



# Cycling performance and thermal stability of lithium polymer cells assembled with ionic liquid-containing gel polymer electrolytes

Ye Sun Yun<sup>a</sup>, Jin Hee Kim<sup>a</sup>, Sang-Young Lee<sup>b</sup>, Eun-Gi Shim<sup>c</sup>, Dong-Won Kim<sup>a,\*</sup>

<sup>a</sup> Department of Chemical Engineering, Hanyang University, Seungdong-Gu, Seoul 133-791, Republic of Korea

<sup>b</sup> Department of Chemical Engineering, Kangwon National University, Chuncheon, Kangwondo 200-701, Republic of Korea

<sup>c</sup> Electrolyte Development Team, Techno SemiChem Co. Ltd., Yongin-si, Gyeonggi-do 446-599, Republic of Korea

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## ABSTRACT

Gel polymer electrolytes containing 1-butyl-1-methylpyrrolidinium bis(trifluoromethanesulfonyl)imide and a small amount of additive (vinylene carbonate, fluoroethylene carbonate, and ethylene carbonate) are prepared, and their electrochemical properties are investigated. The cathodic limit of the gel polymer electrolytes can be extended to 0V vs. Li by the formation of a protective solid electrolyte interphase on the electrode surface. Using these gel polymer electrolytes, lithium metal polymer cells composed of a lithium anode and a  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  cathode are assembled, and their cycling performances are evaluated at room temperature. The cells show good cycling performance, comparable to that of a cell assembled with gel polymer electrolyte containing standard liquid electrolyte (1.0M LiPF<sub>6</sub> in ethylene carbonate/diethylene carbonate). Flammability tests and differential scanning calorimetry studies show that the presence of the ionic liquid in the gel polymer electrolyte considerably improves the safety and thermal stability of the cells.

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## 1. Introduction

Rechargeable lithium polymer batteries using lithium metal as the anode are one of the most attractive candidates for high energy density power sources in portable electronic devices and electric vehicles [1], because lithium metal offers the largest specific capacity as a negative electrode. In this cell, the use of a gel polymer electrolyte enables fabrication of thin batteries with design flexibility [2]. However, safety issues surrounding these batteries must be addressed before they can be widely utilized since highly flammable organic solvents in the gel polymer electrolytes can cause fire or explosions under short circuit or abusive conditions. In the quest for a non-flammable electrolyte system, ionic liquids (ILs) have been extensively studied and are recognized as one of the safest electrolytes for use in lithium batteries [3–7]. ILs have attractive properties, such as a negligible vapor pressure, low flammability, high ionic conductivity and high thermal stability. Among the various ILs, those based on pyrrolidinium cations and bis(trifluoromethanesulfonyl)imide anions have been considered to be promising for battery applications, due to their reasonably wide electrochemical stability and high ionic conduc-

tivity [8–13]. Unfortunately, however, they easily decompose on the negative electrode at a positive potential relative to that of Li/Li<sup>+</sup>, which results in the formation of an unstable solid electrolyte interphase (SEI) on the electrode. One of the efficient ways to form an electrochemically stable SEI is to use reducible compounds, which tend to decompose on the anode surface before the reduction of ionic liquid during charging [14–17]. To provide a protective SEI that overcomes the reductive decomposition of the ionic liquid, we added different kinds of organic solvent to the IL-based gel polymer electrolytes. Various inorganic materials, such as LiCoO<sub>2</sub>, LiNiO<sub>2</sub>, LiMn<sub>2</sub>O<sub>4</sub>, LiNi<sub>x</sub>Co<sub>y</sub>Mn<sub>1–y</sub>O<sub>2</sub> and LiFePO<sub>4</sub> have been developed and applied as the cathode material in lithium batteries. Among these materials, LiNi<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> is of particular interest as an active cathode material because of its high capacity, good structural stability and relatively low cost [18–21]. With the goal of developing high energy density lithium batteries with improved safety, we assembled lithium metal polymer cells composed of a lithium anode, an IL-based gel polymer electrolyte and a LiNi<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> cathode, and evaluated their cycling performance. In the present work, 1-butyl-1-methylpyrrolidinium bis(trifluoromethanesulfonyl)imide (BMP-TFSI) was chosen as an IL component, and vinylene carbonate (VC), fluoroethylene carbonate (FEC) and ethylene carbonate (EC) were selected as the SEI-forming additives. The flammability of the electrolyte solution and the thermal stability of the delithiated cathode were also investigated.

