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# 1-Alkyl-2,3-dimethylimidazolium bis(trifluoromethanesulfonyl)imide ionic liquids as highly safe electrolyte for Li/LiFePO<sub>4</sub> battery

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## ARTICLE INFO

Article history: Received 3 November 2009 Received in revised form 6 March 2010 Accepted 13 March 2010 Available online 23 March 2010

Keywords:

1-Alkyl-2,3-dimethylimidazolium bis(trifluoromethanesulfonyl)imide lonic liquid Lithium battery Safety LiFePO4

## ABSTRACT

Several 1-alkyl-2,3-dimethylimidazolium bis(trifluoromethanesulfonyl)imide ionic liquids (alkyl-DMimTFSI) were prepared by changing carbon chain lengths and configuration of the alkyl group, and their electrochemical properties and compatibility with Li/LiFePO<sub>4</sub> battery electrodes were investigated in detail. Experiments indicated the type of ionic liquid has a wide electrochemical window (-0.16 to 5.2 V vs. Li<sup>+</sup>/Li) and are theoretically feasible as an electrolyte for batteries with metallic lithium as anode. Addition of vinylene carbonate (VC) improves the compatibility of alkyl-DMimTFSI-based electrolytes towards lithium anode and LiFePO<sub>4</sub> cathode, and enhanced the formation of solid electrolyte interface to protect lithium anodes from corrosion. The electrochemical properties of the ionic liquids obviously depend on carbon chain length and configuration of the alkyl, including ionic conductivity, viscosity, and charge/discharge capacity etc. Among five alkyl-DMimTFSI-LiTFSI-VC electrolytes, Li/LiFePO<sub>4</sub> battery with the electrolyte-based on amyl-DMimTFSI shows best charge/discharge capacity and reversibility due to relatively high conductivity and low viscosity, its initial discharge capacity is about 152.6 mAh  $g^{-1}$ , which the value is near to theoretical specific capacity (170 mAh  $g^{-1}$ ). Although the battery with electrolyte-based isooctyl-DMimTFSI has lowest initial discharge capacity (8.1 mAh  $g^{-1}$ ) due to relatively poor conductivity and high viscosity, the value will be dramatically added to  $129.6 \text{ mAh g}^{-1}$ when 10% propylene carbonate was introduced into the ternary electrolyte as diluent. These results clearly indicates this type of ionic liquids have fine application prospect for lithium batteries as highly safety electrolytes in the future.

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# 1. Introduction

The realization of large-scale electrochemical devices is strongly expected all over the world, owing to the demand for energy storage devices, for example, load-leveling systems. Although many batteries such as sodium–sulfur battery and redox-flow battery have been in practical use as megawattclass, large-scale batteries up to now, there has been much concern regarding lithium secondary batteries for the purpose of achieving high-energy-density and maintenance-free battery systems [1]. For lithium secondary batteries, metallic lithium is an ideal battery material for the concern of energy density since its theoretical capacity is as high as 3860 mA/g. However, one limitation for its practical use is the growth of dendrites during cycling, which causes non-reversibility and even safety hazard [2]. Another safety concern is the high flammability and high vapor pressure of conventional organic solvents such

\* Corresponding author at: School of Chemical and Material Engineering, Jiangnan University, Wuxi 214122, China. Tel.: +86 5105811863; fax: +86 5105811863. *E-mail address:* zaijunli@263.net (L. Zaijun). as ethylene carbonate and ethyl methyl carbonate commonly used in lithium ion batteries nowadays.

Ionic liquids are molten at room temperature, namely coulombic liquids. Ionic liquids have many important specific properties, including reasonably high conductivity, negligible vapor pressure, high thermal stability and non-flammability. All these properties make ionic liquids are very interesting new materials for electrochemical uses. Among all possible applications, those directed to lithium ion batteries are highly appealing. Ionic liquids as electrolyte for lithium batteries have been widely examined in the recent years, which based on different cation of the ionic liquids such as imidazolium [3-7], phosphonium [8,9], quaternary ammonium [10], sulfonium [11] and pyrrolidinium [12-15]. The disadvantages of ionic liquids, higher viscosity, lower conductivity and poor compatibility with electrodes than conventional organic solvents, lead to poor electrochemical performance of the batteries with ionic liquids electrolytes. To resolve the problem, some organic additives such as ethylene carbonate (EC), vinylene carbonate (VC), and special anions of ionic liquids such as bis(fluorosulfonyl)imide (FSI), etc [16], were used to stabilize and protect interface between electrodes and ionic liquid phase against

