Effect of a novel amphipathic ionic liquid on lithium deposition in gel polymer electrolytes

Nam-Soon Choi [a,*], Bonjae Koo [a], Jin-Tak Yeon [a], Kyu Tae Lee [a,*], Dong-Won Kim [b]

[a] Interdisciplinary School of Green Energy, Ulsan National Institute of Science and Technology (UNIST), 100 Banyeon-ri, Eonyang-eup, Ulju-gun, Ulsan 689-798, Republic of Korea
[b] Department of Chemical Engineering, Hanyang University, Seungdong-Gu, Seoul 133-791, Republic of Korea

ABSTRACT

A novel dimeric ionic liquid based on imidazolium cation and bis(trifluoromethanesulfonyl) imide (TFSI) anion has been synthesized through a metathesis reaction. Its chemical shift values and thermal properties are identified via ^1H nuclear magnetic resonance (NMR) imaging and differential scanning calorimetry (DSC). The effect of the synthesized dimeric ionic liquid on the interfacial resistance of gel polymer electrolytes is described. Differences in the SEM images of lithium electrodes after lithium deposition with and without the 1,1′-pentyl-bis(2,3-dimethylimidazolium) bis(trifluoromethane-sulfonyl)imide (PDMITFSI) ionic liquid in gel polymer electrolytes are clearly discernible. This occurs because the PDMITFSI ionic liquid with hydrophobic moieties and polar groups modulates lithium deposit pathways onto the lithium metal anode. Moreover, high anodic stability for a gel polymer electrolyte with the PDMITFSI ionic liquid was clearly observed.

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1. Introduction

The demand for rechargeable batteries with a higher energy density is becoming ever-more critical due to the emergence of applications such as electric vehicles and the many new types of portable electronic devices currently available. New cell chemistries are required because of the limitations associated with insertion electrodes and liquid electrolytes. The lithium-air battery potentially has a much higher energy density than lithium-ion batteries because the cathode active material is not stored in the lithium-air battery (at the beginning of the discharge process) [1,2]. Abraham et al. first reported a rechargeable lithium-air battery in 1996; it consisted of a Li⁺ conductive organic polymer electrolyte membrane, a Li metal anode, and a carbon composite electrode [3]. In this system, oxygen is reduced upon discharge to form lithium oxides (Li_2O_2 or Li_2O). The lithium oxides are then electrochemically decomposed to Li and O_2 upon charging. Although lithium-air batteries have a significantly higher energy density than conventional Li-ion battery systems, the reversibility and safety of the system present significant technical challenges for practical applications [1,4]. A lithium metal foil has a high level of chemical and electrochemical reactivity toward nonaqueous electrolytes. Accordingly, the use of a lithium metal foil as an anode can lead to

the formation of a solid electrolyte interphase (SEI) layer, which tends to form dendrites on the lithium surface [5–9]. The unstable SEI layer that forms on a lithium electrode cannot properly accommodate the dramatic morphological changes of the lithium metal during the lithium deposition and dissolution because these processes lack uniformity under a high current density. In addition, the SEI layer can be easily broken during lithium deposition and dissolution [8]. This behavior results in a considerable loss of active lithium metal and solution components due to the complex surface reactions and eventually degrades the lithium electrode. Note that the formation of an appropriate SEI layer protects lithium electrodes against further corrosion. Moreover, the non-uniform current distribution of the lithium electrode leads to the formation of dendritic lithium, which provokes an internal short circuit in lithium-air batteries [10–12]. Dendritic lithium can be cut and isolated from the anode substrate during the discharge. This isolated lithium causes a loss of anode materials and leads to a shorter life cycle. Thus, the formation of dendritic lithium and the electrochemical instability of lithium metal electrodes are significant challenges for the operation of Li-air batteries. Moreover, the control of the lithium electrode–electrolyte interface is extremely important for the successful production of Li-air batteries with a high energy density, a good cycling efficiency, and superior safety. Moisture-stable electrolytes should be considered in lithium-air batteries to prevent the lithium electrode from reacting vigorously with water traces in ambient air. Commercial Li-ion batteries commonly use the electrolyte lithium hexafluorophosphate ($LiPF_6$) in a

* Corresponding authors.
    E-mail addresses: nschoi@unist.ac.kr (N.-S. Choi), ktlee@unist.ac.kr (K.T. Lee).