\* Corresponding author. Tel.: +82 2 2220 2337; fax: +82 2 2298 4101.  
E-mail address: [dongwonkim@hanyang.ac.kr](mailto:dongwonkim@hanyang.ac.kr) (D.-W. Kim).

## 2. Experimental

### 2.1. Preparation of gel polymer electrolytes and electrodes

BMP-TFSI was purchased from Chem Tech Research Incorporation and was used after drying under a vacuum at 100 °C for 24 h. The water content in BMP-TFSI after drying was determined to be 8 ppm by Karl Fisher titration, with an ionic conductivity of  $2.4 \text{ mS cm}^{-1}$ . IL electrolyte was prepared by dissolving 1.0 M lithium bis(trifluoromethanesulfonyl)imide (LiTFSI) in BMP-TFSI. Anhydrous carbonate solvent (VC, FEC, EC) was added to the IL electrolyte at a concentration of 10 wt%. The standard liquid electrolyte used for comparative purposes was 1.0 M  $\text{LiPF}_6$  in ethylene carbonate (EC)/diethylene carbonate (DEC) (3:7, v/v, Techno Semichem Co., Ltd., battery grade). A porous polymer membrane based on poly(vinylidene fluoride-co-hexafluoropropylene) (P(VdF-co-HFP)) was prepared according to the procedure previously reported [22]. A gel polymer electrolyte was then prepared by immersing the P(VdF-co-HFP) membrane in IL-based electrolyte or liquid electrolyte.

### 2.2. Electrode preparation and cell assembly

$\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  powders were synthesized by the coprecipitation method, as described previously in detail [20]. The cathode was prepared by coating the N-methyl pyrrolidone (NMP)-based slurry containing  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ , poly(vinylidene fluoride) (PVdF) and super-P carbon (85:7.5:7.5 by weight) on an aluminum foil. Electrode thicknesses ranged from 50 to 60  $\mu\text{m}$  after roll pressing, and active mass loading corresponded to the capacity of about  $1.6 \text{ mAh cm}^{-2}$ . The anode consisted of a 100  $\mu\text{m}$ -thick lithium foil that was pressed onto a copper current collector. The lithium metal polymer cell was assembled by sandwiching the gel polymer electrolyte between the lithium anode and the  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  cathode. Subsequently, the cell was enclosed in a metalized plastic bag and was vacuum-sealed. All cell-assembly was performed in a dry box filled with argon gas.

### 2.3. Measurements

Linear sweep voltammetry was performed to investigate the electrochemical stability of the IL-based gel polymer electrolyte on a stainless steel working electrode, with counter and reference electrodes of lithium metal at a scanning rate of  $1.0 \text{ mV s}^{-1}$ . The self-extinguishing time (SET) was measured to quantify the flammability of the mixed electrolyte, as described in previous literature [23]. Briefly, SET was obtained by igniting the pre-weighed electrolytes soaked in an inert glass-fiber wick ( $3 \times 3 \text{ cm}^2$ ), followed by recording the time it took for the flame to extinguish. AC impedance measurements were performed using an impedance analyzer over the frequency range of 1 mHz to 100 kHz with an amplitude of 10 mV. The coulombic efficiency of lithium in the IL-based gel polymer electrolyte was measured by constant-current lithium deposition and dissolution test with a 2032-type coin cell, as given in previous literature [24]. Lithium was deposited on the stainless steel electrode under 0.01 mA for 200 min and then stripped under the same current until the potential of stainless steel electrode reached 2.0 V vs. Li. Charge and discharge cycling tests of the cells were conducted at a constant current density of  $0.16 \text{ mA cm}^{-2}$  (0.1C rate) over a voltage range of 3.0–4.3 V with battery testing equipment at room temperature. For differential scanning calorimetry (DSC) experiments, the cells were fully charged to 4.3 V after 100 cycles and were disassembled in the dry box. Approximately 5 mg of the cathode scraped from the current collector was hermetically sealed in a stainless steel pan, and measurements were taken at a heating rate of  $1 \text{ }^\circ\text{C min}^{-1}$ . Before DSC