<sup>0013-4686/\$ -</sup> see front matter © 2010 Elsevier Ltd. All rights reserved. doi:10.1016/j.electacta.2010.03.043

an undesirable irreversible reaction of ionic liquid components. As the ionic liquids-based imidazolium cation are relatively inexpensive and have been well exploited, these have been reported as ionic liquid electrolytes for lithium batteries, however, the problem of a stability limit up to the redox potential of lithium metal has been pointed out, and there have been very few reports on rechargeable batteries that use lithium metal as an anode. In recent years, some research groups tested 2-substituted imidazolium ionic liquid as electrolyte for lithium secondary batteries [17–19]. The results revealed introducing an electron-donating methyl into the second position of the imidazolium cation will promote charge delocalization in the imidazolium cation and improve the reduction stability of imidazolium-based ionic liquids. However, studies devoted on influence of the ionic liquid's structure on the electrochemical performance are scarce.

In this study, several 1-alkyl-2,3-dimethylimidazolium bis(trifluoromethanesulfonyl)imide ionic liquids which abbreviated as alkyl-DMimTFSI (see Fig. 1), were designed and examined as highly safe electrolyte for Li/LiFePO<sub>4</sub> battery. We mainly focused on the study on the influence of the ionic liquid's structure on their physicochemical properties, interfacial properties and battery performance.

## 2. Experimental

#### 2.1. Preparation of ionic liquid electrolytes

Butyl-DMimTFSI was prepared through an usual route [20,21]. First, 1.0 mol of freshly distilled 1-bromobutane (Aldrich, 98%) and 1.0 mol of 1,2-dimethylimidazole (Aldrich, 98%) were transferred into a 500 mL of the round bottom flask fitted with a reflux condenser and nitrogen protecting facilities. 50 mL of toluene was added to the flask. The mixture was stirred at 70 °C for 18 h, to form a transparent viscous liquid product, 1-butyl-2, 3dimethylimidazolium bromide. The liquid product was cooled and transferred to a 2000 mL beaker, and 1.0 mol of LiTFSI dissolved in 500 mL of water was added under stirring, and the mixture was continuously stirred for 4 h at room temperature. The resulting organic phase was extracted with dichloromethane, washed with water, and then purified with activated carbon and acid alumina. The final product butyl-DMimTFSI was a clear colorless at room temperature. The ionic liquid was dried in vacuum at 80 °C for 12 h and at 120 °C for 24 h to remove residual solvent and water. Other ionic liquids studied were prepared using a similar procedure, with different bromoalkane replaced by 1-bromobutane, respectively.

The <sup>1</sup>H NMR data of five ionic liquids were listed in the following (UNITYINOVA 400, 300 MHz, DCCl<sub>3</sub>): butyl-DMimTFSI: 0.960 (t, 3H), 1.380 (m, 2H), 1.767 (m, 2H), 2.594 (s, 3H), 3.790 (s, 3H), 4.040 (t, 2H), 7.172 (d, 1H), 7.204 (d, 1H); amyl-DMimTFSI: 0.900 (t,3H), 1.326 (m, 4H), 1.788 (m, 2H), 2.588 (s, 3H), 3.786 (s, 3H), 4.029 (t, 2H), 7.174 (d,1H), 7.206 (d,1H); octyl-DMimTFSI: 0.876 (t, 3H), 1.277 (m,10H), 1.788 (m, 2H), 2.600 (s,3H), 3.801 (s, 3H), 4.032 (t, 2H), 7.163 (d,1H), 7.210 (d,1H); isooctyl-DMimTFSI: 0.872 (m, 6H), 1.277 (m, 8H), 1.701 (m, 1H), 2.567 (s, 3H), 3.787 (s, 3H), 3.914 (d, 2H), 7.109 (d, 1H), 7.216 (d, 1H); and decyl-DMimTFSI: 0.866 (t, 3H), 1.285 (m, 14H), 1.794 (m, 2H), 2.587 (s, 3H), 3.788 (s, 3H), 4.020 (t, 2H), 7.155 (d, 1H), 7.198 (d, 1H).

The binary electrolyte (0.2 mol/L solution of LiTFSI in the ionic liquid) was prepared by dissolving dried LiTFSI in the ionic liquid in an argon-filled glove box and stirring the solution at room temperature for 48 h. The ternary electrolyte was obtained through the addition of 5 wt% VC to the above binary electrolyte.

## 2.2. Test of stability of electrodes in the ionic liquid electrolytes

Evaluation of the electrochemical stability windows of the ionic liquids, respectively, were carried out by linear-sweep voltammetry (LSV, scan rate of  $10 \text{ mV s}^{-1}$ ) in a two-electrode cell with glass carbon electrode working electrode (surface area  $0.9 \text{ cm}^2$ ) and lithium foil as both counter and reference electrode. In LSV experiment, the starting potential is -1 V, and potential scanning direction is from -1 V to 6 V. The glass carbon was polished before each potential scanning.

