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Ionic Conduction Characteristics of C_8mimX Ionic Liquids and Their Hybrids Towards Application to Charge and Lithium-Ion Storage

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Abstract: *Room temperature ionic liquids (RTILs) have attracted much attention in electrochemical energy storage systems for their advantageous properties over traditional lithium salt/carbonate solvent electrolytes in terms of negative vapor pressure and non-flammability. RTILs can be used as the solvent-free electrolytes in electrochemical double layer capacitors or act as the important additives to the carbonate electrolytes in lithium-ion batteries and lithium-ion capacitors. Compared with the common 1-ethyl-3-methylimidazolium (C_2mim^+)-based systems, long-chained RTILs are advantageous for their high electrochemical stability (up to 7V vs Li/Li^+) which have the potential to double the operating voltage and energy density. This study is to report ionic conducting characteristics of 1-octyl-3-methylimidazolium (C_8mim^+) based RTILs, i.e. C_8mimX (X = BF_4^-, PF_6^-, and Cl^-) and their hybrids with diethyl carbonate (DEC) solvent as well as the traditional Li-ion electrolyte like 1M $LiPF_6$-EC/DEC (1:1) at various preset molar ratios. The influences of the hybrid electrolyte composition and temperature on the ionic conduction characteristics were systematically studied.*

Keywords: ionic liquids, conduction, hybrids, molar composition, activation energy, energy storage

1. INTRODUCTION

Electrochemical energy storage systems play a dominant role in electric and hybrid transportation. Recently, lithium-ion batteries (LIBs) and electrochemical double layer capacitors (EDLCs) have been equipped on some models of vehicles such as Tesla and Chevrolet Volt etc. Along with the continuous development towards improving energy and power density of LIBs and EDLCs, the safety issue raises much attention. Improvement of electrolytes to endure overheating and/or overcharging is one of the crucial research area. Compared with conventional carbonate-based organic electrolytes, room temperature ionic liquids (RTILs) are excellent in terms of broad electrochemical stability window, negligible volatility, non-flammability, and thermal stability. These merits are attractive as either electrolyte alternatives or additives towards improving energy output of LIBs and EDLCs, extending their operational temperatures, and alleviating thermal runaway hazard [1-8].

However, many common RTILs possess a few intrinsic properties which restrict their direct applications to energy storage. Long-chained immidazolium (IMI) or piperidinium (PP) based RTILs exhibit insufficient ionic conductivities at ambient temperatures [9-11]. These RTILs also have poor wettability to the common separator and electrode surface leading to high electrolyte and interface impedances [12]. For applicable to LIBs and lithium-ion capacitors (LICs), addition of Li-salt in RTILs is indispensable to render lithium-ion transport between electrodes but at the sacrifice of conductivity [13-17]. Thanks to the broad solute/solvent capabilities, RTILs' properties can be tuned via mixing with other agents, such as different ILs [18-21] or aprotic solvents [22-29]. Upon addition of aprotic solvents, e.g. ethylene carbonate (EC), propylene carbonate (PC), to RTILs including IMI, PP, or pyridinium (PYR) series, improved conductivities and wettability were reported [12, 23-25].

In this paper, we will present our studies on ionic conducting characteristics of 1-octyl-3-methylimidazolium (C_8mim^+) -based RTILs with different anions, i.e. C_8mimX (X = BF_4^-, PF_6^-, and Cl^-). The C_8mimX system is selected based on its higher electrochemical stability (up to 7V vs Li/Li^+) than the common 1-ethyl-3-methylimidazolium-based system (C_2mimX) [30,31]. Moreover, the ionic conductivities of their binary hybrid systems with the addition of diethyl carbonate (DEC), a common solvent used in the electrolyte of LIBs and LICs, are studied. Past studies on adding pure lithium salts into neat RTILs constantly revealed that ionic conductivities reduced significantly with the increase in the lithium salt concentration. This was attributed to an increase in the viscosity of the liquid. In this study, we added traditional lithium electrolyte solution, i.e. 1M $LiPF_6$ - EC/DEC (1:1), into C_8mimPF_6

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*To whom correspondence should be addressed: Email: hong.huang@wright.edu in consideration of both providing lithium ion source and meanwhile reducing the viscosity of the hybrid. We will present and discuss the conductivities changing with temperature and molar percentage of the neat RTIL in the hybrid systems. It is hopeful to provide experimental database and guidance towards their potential applications to charge and/or Li-ion storage.