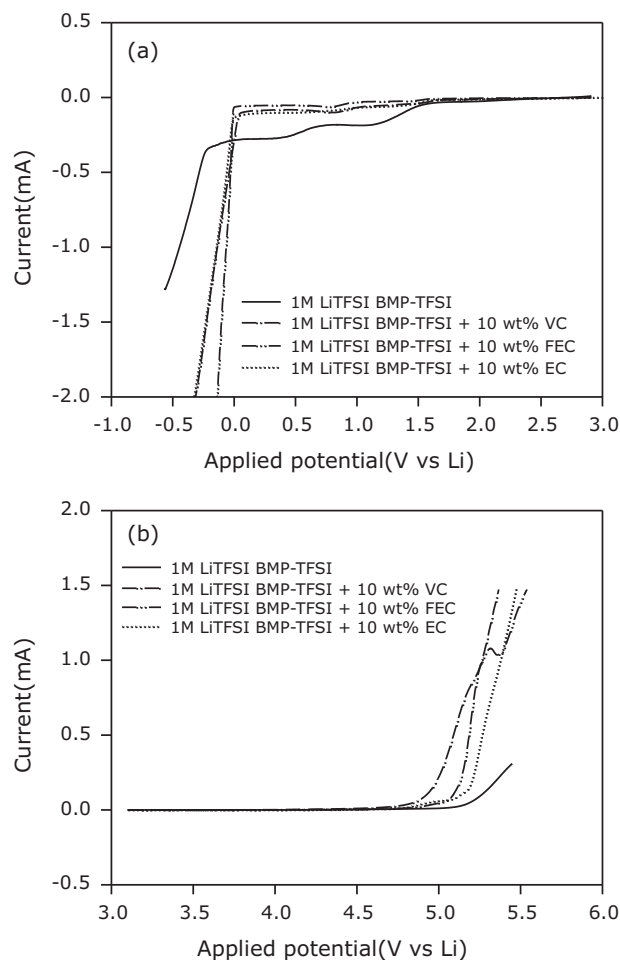
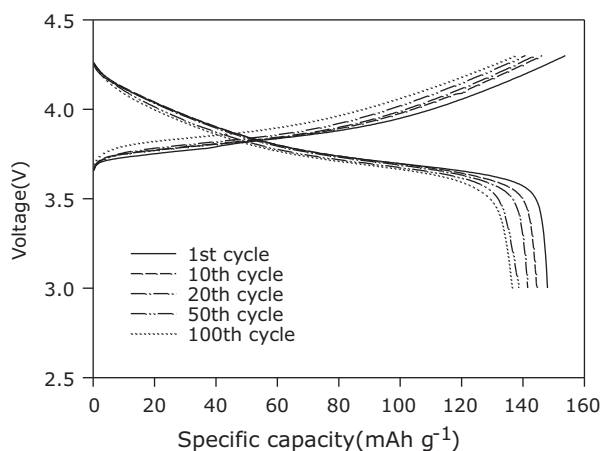


Fig. 1. Linear sweep voltammograms of the IL-based gel polymer electrolytes with different additives: (a) cathodic scan and (b) anodic scan.

measurements, the amount of the cathode active material and the electrolyte solution was measured, and heat flow was calculated based on the weight of the cathode active material.