The Li/electrolyte interfacial resistance was measured in Li/electrolyte/Li symmetric cells through the ac impedance on Zahner–Elektrik IM6e electrochemical workstation with the frequency ranging from 200 kHz to 10 MHz and an ac signal of 5 mV in amplitude as the perturbation at room temperature.

The characteristics of LiFePO<sub>4</sub>/electrolyte interface were investigated by electrochemical impedance spectroscopy (EIS) and the cyclic voltammetry (CV) in a LiFeO<sub>4</sub>/electrolyte/Li cell with LiFePO<sub>4</sub>



decyl-DMimTFSI

Fig. 1. Chemical structures of the ionic liquids.

as working electrode and lithium foil as both counter and reference electrode. The LiFePO<sub>4</sub>/electrolyte interfacial resistance was measured in through the ac impedance on Zahner–Elektrik IM6e electrochemical workstation with the frequency ranging from 200 kHz to 10 MHz and an AC signal of 5 mV in amplitude as the perturbation at room temperature. The cyclic voltammetry of the LiFePO<sub>4</sub> electrode in 0.2 M LiTFSI-ionic liquid-VC solution was carried out in LiFePO<sub>4</sub>/electrolyte/Li cells (0.02 mV s<sup>-1</sup>) at room temperature using Zahner–Elektrik IM6e electrochemical workstation.

#### 2.3. Cell assemblies and electrochemical measurements

Li/electrolyte/LiFePO<sub>4</sub> cells were used for electrochemical measurement. LiFePO<sub>4</sub> was purchased from Hunan Shanshan Advanccd Masterial Co., Ltd. The LiFePO<sub>4</sub> surface was particularly modified and its D<sub>50</sub> and BET specific surface area are 5–6  $\mu$ m and 9–11 m<sup>2</sup>/g. LiFePO<sub>4</sub> cathode electrode was made up of LiFePO<sub>4</sub>, acetylene black, and polytetrafluoroethylene binder with a weight ratio of 85:10:5. The metallic lithium anode, Celgard 7320 separator, the ionic liquid electrolyte, and LiFePO<sub>4</sub> cathode, which the thickness of LiFePO<sub>4</sub> cathode is about to 100  $\mu$ m, were assembled in sequence into cells in an argon-filled dry glove box. The galvanostatic charge/discharge tests were performed at a C/20 rate and potential of 2.2–3.8 (vs. Li<sup>+</sup>/Li) at room temperature under the land CT2001 battery tester.

## 3. Results and discussion

#### 3.1. Physicochemical and electrochemical properties

To understand electrochemical behavior of the ionic liquid electrolytes, some important physicochemical and electrochemical properties of neat ionic liquids and corresponding binary electrolytes were well investigated. Fig. 2A represents typical DSC curves of neat ionic liquids. Fig. 2A indicates clearly there are no obvious endothermic peak and exothermic peak in the temperature range between -10°C and 350°C on the DSC curves of these ionic liquids, which real the type of ionic liquids has excellent heating stability. Fig. 2B exhibits the conductivity of neat ionic liquid (red column) and binary electrolyte (yellow column). From Fig. 2B we can clearly see the conductivity of the ionic liquid is obviously higher than that of its binary electrolyte. This is the reason that addition of LiTFSI increases viscosity of the ionic liquid and results in decreasing the conductivity. Moreover, Fig. 2B also shows ion conductivity of the ionic liquid seriously depend on the molecular structure, the conductivity will decrease with increasing carbon number of alkyl group in the ionic liquid. We think this is mainly due to rapidly increasing the ionic liquid viscosity with increasing number of the carbon number in alkyl group (see Fig. 2C). Accordingly, the isooctyl-DMimTFSI and decyl-DMimTFSI are difficult to be directly applied to lithium batteries due to poor conductivity. Fig. 2D displays LSV to clarify the anodic and cathodic electrochemical stability of neat ionic liquids and binary electrolytes at room temperature. Electrochemical windows of the neat ionic liquids and binary electrolytes (insert in Fig. 2D) were -0.4 to 5.2 V and -0.16to 5.2 V, respectively. As the anodic limiting current flow observed at 5.2 V (vs. Li<sup>+</sup>/Li) corresponds to the oxidation of the TFSI<sup>-</sup> anions, while the cathodic limiting current at -0.1 V (vs. Li<sup>+</sup>/Li) indicated the decomposition of the alkyl-DMim<sup>+</sup> cations. Additionally, CV exhibits a pair of redox peaks around 0 V (vs. Li<sup>+</sup>/Li) in the presence of LiTFSI (no show), which corresponds to the deposition and dissolution of lithium on and from the copper substrate. Thus, five new alkyl-DMimTFSI ionic liquids are of excellent electrochemical sta-



