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Improving electrochemical properties of room temperature ionic liquid (RTIL) based electrolyte for Li-ion batteries

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A B S T R A C T

Room temperature ionic liquids (RTILs) with high safety characteristic usually have high viscosity and melting point, which is adverse for the application of RTIL-based electrolytes in Li-ion batteries. In this investigation, a promising RTIL, i.e. PP13TFSI consisting of N-methyl-N-propylpiperidinium (PP13) cation and bis(trifluoromethanesulfonyl)imide (TFSI) anion is synthesized. The effect of the content of Li salt in the electrolytes containing PP13TFSI and LiTFSI on the ionic conductivity and cell performance is investigated. The electrolyte of 0.3 mol kg^{-1} LiTFSI/PP13TFSI is recommended for its higher lithium transference number and discharge capacity in the $LiCoO_2$/Li cell than other electrolytes. In addition, it is found that, by introducing 20% diethyl carbonate (DEC) as a co-solvent into pure RTIL electrolyte, the rate capability and low-temperature performance of the $LiCoO_2$/Li cells are improved obviously, without sacrificing its safety characteristics. It suggests that a component with low viscosity and melting point, i.e. DEC, is necessary to effectively overcome the shortcomings of RTIL for the application in Li-ion batteries.

1. Introduction

With fossil energy decreased and exhausted, renewable energy (i.e. wind and solar energy) is expected to play a leading role in the future. Meanwhile, the energy storage and converse devices are necessary for storing the electric power because of the unstable output from the renewable energy like wind power or sunshine. Li-ion batteries have many advantages, such as higher energy density, longer cycling lifetime and lower self-discharge rate than other rechargeable batteries, so that they can play an important role on the electric power storage [1]. However, there are some issues restricting the development of the large-scale Li-ion batteries such as batteries for electric vehicles (EV) and hybrid electric vehicles (HEV), even though the small-scale Li-ion batteries have been successfully put into practice for portable electronic products. One of the biggest barriers is the safety concern, which is mainly owing to volatile and flammable organic solvents used in the electrolyte. Recently, room temperature ionic liquids (RTILs) have been extensively studied as new solvents in electrochemical batteries and supercapacitors due to their extremely low vapor pressure and good flame resistance [2–14]. The unique properties make RTILs attractive candidates for the electrolytes of Li-ion batteries with excellent safety characteristics.

Sakaebe proposed a novel RTIL, N-methyl-N-propylpiperidinium bis(trifluoromethanesulfonyl)imide (PP13TFSI) as the solvent of electrolyte, which exhibited the electrochemical stability up to lithium reduction potential (<0 V versus Li/Li$^+$) and excellent properties in Li/$LiCoO_2$ cells [2,5–9]. After that, a lot of RTILs with similar structure with PP13TFSI were investigated as the solvent for Li-ion batteries [10,12,13]. In addition to the excellent safety characteristics, they also showed good compatibility with lithium electrode and cycling performance in the batteries. Although PP13TFSI as well as its analogue has been investigated widely, there are still some puzzles for the application in the Li-ion batteries. For example, they usually had relatively high viscosity and melting point, which caused poor rate capability and low-temperature performance [5,10,14]. Also, the ionic conductivity of the binary LiTFSI-RTIL electrolyte systems is much more sensitive to the salt content, since the viscosity of RTILs is much higher than the commonly used carbonate-based electrolytes. However, the salt content in the binary LiTFSI-PP13TFSI electrolyte systems is usually fixed at 0.4 mol/kg or 10 wt.%, and few papers report the influence of the salt content on the physicochemical and cell performances of the electrolytes [8,10].

In this paper, we first investigate the influence of the content of Li salt in the binary LiTFSI-PP13TFSI electrolyte on ionic conductivity and cell performance, and then diethyl carbonate (DEC) as a co-solvent with low viscosity and melting point is introduced into the viscous RTIL electrolyte to improve the rate capability and low-temperature performance of the RTIL-based electrolyte

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in the $LiCoO_2$/Li cells. This approach provides an effective route to overcome the shortcomings of pure RTIL-based electrolyte for the application in Li-ion batteries.