## 3. Results and discussion

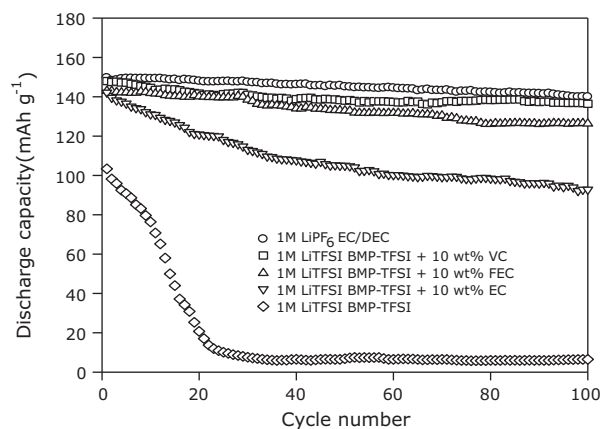
Linear sweep voltammetry curves of the IL-based gel polymer electrolytes prepared with and without additives are shown in Fig. 1(a) and (b), which are obtained from cathodic and anodic scans, respectively. It is observed in Fig. 1(a) that the cathodic current starts to increase around 1.6 V vs.  $\text{Li/Li}^+$  in the case of the IL-based gel polymer electrolyte without additive. Because the lithium deposition should have occurred below 0 V, the cathodic current may correspond to the reductive decomposition of BMP-TFSI. For the IL-based gel polymer electrolytes containing an additive, small and broad reduction peaks are observed in the potential range between 0.5 and 1.8 V. These can be attributed to the reductive decomposition of VC, FEC and EC, which result in the formation of a stable SEI film. This SEI prevents the reductive decomposition of BMP-TFSI and kinetically extends the cathodic stability to 0 V vs.  $\text{Li/Li}^+$ . The current associated with the lithium deposition is, in fact, significantly enlarged, suggesting that the addition of the VC, FEC and EC may indeed allow for ongoing electrochemical processes down to 0 V vs.  $\text{Li/Li}^+$ . This result is consistent with previous works which showed that carbonate-based solvents could effectively prevent the decomposition of ionic liquid [14–17]. In the anodic scan shown in Fig. 1(b), the oxidative current corresponding to the anodic decomposition starts at around 5.2 V for the IL-based gel polymer



**Fig. 2.** Charge and discharge curves of the Li/LiNi<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> cell assembled with gel polymer electrolyte containing 1 M LiTFSI BMP-TFSI and 10 wt.% VC (room temperature, 0.1C rate, cut-off voltage range: 3.0–4.3 V).

electrolyte without additive. On the other hand, the introduction of additive decreases the anodic stability of gel polymer electrolytes, which may originate from the anodic decomposition of the additive. Nevertheless, they show a broad electrochemical stability window higher than 4.8 V, which is necessary for high voltage cathode applications. From these results, it is expected that IL-based gel polymer electrolytes containing a small amount of VC, FEC and EC have an electrochemical stability suitable for allowing the electrochemical operation of Li/LiNi<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> cells considered in the present study.

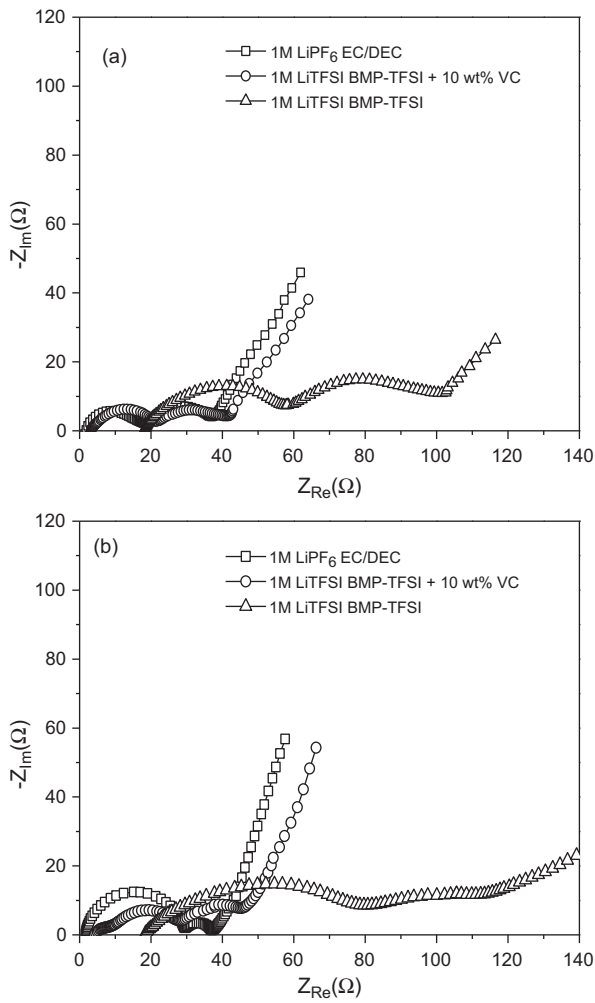
Cycling performances of lithium metal polymer cells prepared with IL-based gel polymer electrolytes were evaluated. Fig. 2 shows the charge–discharge curves of the cell assembled with IL-based gel polymer electrolyte containing VC, which were obtained at a constant current density of 0.16 mA cm<sup>-2</sup> (0.1C rate). The cycling behavior of the cell was comparable to that of the cell assembled with liquid electrolyte-based gel polymer electrolyte with respect to the charge–discharge profiles and the reversible capacity. The cell exhibits a first discharge capacity of 148.0 mAh g<sup>-1</sup>, based on the LiNi<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> active cathode material, and the discharge capacity of the cell declined to 136.4 mAh g<sup>-1</sup> after 100 cycles. Columbic efficiency steadily increased and then stabilized with cycle number, and it was higher than 99% after several initial cycles.