Fig. 2. DSC thermograms (A), conductivity at 25 °C (B), viscosity-temperature curves (C) and electrochemical window (D) for different alkyl-MimTFSI ionic liquid, a, b, c, d and e curves represent butyl-DMimTFSI, anyl-DMimTFSI, octyl-DMimTFSI and isooctyl-DMimTFSI at 25 °C, respectively.



Fig. 3. Nyquist plots of Li symmetric cell without VC (A and C) and with VC (B and D) at open circuit condition at room temperature after the cell was deposited for 1 day (A and B) and 5 days (C and D), a, b, c, d and e represent amyl-DMimTFSI, octyl-DMimTFSI, isooctyl-DMimTFSI, and decyl-DMimTFSI, respectively.

bility under charge/discharge conditions. These results confirmed introducing an electron-donating methyl at the second position of the imidazolium cation will promote charge delocalization in the imidazolium-based ionic liquids and remarkably enhance the stability of the ionic liquid.

## 3.2. Compatibility of the ionic liquid electrolytes with electrodes

#### 3.2.1. Metallic lithium electrode

The availability of dimensionally stable and flexible electrolyte membranes is very desirable for the progress of lithium batteries. To examine compatibility with metallic lithium anode and LiFePO<sub>4</sub> cathode, EIS and CV were used to study on the interfacial properties of the binary and ternary electrolytes. Fig. 3 exhibits Nyquist plots of Li symmetric cell without VC (A and C) and with VC (B and D) at open circuit condition at room temperature after the cell was deposited for 1 day (A and B) and 5 days (C and D). From Fig. 3A and B we clearly see the binary electrolyte offers a fairly high and steep rise of interfacial impedance, and the interfacial impedance of the ternary electrolytes increased slowly and steadily. Since the interfacial impedance composes of charge transfer resistance and SEI film resistance, above results further confirmed the addition of VC possibly well enhance the formation of SEI and protect lithium electrode from corrosion. Thus, the ternary electrolyte has excellent compatibility with metallic lithium anode at room temperature under open circuit conditions.

#### 3.2.2. LiFePO<sub>4</sub> electrode

The investigation of the electrode behavior in the ternary electrolyte was extended to the LiFePO<sub>4</sub> case (see Fig. 4). Fig. 4A presents typical Nyquist plots of the EIS measurements for Li/LiFePO<sub>4</sub> cells incorporating two kinds of electrolytes during different cycles when charged to 3.8 V. The electrolyte exhibits a semicircle in the high-frequency range and an inclined line in the low-frequency range after the 1st (blue curve in Fig. 4A) and 25th cycle (red curve in Fig. 4A). The high-frequency semicircle represents the resistance of the SEI film, and the inclined line corresponds to the lithium ion diffusion process, Warburg diffusion. Li/LiFePO4 cell show the size of semicircle at high-frequency is very similar after and before 1 cycle and slightly increased after 25 cycles, However, the change value in SEI film resistance with VC is remarkably smaller than that of the cell without VC, this results also demonstrate VC addition can form an improved SEI film. In the test intercalation/deintercalation of lithium ions into LiFePO<sub>4</sub> electrode, the cyclic voltammetry performance of the LiFePO<sub>4</sub> electrode in the ternary electrolytes was measured in using a LiFePO<sub>4</sub>/electrolyte/Li cells at 25 °C and the results was shown in Fig. 4B. We see from Fig. 4B the response evidences in the 1st cycle the occurrence of some irreversibility, however, the following cycles proceed with well reproduced peaks without evidence of side reactions. These results demonstrated better compatibility towards LiFePO<sub>4</sub> cathode.