2. Experimental

2.1. Synthesis of LiTFSI-RTIL electrolytes

N-methyl-N-propylpiperidinium bis(trifluoromethanesulfonyl) imide (PP13TFSI) was prepared by stirring two aqueous solutions of PP13-Br and LiTFSI (Ferro, battery grade) at room temperature for 12 h. The amount of LiTFSI was slightly in excess versus that of PP13-Br. PP13-Br was obtained by adding propylbromide (Sinopharm Chemical Reagent Co., Ltd, as received) to N-methylpiperidine (Aldrich, as received) in acetonitrile at 70 °C with stirring for 24 h. The resulting RTIL PP13TFSI was extracted by CH_2Cl_2 and then washed with water. Finally, the RTIL was dried in vacuum at 80 °C for 48 h, followed by a storage in an argon-filled glove box (Mbraun Labmaster 130). The chemical structure and the purity of PP13TFSI were confirmed by ^{1}H NMR (Bruker, 300 M) and thermogravimetric (TG) measurement (Shimadzu).

The binary LiTFSI-RTIL electrolytes were prepared with different concentrations, i.e. 0.1, 0.2, 0.3 and 0.4 mol kg^{-1} in the glove box. Another two electrolytes containing diethyl carbonate (DEC) were prepared by adding LiTFSI into the mixture of RTIL and DEC; and the two electrolytes have 20 wt.% and 40 wt.% DEC (compared to the weight of RTIL) with the same amount of the salt corresponding to 0.4 mol kg^{-1}.

2.2. Thermal, electrical and electrochemical characterization of LiTFSI-RTIL electrolytes

The melting point of RTIL was measured by differential scanning calorimetry (DSC, Shimadzu) in the temperature range from −60 °C to 150 °C at the heating rate of 10 °C min^{-1}. The ionic conductivities of RTIL and the electrolyte solutions were measured over a wide temperature range of −30 to 90 °C using a Model DDS-307A conductometer (Shanghai Precision & Scientific Instrument Co. Ltd., China). Each conductivity measurement at a given temperature was carried out after holding the electrolyte solution at the temperature for 30 min in order to reach full thermal equilibration. The temperature of the electrolyte solution and subsequent cell testing at low temperatures was controlled by a WD4005 low-temperature chamber (Shanghai Experimental Equipment Co. Ltd., China). The lithium transference number was determined cursorily by a dc polarization method with a CHI604 Electrochemical Workstation [15]. By applying a small dc pulse (0.08 V here) to a symmetrical Li/electrolyte/Li cell and measuring the initial current, I_0, and the steady-state current, I_{ss}, which flow through the cell, the lithium ion transference number, t_{Li^+}, is simply given by the equation: $t_{Li^+} = I_{ss}/I_0$ [16]. The self-extinguishing time (SET) was measured to evaluate the flammability of the electrolytes, and the detailed procedures were described elsewhere [17,18].

CR2032 coin cells were used for measuring the electrochemical properties and cell performances. A positive electrode consisting of 84 wt.% $LiCoO_2$, 8 wt.% acetylene black and 8 wt.% PVDF were made on aluminum foils, with the mass loading of 1.5 mg cm^{-2} active material. All cells were assembled in the glove box and then galvanostatically cycled on a multi-channel battery cycler (Neware BTS2300, Shenzhen). For all the $LiCoO_2$/Li cells, the cutoff voltages were set at 2.8 and 4.2 V. Cyclic voltammetry (CV) and linear sweep voltammetry (LSV) (CHI604 Electrochemical Workstation) were used to study the electrochemical stability of the RTIL-based electrolyte in the stainless steel (SS)/Li cells at a scan rate of 0.2 mV s^{-1}. For the CV test, the voltage range was set at 3.0–0 V, and for the LSV, the voltage was limited to 6.0 V.

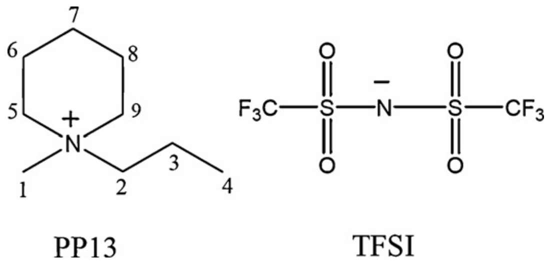

Fig. 1. Schematic illustration of the molecular structure of cation and anion from the room temperature ionic liquid (PP13TFSI).