**Fig. 3.** Discharge capacities as a function of cycle number for the Li/LiNi<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> cells assembled with gel polymer electrolyte containing different electrolyte solutions (room temperature, 0.1C rate, cut-off voltage range: 3.0–4.3 V).

Fig. 3 shows the discharge capacities of the cells prepared with gel polymer electrolyte containing different electrolyte solutions, as a function of cycle number. For the purpose of comparison, the cycling result of the cell assembled with gel polymer electrolyte containing liquid electrolyte is also given in the figure. The initial discharge capacity is lowest in the cell prepared with IL-based gel polymer electrolyte without additive. This can be explained by considering that the use of highly viscous BMP-TFSI in preparing gel polymer electrolyte causes an increase in both electrolyte resistance and charge transfer resistance at the electrode/electrolyte interface. The lower capacity may be also due to the decomposition of the ionic liquid electrolyte on the electrode surface, which prevents reversible electrochemical reactions. On the other hand, the addition of VC, FEC and EC increases the initial discharge capacity up to the level of the cell prepared with liquid electrolyte-based gel polymer electrolyte. This result is due to both the enhancement of ionic conductivity and the formation of a stable SEI film on the electrode surface. It was confirmed that the ionic conductivities of IL-based gel polymer electrolytes increased with the addition of VC, FEC and EC. Fig. 3 also reveals that the capacity retention of the cells is dependent on the type of electrolyte solution in the gel polymer electrolyte. The cell prepared with IL-based gel polymer electrolyte without additive shows significant capacity fading, which is related to undesirable irreversible decomposition reactions of the ionic liquid electrolyte during cycling. On the contrary, the addition of carbonate-based solvents leads to better discharge capacity retention. This result is due to the fact that the additives form an electrochemically stable SEI film on the electrode surface during repeated cycling, preventing the reductive decomposition of the ionic liquid electrolyte, as depicted in Fig. 1(a). Among various additives, VC led to the best results with good capacity retention, that is, 92.2% of the initial discharge capacity after 100 cycles, which is close to that of the cell prepared with gel polymer electrolyte containing standard liquid electrolyte. On the other hand, the cell containing EC as an additive exhibited poor capacity retention as compared to those of cells containing VC or FEC.

