relaxation peak at 15.65 GHz

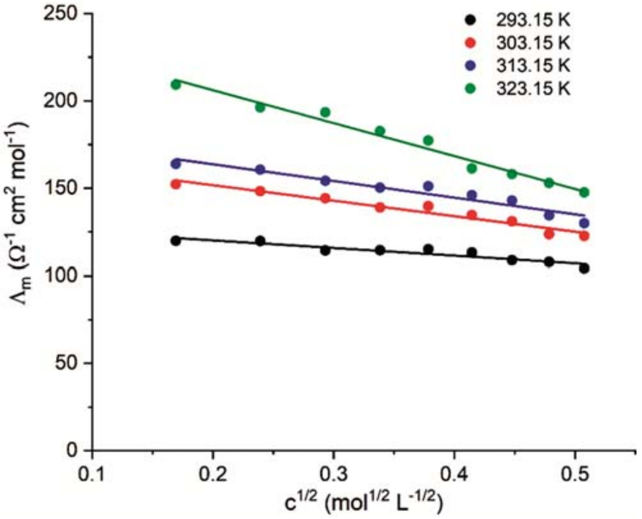

Fig. 6 – Molar conductivity ($\wedge_m$) of [Bmim][Cl] as a function of square root of molar concentration at different temperatures.

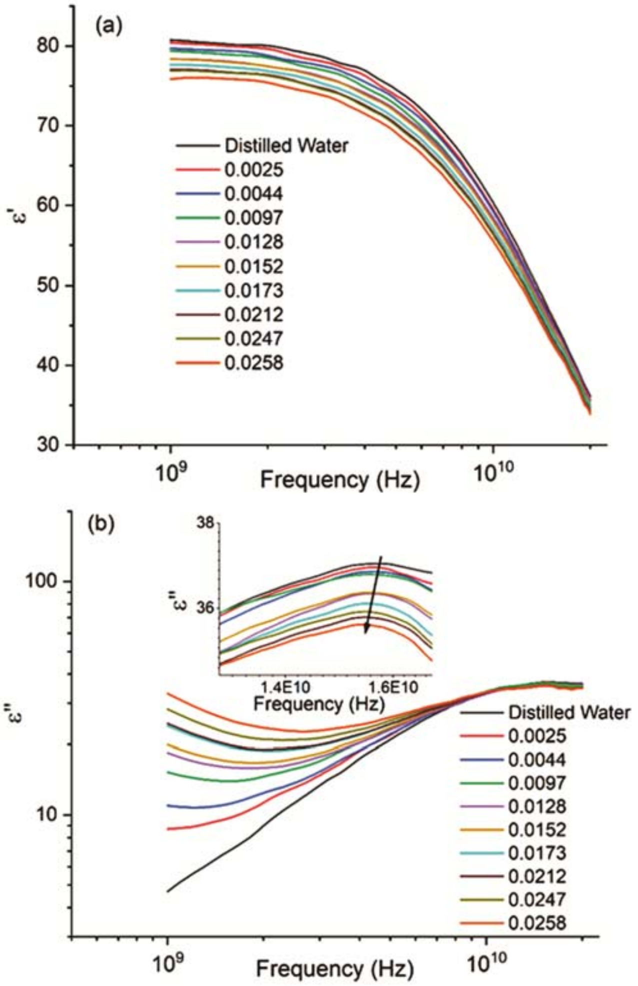

Fig. 7 – Spectra of (a) real and (b) imaginary part of complex dielectric function against high frequency for mixtures of [BMiM][Cl] and water at 293.15 K

Table 3 – Static permittivity (ε_0) and permittivity at optical frequency (ε_∞) and Kirkwood correlation factor (g_{eff}) at different temperatures.