The thermal stability of different electrolytes with charged $LiCoO_2$ electrode was evaluated by using a Calvet-type calorimeter (Setaram C80). $LiCoO_2$/Li cells with different electrolytes were charged to 4.2 V after three formation cycles, followed by being disassembled in the glove box. The charged electrodes were rinsed in dimethyl carbonate (DMC) and then dried in vacuum. Finally, a mixture of 20 mg electrolyte supplied and 20 mg charged electrode materials scraped from the aluminum foil was placed in a high-pressure stainless steel vessel with dry argon atmosphere. The measurement was performed in the temperature range from ambient temperature to 300 °C at a 0.2 °C min^{-1} heating rate.

3. Results and discussion

3.1. Structure and thermal behavior of PP13TFSI

The structure of PP13TFSI (Fig. 1) was confirmed by ^{1}H NMR spectra ($CDCl_3$, chemical shift, ppm relative to TMS): 3.4 (3H, 1), 1.1 (3H, 4), 3.8 (2H, 2), 3.6–3.7(4H, 5,9), 1.6–1.9(8H, 3,6,7,8). Fig. 2 shows the TG/DTA curves of the RTIL synthesized, which confirms the expected high thermal stability. From the TG curve, the RTIL only exhibits a decomposition process starting at 342 °C and ending at 484 °C, with ~100% weight loss. The DTA curve also indicates only one endothermic decomposition process with a peak at 453 °C, and the heat generation is −153.5 J g^{-1}. The TG/DTA results also validate that the RTIL that we have synthesized has the high purity.

3.2. Electrochemical stability of PP13TSFI

The electrochemical stability of PP13TSFI is investigated by the voltammetry, including CV and LSV shown in Figs. 3 and 4. As shown in Fig. 3, the negligible electrochemical behavior with the peak cur-

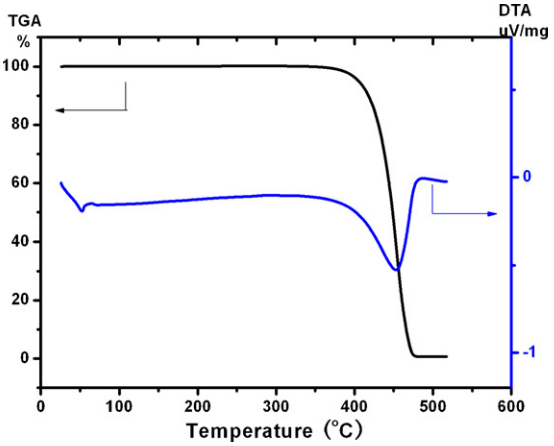

Fig. 2. TG/DTA curves of PP13TFSI synthesized. The heating rate is 10 °C min^{-1}.

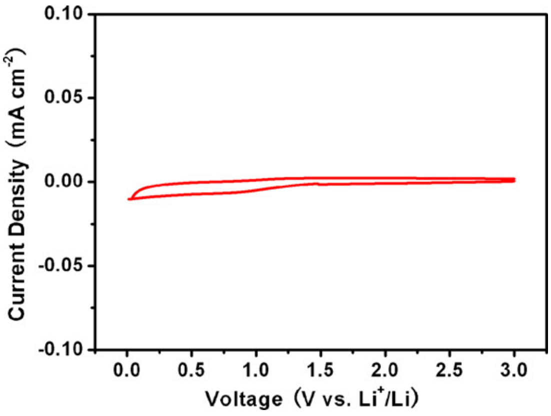

Fig. 3. Cathodic cyclic voltammetry of 0.4 mol kg^{-1} LiTFSI/PP13TFSI electrolyte in the stainless steel (SS)/Li cell. The scan rate is 0.2 mV s^{-1}.

rent density lower than 0.01 mA cm^{-2} indicates that PP13TFSI has good cathodic stability in the range of 3–0 V, which is highly advantageous compared with the imidazolium-based ionic liquids, such as 1-ethyl-3-methyl imidazolium (EMI)-TFSI, with high cathodic potential of 1.1 V versus Li$^+$/Li [2]. Thus, PP13TFSI can be used in the battery with metallic lithium anode, where EMI-based RTIL cannot be used. Also, the obviously anodic decomposition potential of PP13TFSI is about 5.5 V (in Fig. 4), which suggests that PP13TFSI has a wide electrochemical window for use in the Li-ion batteries.