2. EXPERIMENTAL ASPECTS

The RTILs used in this study, i.e. C_2mimBF_4, C_8mimBF_4, C_8mimPF_6, and C_8mimCl were purchased from Changchun, China. The DEC solvent was from Sigma Aldrich, USA and the lithium electrolyte 1M $LiPF_6$-EC/DEC (1:1 vol%) was from MTI International, USA. Some key properties of these chemicals, reported in literatures, such as density, molecular weight, ion molar concentration, conductivity and viscosity, are summarized in Table 1.

All the liquids used in this study were dried with 3Å molecular sieves and handled in an Argon filled glove box with a controlled moisture less than 5ppm. For the hybrid liquids, an appropriate volume of the RTIL was mixed with the other agent at the predetermined volume ratio. After stirring and shaking to ensure no separation, the solution was sealed and rested for at least an hour prior to proceeding the conductivity measurements. The molar fraction of solvent in the hybrid solution, x_s, was computed based on the volume (V), density (D), and molecular weight (Mw) of each component (subscript 1 for the additive liquid and 2 for RTIL),

$$x_s = \frac{V_1 D_1 / Mw_1}{V_1 D_1 / Mw_1 + V_2 D_2 / Mw_2} \tag{1}$$

The ionic conductivities were computed based on the cell constant (area and thickness) and the resistance determined with the help of electrochemical impedance spectroscopic (EIS) analyses. Swagelok cells, consisting of a porous polyethylene separator soaked with the studied neat RTILs or their hybrid liquids sandwiched between two same stainless steel electrodes, were assembled in the glove box. The cells were placed in an oven with controlled temperature from 25°C to 65°C with the 10°C interval. Gamry electrochemical system (Reference 600) was used for the EIS measurement. All impedance spectra were collected at the potentiostat mode in the frequency range from 1 Hz to 1000 kHz. The impedances were recorded after the cells fully reached thermal equilibrium at each temperature setting.

3. RESULTS AND DISCUSSION

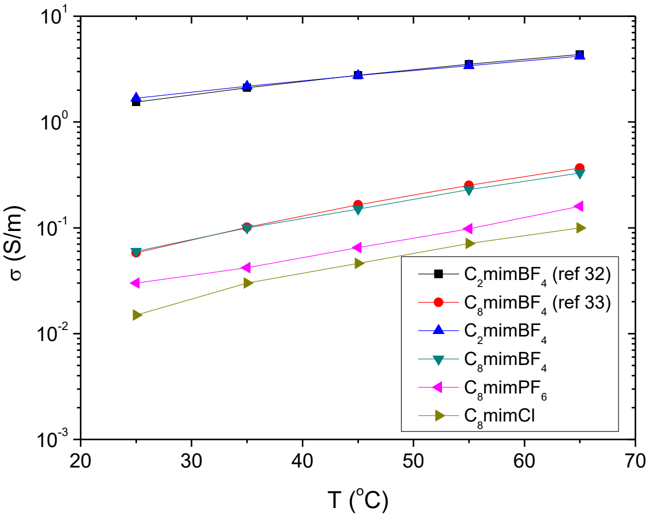

Figure 1. Conductivities as a function of temperature of obtained from C_2mimBF_4 and C_8mimX, compared with data from references.

Figure 1 presents the conductivities as a function of testing temperatures of the four different neat RTILs, i.e. C_2mimBF_4, C_8mimBF_4, C_8mimPF_6, and C_8mimCl. The conductivity values of C_2mimBF_4 and C_8mimBF_4 obtained in this work corroborate well with the published data [32, 33]. The slight discrepancies could result from small variations of temperature and/or the cell configurations in different laboratory settings. As seen in the figure, the conductivities of C_2mimBF_4 are over one order of magnitude higher than those of C_8mimBF_4. The long alkyl chain in C_8mimBF_4 increases the van der Waals interactions among cations by means of the dielectric polarization as well as interactions between hydrocarbon units. The forces increase drag/friction during cation movement, rendering the significant increase of viscosity (see Table 1). Since diffusivity of an ion is determined by the Stokes-Einstein equation,