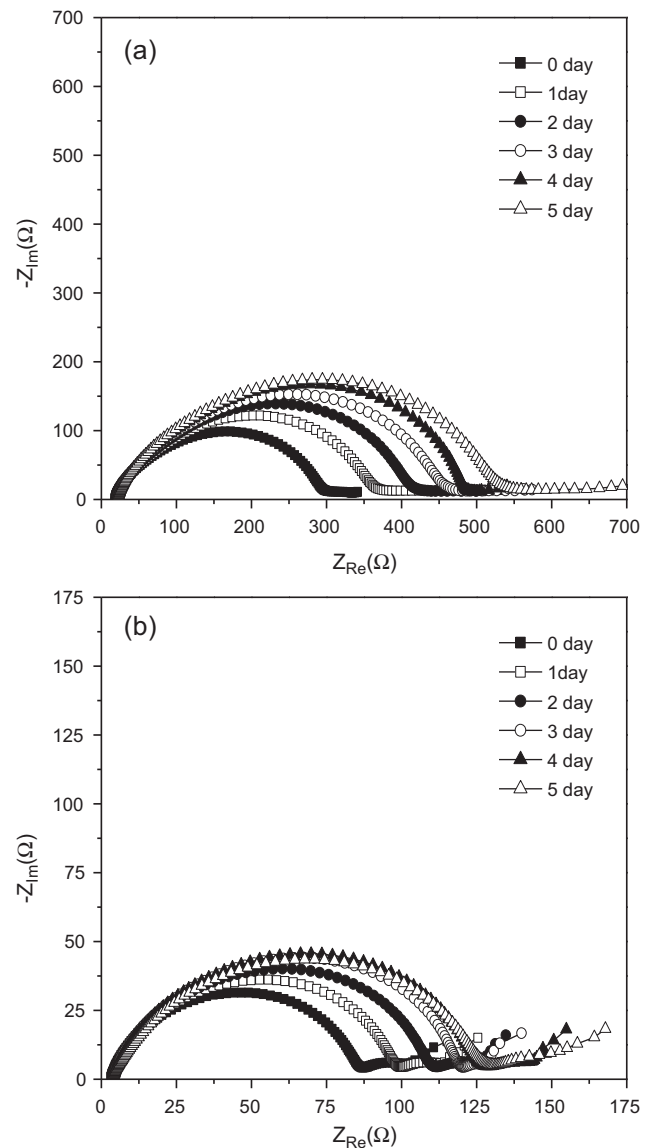
In order to investigate the impedance behavior of cells prepared with gel polymer electrolyte containing different electrolyte solutions, the ac impedance of the cells was measured and the results are shown in Fig. 4. It has been known that ac impedance behavior of the cell depends on the state of charge [25]. For a fair comparison, we tried to investigate the ac impedance spectra at charged state of the cell in this work. Fig. 4(a) was obtained at the charged state after 1 cycle, and Fig. 4(b) was measured at the charged state after 100 cycles. In both spectra, two overlapping semicircles are observed. According to previous impedance analysis studies [26,27], the semicircle in the high frequency range can be attributed to resistance due to Li<sup>+</sup> ion migration through SEI film on the electrode ( $R_f$ ), while the semicircle in the medium-to-low frequency range is due to the charge transfer resistance between the electrode and electrolyte ( $R_{ct}$ ). It is clearly seen that both the electrolyte resistance and the interfacial resistance decrease by the addition of a small amount of VC into the IL-based gel polymer electrolyte. After 1 cycle, the cell assembled with the gel polymer electrolyte containing liquid electrolyte has the lowest electrolyte resistance and the lowest interfacial resistance, as expected. The highest  $R_f$  is observed in the cell assembled with IL-based gel polymer electrolyte without additive, while the cell assembled with IL-based gel polymer electrolyte containing VC has small value of  $R_f$ . As mentioned previously, the addition of VC leads to the formation of a stable SEI that protects against a reductive decomposition of the ionic liquid on the electrode, which permits the reversible migration of Li<sup>+</sup> ions through SEI. El Ouatani et al. previously reported that VC improved the properties of the SEI film at the surface of the electrodes [28]. Accordingly, the cell assembled with the IL-based gel polymer electrolyte containing VC is shown to have much lower



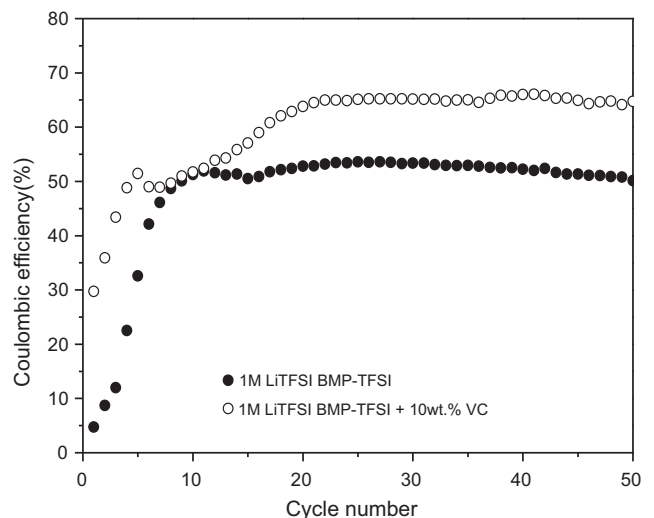
**Fig. 4.** AC impedance spectra obtained at charged state (a) after 1 cycle and (b) after 100 cycles of the Li/LiNi<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> cells assembled with gel polymer electrolytes containing different electrolyte solutions.

overall resistance than that of cell without VC, after repeated cycles. These results are consistent with the cycling behavior, as shown in Fig. 3.