3.3. Electrical properties of LiTFSI-PP13TFSI electrolytes

It was reported that PP13TFSI had a high viscosity of 151 mPa s at 25 °C, and after addition of lithium salt, i.e. LiTFSI, the electrolyte become more viscous [14]. High viscosity of RTIL-based electrolytes could have an adverse effect on the ionic mobility and thus the ionic conductivity of the electrolytes. Hence, here we investigate the effect of concentration of lithium salt on the ionic conductivity and lithium ion mobility in the binary LiTFSI-PP13TFSI electrolyte. Fig. 5 shows the temperature dependence of the ionic conductivity of electrolytes with different concentrations of the lithium salt. It can be easily found that, with increasing the concentration of the lithium salt, the ionic conductivity gradually decreases, which is mainly because a significant increase of viscosity after introducing lithium salt depresses the motion and transfer of free ions in the electrolyte systems. In addition, the conductivity variation with temperature of all samples follows the Vogel–Tamman–Fulcher (VTF) behavior as shown in the inserted graph of Fig. 5. The VTF equation is as follows:

$$\sigma = \sigma_0 \exp\left[\frac{-E_a/R}{T - T_0}\right]$$

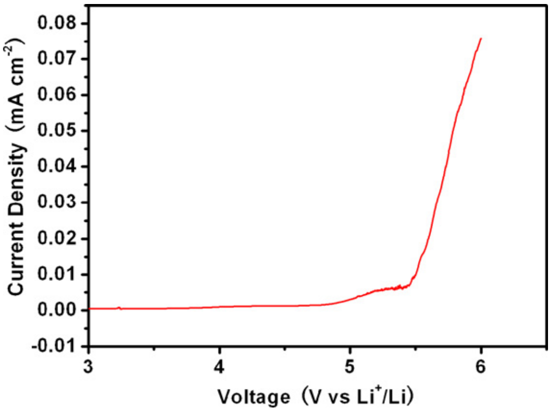

Fig. 4. Anodic linear sweep voltammetry of 0.4 mol kg^{-1} LiTFSI/PP13TFSI electrolyte in the SS/Li cell. The scan rate is 0.2 mV s^{-1}.

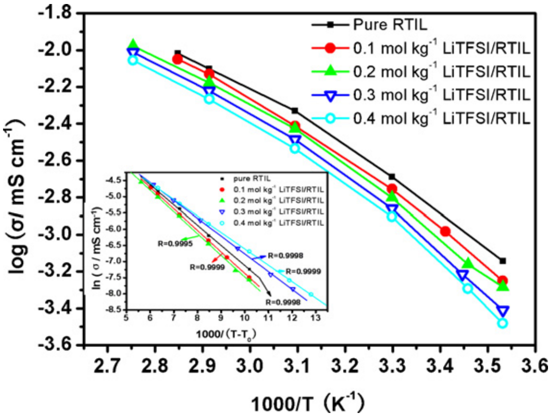

Fig. 5. Temperature dependence of ionic conductivity of electrolytes with different concentration of lithium salt. The inserted graph shows conductivity variation with temperature following VTF equation, and R is correlation coefficient for linear fitting.

In the VTF equation, E_a is the energy of activation, σ_0 is the pre-exponential factor, T_0 is the theoretical glass transition temperature and T is the absolute temperature. For each sample, the T_0 is determined by fitting the temperature-dependent conductivity data to the VTF equation for the best linearity relationship. E_a is determined from the linear slope. The deduced values of T_0 and E_a are listed in Table 1. Though the pure RTIL has the highest ionic conductivity, it cannot be used solely as the electrolyte in the Li-ion batteries and the addition of LiTFSI is necessary for Li$^+$ conduction that has more direct and important effect on the cell performance. Thus, the lithium transference number was cursorily determined by a dc polarization method at room temperature. From Table 1, it can be found that T_0, which indirectly reflects on the viscosity of the electrolyte systems, increases with the concentration of lithium salt. With LiTFSI added into the RTIL, the ionic concentration increases. However, owing to the interaction between the anion (TFSI$^-$) and cations (Li$^+$ and PP13$^+$), the higher viscosity makes the ionic motion and transfer more difficult, which results in the ionic conductivity of the electrolytes reduced rather than raised. E_a is a parameter reflecting on the temperature dependence of ionic conductivity of electrolytes. With the concentration of LiTFSI increasing, E_a firstly ascends for the increased ionic concentration, and then descends because the increasing viscosity becomes the more important factor. The lithium transference number, t_{Li^+}, has the similar variation as E_a. When the lithium salt is added into the RTIL, the free Li$^+$ appears and becomes more and more with the concentration increasing, so t_{Li^+} increases. But when the amount of Li$^+$ exceeds a certain value, the excessive Li$^+$ along with the anion constitutes ion pairs, which results in the increase of viscosity of electrolyte systems. So, t_{Li^+} descends after the concentration