$$D_i = \frac{kT}{c\pi\eta r_i} \tag{2}$$

where k is the Boltzman constant, T is the absolute temperature, C is a constant, η is the fluid viscosity, and r_i is the effective radius. The increased viscosity reduces both cation and anion diffusivities. The enlarged cations further reduces the cation ion mobility. Although the long alkyl cation chain in C_8mimBF_4 reduces molar concentration by about one fold due to the reduced density and increased molecular weight (see Table 1), the absolute low conductivities in C_8mimBF_4 indicate that viscosity and ion size in RTILs are the dominant factor impacting the net conductivities.

Table 1. Density (D), molecular weight (Mw), ion molar concentration (c=D/Mw), viscosity at 25°C (η), and ionic conductivity at 25°C (σ) of the neat chemicals investigated in this work. The values were obtained from reference [32].

	D (g/cm^3)	Mw (g/mol)	c (M)	σ (S/m)	η (mPa.s)
C_2mimBF_4	1.28	198	6.46	1.4	40
C_8mimBF_4	1.08	282	3.83	0.06	135
C_8mimCl	1.00	231	4.33	TBD	337
C_8mimPF_6	1.24	340	3.65	TBD	710
DEC	0.98	118	NA	NA	0.75
1M $LiPF_6$/EC-DEC	1.26	103	1	0.85	3.6

It is also seen in Figure 1 that the conductivities of the three RTILs with the same cation but different anions increase in the order of BF_4^- > PF_6^- > Cl^-. As discussed previously, the larger the ion the stronger the van der Waals interaction, and hence the higher the viscosity. The volumes of PF_6^- and BF_4^- anions are 0.109 nm^3 and 0.073 nm^3, respectively [34], which agree with the higher viscosity of C_8mimPF_6. Experimentally, it was determined that the self-diffusion coefficient of BF_4^- is higher than that of PF_6^- in C_4mimX [35]. Both the smaller size and faster diffusion coefficient of BF_4^- anion can be attributed to the higher conductivities of C_8mimBF_4 than those of C_8mimPF_6. Although Cl^- anion (0.046 nm^3) is much smaller than BF_4^- , there exists strong H-bonding between Cl^- anion and hydrocarbon chain, which is the consequence of its higher viscosity and lowest conductivity among the

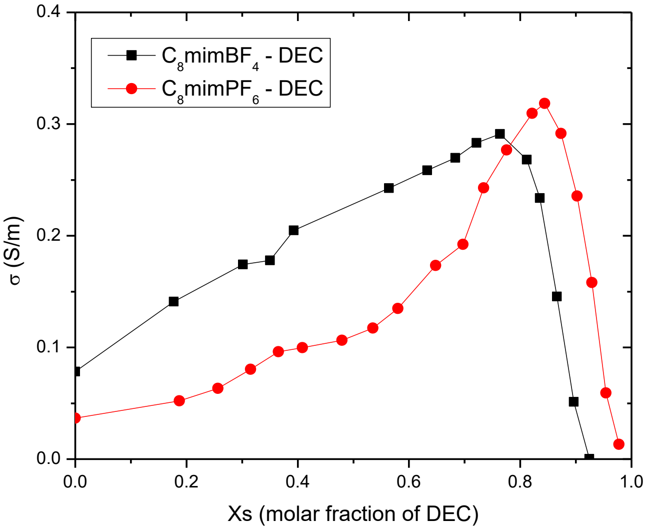

Figure 2. Conductivities of C_8mimBF_4 – DEC and C_8mimPF_6 – DEC hybrids as a function of the DEC molar fraction.

three anions [36,37].