We attempted to investigate the interfacial behavior of a lithium electrode in prolonged contact with IL-based gel polymer electrolyte. Fig. 5 shows the time evolution of the ac impedance spectra of a Li/gel polymer electrolyte/Li cell under open-circuit potential conditions. The spectra are in the form of distorted semicircle with the real axis intercept at high frequency, denoting the bulk electrolyte resistance ( $R_b$ ), and that at the low frequency denoting the electrode/electrolyte interfacial resistance ( $R_i$ ). In case of IL-based gel polymer electrolyte without additive, the interfacial resistance continuously increased with time, as shown in Fig. 5(a), which can be attributed to the formation of a passive layer due to the reactivity of the lithium electrode and the ionic liquid electrolyte. On the other hand, the interfacial resistance in IL-based gel polymer electrolyte containing VC increased initially and eventually stabilized at a constant value, as shown in Fig. 5(b). The final steady-state value of  $R_i$  indicated no further growth of the passive film, which suggests that this electrolyte system is highly compatible with the metallic lithium anode. The lithium deposition and dissolution efficiency in the IL-based gel polymer electrolyte without and with VC is shown in Fig. 6. The coulombic efficiency increased by adding VC into the IL-based gel polymer electrolyte and reached over about

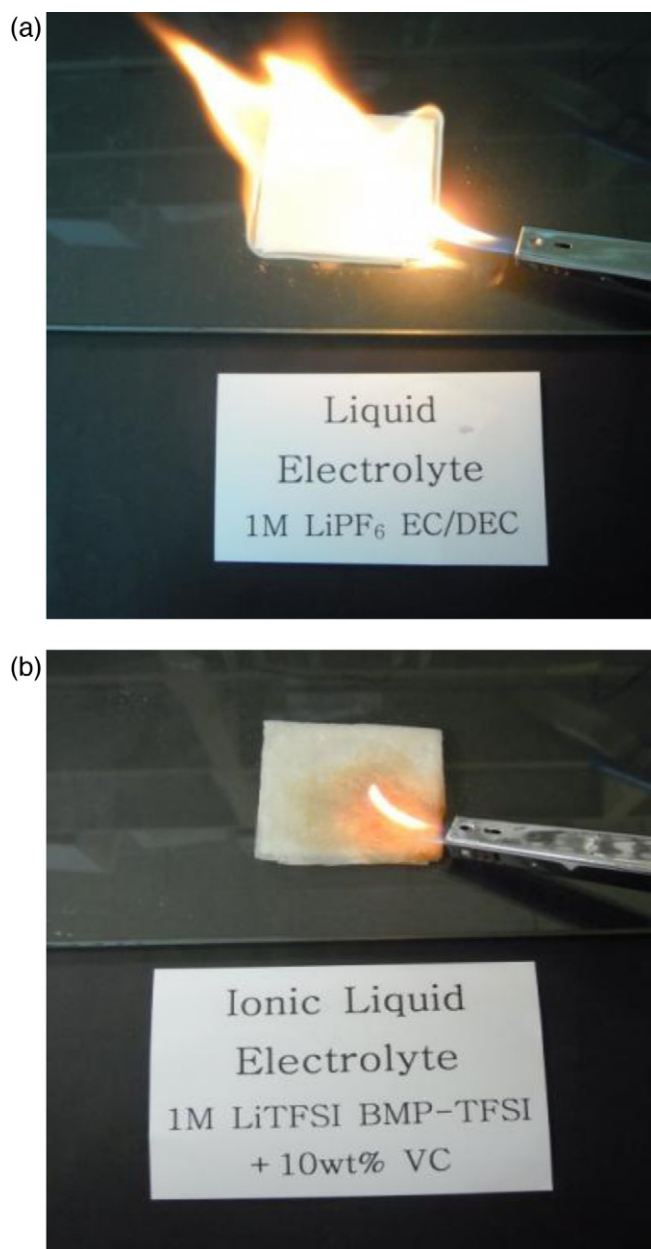


**Fig. 5.** AC impedance spectra of a Li/gel polymer electrolyte/Li cell as a function of storage time at 25 °C. (a) IL-based gel polymer electrolyte without VC and (b) IL-based gel polymer electrolyte with VC.