Table 1
Data from the VTF equation of temperature dependence of ionic conductivity of electrolytes with different concentration of lithium salt, and results of lithium transference number.

	Concentration (mol kg^{-1})				
	Pure	0.1	0.2	0.3	0.4
T_0 (K)	185	185	185	200	205
E_a (kJ mol^{-1})	5.18	5.53	5.60	4.59	4.22
t_{Li^+}	N/A	0.35	0.41	0.46	0.31

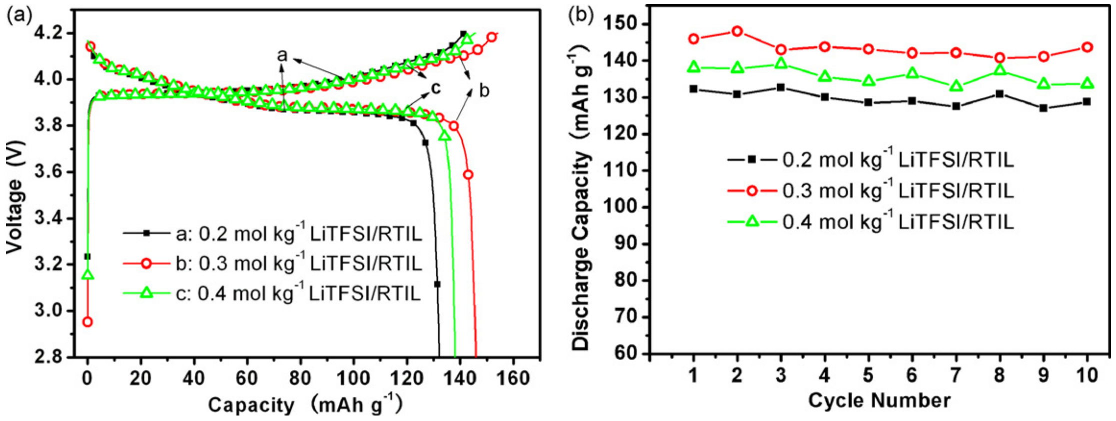

Fig. 6. First voltage profiles (a) and cycling performance (b) of the $LiCoO_2$/Li cells with different electrolytes: (a) 0.2 mol kg^{-1} LiTFSI/RTIL, (b) 0.3 mol kg^{-1} LiTFSI/RTIL, (c) 0.4 mol kg^{-1} LiTFSI/RTIL. The current rate is set at 1/10 C.

Table 2
Data from the VTF equation of temperature dependence of ionic conductivity of electrolytes with different amounts of DEC.

	LiTFSI/RTIL	LiTFSI/RTIL + 20% DEC	LiTFSI/RTIL + 40% DEC
T_0 (K)	205	160	160
E_a (kJ mol^{-1})	4.22	5.25	4.40

of lithium salt exceeds a certain extend. Here the electrolyte with 0.3 mol kg^{-1} LiTFSI has the highest Li^+ transference number.