Weight fraction of [BMiM][Cl]	Static permittivity ε_0			Permittivity at optical frequency ε_∞		
	293.15 K	303.15 K	313.15 K	293.15 K	303.15 K	313.15 K
0	80.28	76.13	73.63	1.7689	1.7718	1.7676
0.0025	79.99	76.00	73.24	1.7750	1.7721	1.7694
0.0044	79.52	75.95	72.87	1.7788	1.7764	1.7740
0.0097	78.83	75.81	72.62	1.7873	1.7849	1.7825
0.0128	78.26	75.66	72.37	1.7910	1.7886	1.7862
0.0152	77.95	75.57	72.07	1.7940	1.7916	1.7892
0.0173	77.31	75.12	71.71	1.7967	1.7943	1.7918
0.0212	76.72	75.09	71.55	1.8007	1.7983	1.7959
0.0247	76.24	74.97	71.24	1.8034	1.8010	1.7985
0.0258	75.87	74.51	70.79	1.8042	1.8018	1.7994

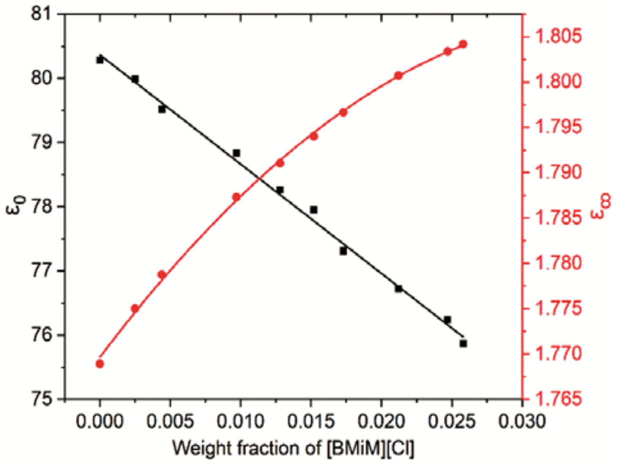

Fig. 8 – Plots of static permittivity (ε_0) and permittivity at optical frequency (ε_∞) against weight fraction of [BMiM][Cl] at 293.15 K temperature.

frequency, however, dielectric loss spectra of aqueous solutions of [BMiM][Cl] exhibit two process leading to energy absorption. These are translational motion of ions causing ionic conduction loss and dipolar loss due to rotational motion of water molecule. As the concentration of [BMiM][Cl] increases the contribution to ionic conduction loss dominates over dipolar loss and consequently ε"(f) values increase with decreasing frequency. The dielectric loss peak frequency of the relaxation process of water shifts towards lower frequency with increase in concentration of [BMiM][Cl] (inset of Fig. 8(b)). Addition of [BMiM][Cl] affects the water relaxation region. As concentration of [BMiM][Cl] increases a higher amount of water is influenced by inter-molecular forces arising from the ion pairs, which causes dielectric dispersion at frequencies less than the pure water.

Determined values of permittivity at optical frequency (ε_∞) at 293.15, 303.15 and 313.15 K temperatures are reported in Table 3. Variation in ε_∞ with concentration of [BMiM][Cl] at 293.15 K temperature is shown in Fig. 8. From this figure it can be seen that the ε_∞ values increases non linearly with concentration of [BMiM][Cl]. Non-linearly in this plot clearly indicates that the electronic polarization of water molecules is influenced due to interaction between water molecules and ion pairs of [BMiM][Cl].

5 Conclusions

Two relaxation processes corresponding to ionic conduction and electrode polarization phenomena were observed, in aqueous solution of 1-butyl-3-methylimidazolium chloride [BMiM][Cl] in the frequency range 20 Hz to 2 MHz. In the low frequency region, ionic conduction and electrode polarization have a dominant influence, leading to the large values of complex dielectric constant at all temperatures of measurement. Perfect scaling was observed in σ''_{ac} against frequency spectra with respect to concentration variation. Concentration dependence of dc ionic conductivity (σ_{dc}) could be well fitted to the Casteel-Amis equation, at all four temperatures. Molar conductivity ($\wedge_m$) and molar conductivity at infinite dilution ($\wedge^0$) were also determined and reported. These quantities can be used to gain information of the effect of the solvent structure on the ionic association and solvation of ionic liquids. Static dielectric constant of aqueous solution of [BMiM][Cl] was determined from complex permittivity measurements in the microwave frequency range and was found to decrease with concentration of [BMiM][Cl]. Dipolar relaxation peak of water observed in the dielectric loss spectra shift towards lower frequency side with concentration of [BMiM][Cl].

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