## 3.3. Battery performance

The charge/discharge performance Li/LiFePO<sub>4</sub> cells incorporating different kind of ionic liquid electrolytes were well examined and their initial discharge capacities were shown in Fig. 5. It was found charge/discharge performance obviously depended on the kind of ionic liquid. Due to high ion conductivity and relatively low viscosity, Li/LiFePO<sub>4</sub> cells which incorporating butyl-DMimTFSI and amyl-DMimTFSI ionic liquids have better charge/discharge capacities than that of cells with other three ionic liquid electrolytes. Among five new ionic liquid electrolytes, the amyl-DMimTFSI has best electrochemical properties, its initial discharge capacity is about 152.6 mAh g<sup>-1</sup>. The value is near to theoretical specific capacity (170 mAh g<sup>-1</sup>) and much higher than that of PP<sub>14</sub>TFSI ionic liquid electrolyte [13]. Although the cell with isooctyl-DMimTFSI



Fig. 4. Nyquist plots of Li/electrolytes/LiFePO<sub>4</sub> cell after special charge/discharge was carried out for 1 (A) and 25 cycles (B). (C) Cyclic voltammetry of Li/LiTFSI + amyl-DMimTFSI + VC/LiFePO<sub>4</sub> cell.

ionic liquid electrolyte exhibited smallest initial discharge capacity (8.1 mAh g<sup>-1</sup>), it well be dramatically added to 129.6 mAh g<sup>-1</sup> when 10% propylene carbonate was introduced into the ternary electrolyte as diluent, which demonstrate the viscosity of the ionic liquids is main factor to effect on the cell performance for this type of ionic liquids.

Typical galvanostatic charge/discharge and cyclic performance for Li/LiFePO<sub>4</sub> cell were shown in Figs. 6 and 7 with amyl-DMimTFSI ionic liquid electrolyte at 0.05 C rate, respectively. Fig. 6 indicates the cell with the electrolyte offer one charge and discharge plateau around 3.4 V, i.e. within the stability domain of the electrolyte solution. Fig. 7 exhibited the discharge capacities of the cell become 148 mAh g<sup>-1</sup> after 120 cycles, which is about to 97% of the initial capacity. The reversibility is obviously better than that of classical 1-alkyl-3-dimethylimidazolium type ionic liquid electrolytes. Because substituting the chemically stable methyl group reduces the high reactivity and controls the side reaction with the metallic lithium electrode, a high reversibility was achieved in the battery systems that used the alkyl-DMimTFSI ionic liquids. Moreover, the capacity approaches the theoretical value and thus, we may conclude that also the LiFePO<sub>4</sub> electrode behaves in the ionic liquid-based solution as well as in the common organic electrolytes.



**Fig. 5.** Initial discharge capacity of the cell with different ionic liquid ternary electrolyte, in which a, b, c, d and e column represent the cell with amyl-DMimTFSI, butyl-DMimTFSI, octyl-DMimTFSI, decyl-DMimTFSI and isooctyl-DMimTFSI ternary electrolyte, respectively. Ternary electrolytes were prepared using the method described in Section 2.1.



**Fig. 6.** Galvanostatic charge/discharge for Li/LiFePO<sub>4</sub> cell with the amyl-DMimTFSI ternary electrolyte for 1st cycle (1) and 120th cycle (2). The amyl-DMimTFSI ternary electrolyte was prepared using the method described in Section 2.1.



**Fig. 7.** Cycle-life performance for Li/electrolytes/LiFePO<sub>4</sub> cell with amyl-DMimTFSI ternary electrolyte (square) and binary electrolyte (triangle) at room temperature. The amyl-DMimTFSI ternary electrolyte and binary electrolyte were prepared using the method described in Section 2.1.

#### 3.4. Conclusions

In the paper, five new 1-alkyl-2,3-dimethylimidazolium bis(trifluoromethanesulfonyl)imide ionic liquids were designed and investigated as highly safe electrolyte for Li/LiFePO<sub>4</sub> battery. The results show the type of ionic liquid electrolytes have very wider electrochemical window (-0.16 to 5.2 V vs. Li<sup>+</sup>/Li) and are theoretically feasible as an electrolyte for batteries with metallic Li as anode. Substituting the chemically stable methyl group reduces the high reactivity and controls the side reaction with the metallic lithium anode and LiFePO<sub>4</sub> cathode under charge/discharge conditions. This results in improvement of the electrochemical stability and compatibility with electrodes. The electrochemical properties seriously depend on the kind of the ionic liquid, including ionic conductivity, viscosity, charge/discharge capacity and reversibility. Two noel ionic liquids, butyl-DMimTFSI and amyl-DMimTFSI, exhibit better electrochemical performance and can be devel-

oped for Li/LiFePO<sub>4</sub> batteries as highly safe electrolytes. Although another three ionic liquids are not suit as electrolytes for Li/LiFePO<sub>4</sub> battery as electrolyte due to relatively low conductivity and big viscosity, these can be used as additive of electrolytes for various batteries to improve conductivity and safety

## Acknowledgements

The authors acknowledge the financial support from the National Natural Science Foundation of China (Nos. 20771045 and 20676052), the Natural Science Foundation of Zhejiang Province (No. Y4080404) and Qing Lan Project.

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