3.4. Electrochemical performance of $LiCoO_2$/Li cells with LiTFSI-PP13TFSI electrolytes

Fig. 6 shows the electrochemical performance of $LiCoO_2$/Li cells with three electrolytes containing different concentrations of LiTFSI. The current density is 0.02 mA cm^{-2}, which is equivalent to 1/10 C. It can be seen that all the cells have a high coulombic efficiency of about 95% (Fig. 6a) and good cycling performance (Fig. 6b). Obviously, the cell with the electrolyte containing 0.3 mol kg^{-1} LiTFSI has the highest discharge capacity of 146 mAh g^{-1}, which is coincident with the highest lithium transference number. Although the electrolyte of 0.3 mol kg^{-1} LiTFSI/RTIL has lower ionic conductivity than the electrolyte of 0.2 mol kg^{-1} LiTFSI/RTIL, and lower concentration of LiTFSI than the electrolyte of 0.4 mol kg^{-1} LiTFSI/RTIL, it has the best overall performance and thus is recommended to replace 0.4 mol kg^{-1} or 10 wt.% usually used in previous papers.

3.5. Effects of DEC addition on the LiTFSI-PP13TFSI electrolytes

PP13TFSI has a good electrochemical stability, but its high viscosity (151 mPa s) and high melting point (8.7 °C, reported by Sakaebe [5]) brings poor rate capability and low-temperature performance in the Li-ion batteries. Thus, a co-solvent with low viscosity and melting point, DEC, is introduced into the RTIL to improve the rate capability and low-temperature performance of the relative cells. Fig. 7 shows the temperature dependence of ionic conductivity of electrolytes containing 0%, 20% and 40% DEC. Obviously, the ionic conductivity is substantially improved after the introduction of DEC. Also, conductivity variation with temperature of all samples follows the VTF behavior in the inserted graph of Fig. 7. And the corresponding E_a and T_0 based on the VTF equation are all listed in Table 2. After introduction of DEC, T_0 is sharply reduced, which suggests that the viscosity of the electrolyte system should obviously be decreased. The decreased viscosity brings the greater E_a due to the introduction of 20% DEC, but after adding more DEC, the ionic density, especially contributed from the RTIL, is decreased so that E_a is reduced to a certain extent.

High viscosity of RTIL usually brings poor rate capability of the Li-ion batteries. Some groups replaced $TFSI^-$ with bis(fluorosulfonyl)imide anion (FSI^-) and obtained the RTILs with low viscosity, but the improvement of rate capability was still limited [14,19]. For example, at 2 C discharge rate, the $LiCoO_2$/Li cell with PP13FSI had only 40% capacity retention of the discharge capacity at 0.1 C [13]. So, we think it is difficult to solve the problem of poor rate capability only by designing the structure of RTILs owing to their intrinsical flaw. The experience of using mixed solvents in the state-of-the-art electrolyte can give a solution. Nakahara prepared a nonflammable $LiPF_6$ based electrolyte with 50% PP13TFSI and 50% (EC-DMC-EMC) carbonate mixture, which exhibited higher reversible capacity at room temperature, significantly worse capacity retention at lower temperature and better rate performance than the electrolyte without RTIL [20]. Herein, we manage to improve the rate capability and low-temperature performance by incorporating DEC with low viscosity and melting point into PP13TFSI. Fig. 8 shows the rate capability of the $LiCoO_2$/Li cells with different electrolytes. When the discharge rate is below C/2, there is slight difference between discharge capacities of the cells containing three electrolytes. However, a significant capacity loss is found at C/2, for the electrolyte of 0.4 mol kg^{-1} LiTFSI/RTIL. And the discharge capacity is about 30 mAh g^{-1} at 1 C, which is only 23% of the discharge capacity at 0.1 C. However, after 20% DEC is added, excellent rate capability is obtained, that is 89% at 1 C and 78% at 2 C

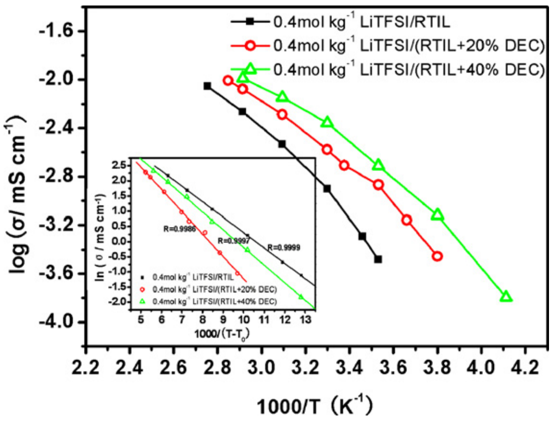

Fig. 7. Temperature dependence of ionic conductivity of electrolytes with different amounts of DEC. The inserted graph shows conductivity variation with temperature following VTF equation, and *R* is correlation coefficient for linear fitting.