**Fig. 6.** Coulombic efficiency for lithium deposition and dissolution under constant current in the IL-based gel polymer electrolyte without and with VC.

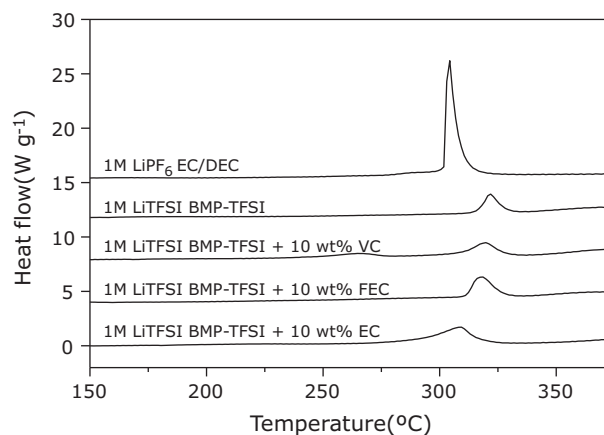




**Fig. 7.** Photographic images showing the flammable behavior of electrolyte solution during flammability test: (a) 1 M LiPF<sub>6</sub> EC/DEC and (b) 1 M LiTFSI BMP-TFSI + 10 wt.% VC.

60% after several cycles, which may be ascribed to the formation of stable surface film on electrode in the presence of VC.

Non-flammability of the IL-based electrolyte is confirmed in Fig. 7, which shows photographic images showing the flammable behavior of electrolyte solutions during flammability test. As can be seen in the figure, the liquid electrolyte is very flammable, and its SET value is measured to be about 31 g<sup>-1</sup>. By contrast, VC-containing IL electrolyte does not show any combustion, even during ignition with the flame source (i.e., SET value is 0 g<sup>-1</sup>), which indicates its non-flammable behavior. The thermal stability of the delithiated cathode materials is also of great import for battery safety. DSC measurements were thus performed to evaluate the thermal stability of the cathode material in a fully charged state. Fig. 8 shows the DSC profiles of the cathode materials charged to 4.3 V, which are obtained after 100 cycles. In this figure, the DSC trace of Li<sub>1-x</sub>Ni<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> in the cell prepared with gel



**Fig. 8.** DSC profiles of Li<sub>1-x</sub>Ni<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> cathode materials charged to 4.3 V after 100 cycles in the cells assembled with gel polymer electrolyte containing different electrolyte solutions.

polymer electrolyte containing standard liquid electrolyte has an exothermic peak with a reaction heat of 584.4 J g<sup>-1</sup> at 304.3 °C. On the other hand, the Li<sub>1-x</sub>Ni<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> material in the cell assembled with gel polymer electrolyte containing IL electrolyte has a much smaller exothermic reaction peak (192.2 J g<sup>-1</sup>) at a higher temperature (321.8 °C). These results suggest that the delithiated Li<sub>1-x</sub>Ni<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> material is less reactive towards the ionic liquid electrolyte, leading to an improvement in thermal stability. The addition of VC, FEC and EC does not significantly affect the thermal stability of the cathode material, as shown in the figure, which indicates that the thermal stability of the cathode material is maintained in the presence of an IL electrolyte with a small amount of additive. Hence, it is concluded that the use of IL-containing gel polymer electrolytes with a small amount of carbonate-based solvent hinders the exothermic reaction of the Li<sub>1-x</sub>Ni<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> electrode with an electrolyte solution that otherwise results in a large heat evolution.

#### 4. Conclusions

Gel polymer electrolytes containing BMP-TFSI and a small amount of SEI-forming additive were prepared and investigated for use in lithium metal polymer batteries. The stable SEI layer derived from VC, FEC and EC successfully protected the electrode from the reductive decomposition of the ionic liquid, which allows for their application in Li/LiNi<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>O<sub>2</sub> cells. The cells assembled with the IL-based gel polymer electrolytes containing VC exhibited reversible cycling behavior, with an initial discharge capacity of 148 mAh g<sup>-1</sup> and low capacity fading at room temperature. Flammability tests and DSC studies showed that the safety of the cells could be improved by using the IL-based gel polymer electrolytes.

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