The conductivities of the neat RTIL series as a function of temperature were fitted with VTF and Arrhenius equations,

$$\sigma = A\exp[-\frac{B}{T-T_o}] \tag{3}$$

$$\sigma = \sigma_o \exp[-\frac{Ea}{RT}] \tag{4}$$

The fitting accuracy (R^2) and the resulting key parameters, i.e. ideal glass transition temperature To and practical activation energy Ea, are listed in Table 2. In general, VTF is more suitable for the RTILs because the imidazolium-based RTILs belong to the glass forming system and the conductivity changing with temperature is determined by the inherent viscosity properties. The obtained to values are consistent with other reports showing around 30-40K lower than the actual Tg values [33, 38]. Since Arrhenius equation also provides reasonable fitting results within the experimental temperature range, the pseudo conduction activation energy (Ea) can be derived. It is seen that the long cation chain leads to higher To but lower Ea suggesting relatively weak ion-pair binding energies. In the C_8mimX series, both To and Ea increase in the anion sequence of $BF_4^- < PF_6^- < Cl^-$, which is consistent with the extent of van der Waals force and Hydrogen bonding.

When ILs were mixed with DEC solute, it was observed that C_8mimPF_6 is completely miscible in DEC but there exists maximum miscibility for C_8mimBF_4 and C_8mimCl in DEC which are in the vicinity of 92 mol% and 33 mol%, respectively. Figure 2 displays the conductivities of C_8mimBF_4 and C_8mimPF_6 with various DEC molar fraction x_s. Several interesting characteristics are observed in the two hybrid systems.

Table 2. The resulted fitting data using VFT equation and Arrhenius equation, respectively.

	Tg (K)	VTF Fitting		Arrhenius Fitting	
		To (K)	R^2	Ea (kJ/mol)	R^2
C_2mimBF_4	182±2	140±5	0.9998	65.5	0.998
C_8mimBF_4	193±1	150±5	0.999	35.5	0.998
C_8mimPF_6	200±2	160±5	0.999	38.8	0.996
C_8mimCl	207±7	180±5	0.998	39.2	0.987

For both hybrid systems, the solution conductivities gradually increase to a maximum point with the increase of DEC molar fraction but then sharply decrease to zero upon further increasing DEC content. The observed phenomenon is the consequence of the two competitive impacts, i.e. increasing charge carrier mobility and decreasing the charge carrier concentration, with the addition of DEC into the IL system. The two factors vary with the composition of the mixture and affect the conductivity in an opposite way. In neat RTILs, there exist a large amount of cation-anion pairs. Initially adding a small amount of DEC may slightly enhance the association due to the weak polarity of DEC whose dielectric constant is 2.8. However, addition of DEC significantly reduces the viscosity of the hybrid liquid because the viscosity of DEC is only 0.75 which is at least two orders of magnitude lower than those of RTILs.

According to Seddon's empirical equation [39], the viscosity of a hybrid liquid η reduces exponentially with the molar fraction x_s of the solute in the mixture, $\eta = \eta_{IL}\exp(-Ax_s)$, where A is a characteristic constant and η_{IL} is the viscosity of neat ionic liquid. Since the RTILs obey Walden's rule, the molar conductivity Λ will exponentially increases with x_s, i.e. $\Lambda = \Lambda_{IL}\exp(Ax_s)$. Apparently, the exponential increase in molar conductivity with the solute content will supersede the slight enhancement in the cation-anion association. On the other hand, adding molecular solvent into neat RTILs will linearly reduce the charge carrier concentration c as a function of the DEC molar fraction x_s. Therefore, the conductivity σ, where $\sigma = \Lambda * c$, in relation with the molar fraction of DEC can be expressed as the following,

$$\sigma = \sigma_{IL}(1-x_s)\exp(Ax_s) \tag{5}$$

According to equation (5), the conductivity s will exhibit Exponential increase with DEC molar fraction until the maximum value at $x_{s,max}$. After passing through the pronounced maximum, the decrease in ion concentration plays a dominant role, leading to reduced conductivities. The $x_{s,max}$ varies depending on the values of characteristic constant A which is determined by the magnitude of interactions between the cation- anion and ion-solvent in the hybrid system. In the C_8mimBF_4 - DEC and C_8mimPF_6 - DEC hybrids, the values of $x_{s,max}$ are determined to be 0.78 and 0.86, respectively. Similar phenomenon was reported by Zhu et al [27] in the RTIL-solute binary systems consisting of imidazolium-based IL and the common solutes like water, ethanol and acetone. The molar fraction of the solute at the maxima $x_{s,\,max}$ is at 0.8. Kalugin [28] and Stoppa [40] also reported the maxima is in the range of 0.75 to 0.9 in the C_nmimBF_4-acrynitrile systems (n = 2, 4, 6).