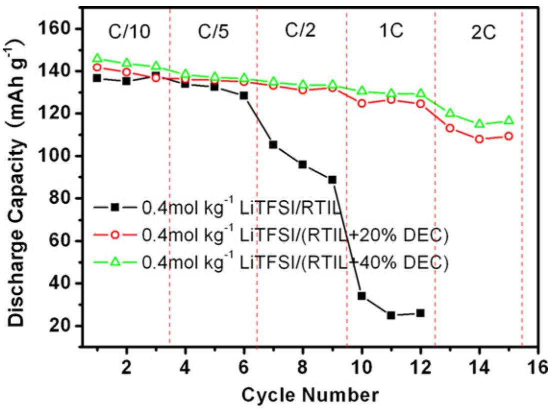

Fig. 8. Rate capability of the $LiCoO_2$/Li cells with different electrolytes: 0.4 mol kg^{-1} LiTFSI/RTIL (close square), 0.4 mol kg^{-1} LiTFSI/(RTIL + 20%DEC) (open circle) and 0.4 mol kg^{-1} LiTFSI/(RTIL + 40%DEC) (open triangle). All the cells are charged to 4.2 V at 1/10 C and then discharged to 2.8 V at different rates.

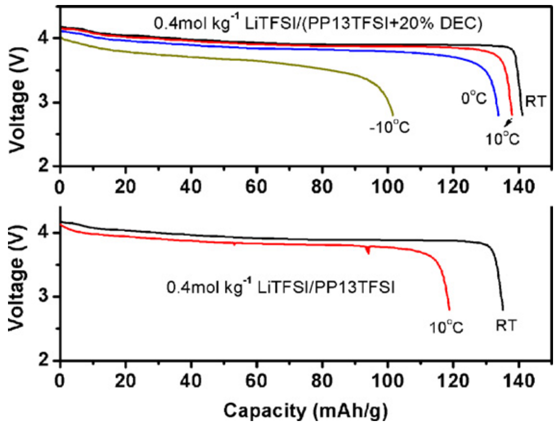

Fig. 10. Discharge capacities of $LiCoO_2$/Li cells with the electrolytes under different temperatures. All the cells are charged to 4.2 V at room temperature and then discharged to 2.8 V under different temperatures. The current rate is 1/10 C.

compared with the discharge capacity at 0.1 C, respectively. Therefore, we can conclude that a small amount of DEC (i.e. 20%) can efficiently improve the rate capability of the RTIL electrolyte in the Li-ion batteries. Definitely, it is beneficial to introduce more DEC (i.e. 40%) for further improving the rate capability, but the gain is quite narrow.

Fig. 9 shows the DSC spectra of pure RTIL and the mixtures of RTIL and DEC. An endothermic peak at 10.5 °C is corresponding to the melting point of PP13TFSI. After 20% DEC is added into the RTIL, the endothermic peak moves to −18.7 °C, and an exothermic peak at −35 °C is attributed to a process of crystallization. When 40% DEC is added, the endothermic peak corresponding to the melting point disappears above −50 °C, but at the range of 100–150 °C, an endothermic process related to the evaporation of DEC appears distinctly. The results of these DSC spectra indicate that introducing DEC can reduce the melting point of the system containing RTIL, but too much DEC brings a high vapor pressure which destroys the safety characteristic. Fig. 10 shows the comparison results between low-temperature performances of the cells with the electrolyte of 0.4 mol kg^{-1} LiTFSI/RTIL and the electrolyte containing 20% DEC. For the cell with the electrolyte of 0.4 mol kg^{-1} LiTFSI/RTIL, the discharge capacity is 119 mAh g^{-1} at 10 °C, which is equal to about 87% of the discharge capacity at room temperature, and at a temperature lower than 10 °C, the electrolyte is frozen and the cell cannot be discharged smoothly. As for the electrolyte containing 20% DEC, the capacity utilization accessed at 10 °C and even 0 °C keeps a high level of above 130 mAh g^{-1}, and also the discharge capacity at −10 °C is 102 mAh g^{-1}, which is about 72% of the capacity at room temperature. Therefore, the low-temperature performance of the cell with the PP13TFSI-based electrolyte can be significantly improved by the introduction of 20% DEC.