Seen in Figure 2, C_8mimBF_4 system has higher conductivities than the C_8mimPF_6 in the viscosity-dominant region, but the conductivities of the C_8mimPF_6 system increase faster and eventually exceed that of C_8mimBF_4 system around the maximum point. In the region passing through the maxima which the concentration-dominant, the C_8mimPF_6 system remains outperformed C_8mimBF_4 in conduction. In the viscosity-dominant region, the conductivity increase rate is determined by the characteristic constant A. The A values are affected not only the activation energy for viscous flow of liquid but also the interaction between IL and the molecule solutes [27]. The strong interaction will hinder the ion to overcome the attraction with its neighbors leading to the decrease of A value. Wang et al [22] and Katsuta [29] reported that the association constant of the C_nmimBF_4 was greater than C_nmimPF_6 when they were

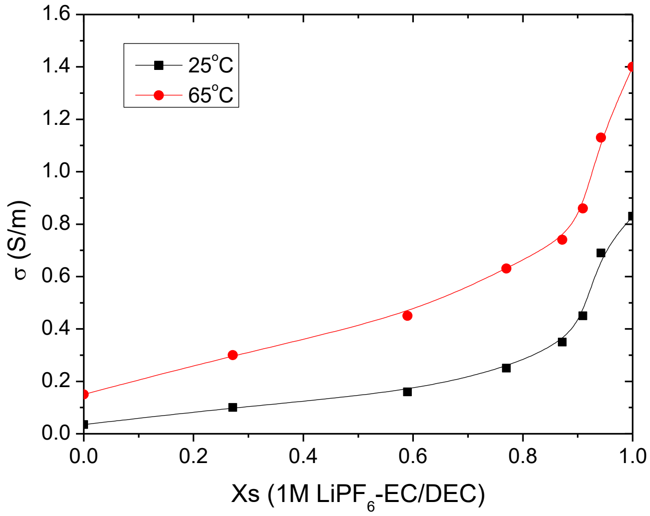

Figure 3. Conductivities as a function of molar fraction of the electrolyte 1M LiPF$_6$ - EC/DEC added into the C$_8$mimPF$_6$ ionic liquid, obtained at 25°C and 65°C.

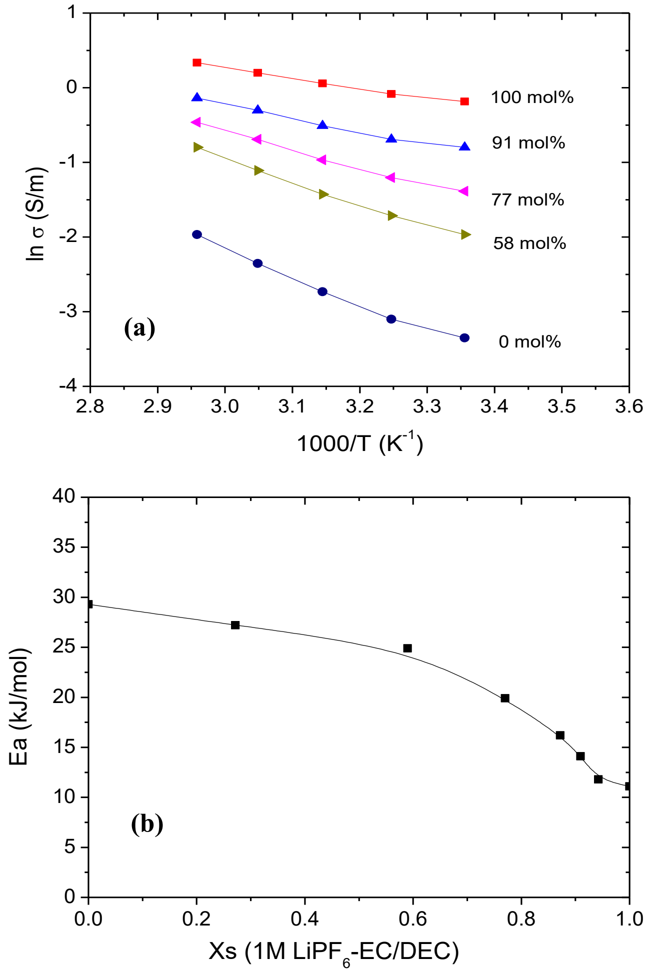

Figure 4. (a) Conductivities as a function of temperature in Arrhenius plots (b) pseudo conduction activation energy as a function of molar fraction of electrolyte 1M LiPF$_6$-EC/DEC added in the C$_8$mimPF$_6$ IL.

mixed with molecular solvent. BF$_4^-$ anion is readily solvated by the DEC, reflecting the slow increase rate in the viscosity region. In the concentration-dominant region, most BF$_4^-$ anion are solvated with DEC resulting in lower mobility and hence lower conductivities.

Figure 3 shows conductivities of the RTIL/electrolyte hybrid solution as a function of the molar fraction of the lithium electrolyte 1M LiPF$_6$ - EC/DEC. It is interesting to note that at $x_s < 0.8$, the conductivities increase with x_s following the same trend of C$_8$mimPF$_6$-DEC and the conductivity values are also very close. This phenomenon suggests that the viscosity is still the dominant factor influencing the ion conductivities. But in the concentration-dominant region, the conductivities rise rapidly. It is submitted that in this region the sufficient amount lithium ions in the EC/DEC solvent actively contribute to the conductivities.

Figure 4 (a) presents the conductivities of the IL-electrolyte hybrids of various concentrations changing with the temperature. All these hybrids exhibit approximately linear relationship in the Arrhenius plots, from which the pseudo activation energy were calculated. Figure 4(b) shows the activation energy as a function of the molar fraction of the electrolyte. Consistent with the observation in conductivity, in the viscosity-dominant region, the hybrids have activation energies over 20 kJ/mol due to the highly activated process of the viscous flow. In the concentration-dominant region, Li ion conduction in the EC/DEC concentrated hybrid liquid is dominant resulting in low activation energy of 12 kJ/mol.

4. CONCLUSION

In this study, ionic conducting characteristics of 1-octyl-3-methylimidazolium (C$_8$mim$^+$) based RTILs, i.e. C$_8$mimX (X = BF$_4^-$, PF$_6^-$, and Cl$^-$), and their hybrids with diethyl carbonate (DEC) solvent as well as the traditional Li-ion electrolyte like 1M LiPF$_6$ - EC/DEC (1:1) are systematically studied. The conductivities of the three neat ILs showed the increase in the sequence of C$_8$mimCl < C$_8$mimPF$_6$ < C$_8$mimBF$_4$, consistently with those in the lower chain C$_n$mimX (n = 2, 4, 6) systems. The smaller size and faster diffusion coefficient of BF$_4^-$ contribute to the highest conductivity in C$_8$mimBF$_4$, while hydrogen bonding existed in C$_8$mimCl is attributed to its lowest conductivity and highest conduction activation energy. Addition of the carbonate solvent DEC significantly influenced the ILs' conductivities. The two competitive factors, i.e. reducing the viscosity and reducing the IL concentration, render the characteristic relationship between the conductivity and the molar fraction of the solvent in the C$_8$mimX - DEC hybrids, which can be divided into solvent viscosity-controlled and concentration-controlled regions. The molar faction of the solvent at the highest conductivities in the C$_8$mimBF$_4$ - DEC and C$_8$mimPF$_6$ - DEC hybrids were determined to be 0.78 and 0.86, respectively. When the traditional electrolyte, e.g. 1M LiPF$_6$ - EC/DEC, was added into the RTIL C$_8$mimPF$_6$, in the solvent viscosity-controlled region, the conductivities increase with x_s following the same trend of C$_8$mimPF$_6$ - DEC with similar conductivity values. However, in the solvent concentration-controlled region, sufficient lithium ions actively participate in the conduction leading to significantly increased conductivities, which is distinguished from the C$_8$mimPF$_6$ - DEC hybrids. The sufficient high ionic conductivities in the C$_8$mimPF$_6$ – 1M LiPF$_6$ - EC/DEC hybrids with a molar fraction xs greater than 0.8 may be feasible for lithium-ion capacitors and lithium-ion batteries and meanwhile add some merits with the presence of the long-chain RTILs.

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