RTIL is a promising candidate for the electrolyte of Li-ion batteries, because its advantages of nonflammability and low vapor pressure are favorable for the safety characteristics of Li-ion batteries. It is definitely a compromise that the way by adding a flammable and volatile organic solvent, i.e. DEC, into PP13TFSI is used to improve the rate capability and low-temperature performance of the RTIL-based electrolyte. In our flammability test, all the three electrolytes with 0, 20% and 40% DEC are validated to be nonflammable, because they cannot be ignited, meaning their SET is all measured to be 0.0 s. Fig. 11 shows the results of C80 calorimetry for the charged $LiCoO_2$ cathode materials and electrolytes. All the three samples have a wide exothermic band at 200–275 °C, with two peaks at 225 °C and 250 °C. There is negligible difference between the three electrolytes on the exothermic peak at 225 °C, but obvious differences between the peaks at 250 °C can be found. Compared with the sample without DEC, only a tiny increase in heat generation (denoted by the area of the exothermic peak) is found for the sample containing 20% DEC, but the heat flow and heat generation of the sample containing 40% DEC are increased sharply. In addition, from the consideration of vapor pressure, the electrolyte containing 40% DEC is also unfavorable. Therefore, the electrolyte containing 20% DEC is recommended for good safety characteristics.

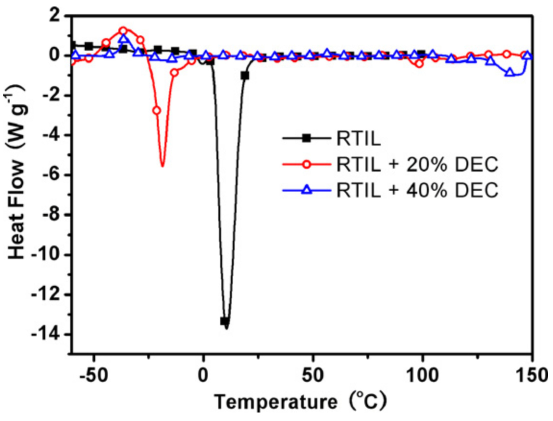

Fig. 9. DSC spectra of pure RTIL and the mixtures of RTIL and DEC. The scanning rate is 10 °C min^{-1}.

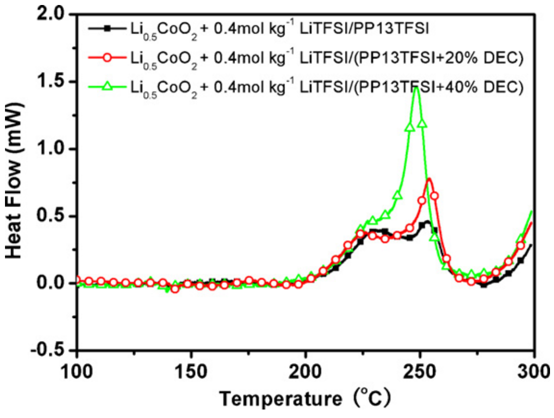

Fig. 11. C80 heat-flow profiles of the charged $Li_{0.5}CoO_2$ with three kinds of electrolytes. The heating rate is set at 0.2 °C min^{-1}.

4. Conclusions

The RTIL-based electrolyte is an appealing candidate to replace the flammable and volatile carbonate-based electrolyte in the Li-ion batteries, owing to its excellent physicochemical properties, i.e. nonflammability and negligible vapor pressure. As one of the most promising RTILs, PP13TFSI is synthesized in this study. The effects of the content of Li salt in the electrolytes containing PP13TFSI and LiTFSI on the ionic conductivity and cell performance are investigated. The electrolyte of 0.3 mol kg^{-1} LiTFSI/PP13TFSI is recommended for its higher lithium transference number and discharge capacity in the $LiCoO_2$/Li cell than other electrolytes. In addition, it is found that 20% DEC as a co-solvent introduced into the RTIL-based electrolyte can improve the rate capability and low-temperature performance, with a negligible damage to the safety characteristics of the Li-ion batteries. It provides an effective approach to overcome the shortcoming of RTIL for the application in Li-ion batteries.

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