mixture of organic solvents. The PF_6^- anion undergoes an equilibrium reaction $(LiPF_6(sol.) \leftrightarrow LiF(s) + PF_5(sol.))$ in solvents. The strong Lewis acid PF_5 tends to react with organic solvents, and labile P–F bonds are highly susceptible to hydrolysis even if trace amounts of moisture are present in the electrolyte solution [13,14]. In this respect, we can say that $LiPF_6$-based electrolytes are not appropriate for lithium-air batteries. Nevertheless, the organic electrolyte Li-air cell architecture with $LiPF_6$ conducting salt is still the most frequently investigated cell architecture [4,15,16]. In 2005, Kuboki et al. reported on the use of an ionic liquid (1-ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl)imide) in an Li-air battery: they pointed out that hydrophobic ionic liquid electrolytes were promising electrolytes for lithium-air batteries because they could prevent hydrolysis of the lithium metal electrode due to the stability this method provides when there is moisture in the air [17]. Besides their chemical and electrochemical stability, some ionic liquids also act as a moisture barrier due to their hydrophobic character. Moreover, compared with organic solvents, ionic liquids have the advantage of undergoing negligible evaporation in Li-air batteries (open systems). In a novel approach, Yu et al. used a hydrophobic ionic liquid–silica–P(VdF-co-HFP) polymer composite electrolyte in Li-air batteries; they obtained an excellent initial discharge capacity of 4080 mAh/g of carbon [18].

Moisture in air can diffuse toward a lithium metal anode through a hydrophilic electrolyte solution, causing chemical oxidation of the lithium metal. That explains why a hydrophobic polytetrafluoroethylene membrane is added to an air cathode to prevent water from invading the electrolyte through the oxygen diffusion holes.

We now report attractive methods of eliminating the formation of dendritic lithium on a lithium anode and of mitigating the undesirable reaction of a lithium electrode with moisture moved from an air cathode in lithium-air batteries. In this work, we used a novel hydrophobic ionic liquid, which was expected to hinder the penetration of moisture into a gel polymer electrolyte. The hydrophobic ionic liquid was prepared by combining an imidazolium cation with hydrophobic hydrocarbon moieties (1,1-pentyl-bis(2,3-dimethyl imidazolium) cation) and a bis(trifluoromethanesulfonyl)imide (TFSI) anion. In a previous work, a geminal imidazolium ionic liquid (1,4-bis(3-tetradodecylimidazolium-1-yl) butane bromide) with long aliphatic chains was synthesized [19]. In this study, we synthesized a novel dimeric ionic liquid with hydrophobic moiety and it was used as a battery electrolyte for the first time. We investigate the interfacial resistance levels of gel polymer electrolytes containing hydrophobic dimeric ionic liquids. We also examine how the novel ionic liquid affects the morphology of a lithium electrode after lithium deposition.

2. Experimental

2.1. Synthesis of the 1,1′-pentyl-bis(2,3-dimethylimidazolium) bis(trifluoromethanesulfonyl) imide (PDMITFSI) ionic liquid

The imidazolium-based ionic liquid was synthesized according to a standard procedure [20,21]. First we added 1,5-dibromopentane (9.20 g, 40 mmol) to 1,2-dimethyl imidazole (7.69 g, 80 mmol) under constant stirring. The mixture was allowed to react at 5 °C for 3 h while being stirred; then, as depicted in Fig. 1(a), the reaction was allowed to continue at room temperature for 5 h in a nitrogen atmosphere. The resulting white powder was filtered, washed with ethyl acetate, and dried in a vacuum at 30 °C for 10 h to produce a white solid form of 1,1′-pentyl-bis(2,3-dimethylimidazolium) bromide (PDMIBr, 15.3 g, 46.9 mmol). ^{1}H NMR (D_2O; TMS) of PDMIBr: 1.23 (q, 2H), 1.75 (q, 4H), 2.45 (s, 6H), 3.64 (s, 6H), 3.93 (t, 4H), 7.23 (d, 4H) (Fig. 2). Ionic liquids

based on TFSI are generally prepared through a metathesis reaction between the corresponding bromide salt with the desired cation and a slight excess of lithium bis(trifluoromethanesulfonyl)imide (LiTFSI) 1.05 equiv.). As shown in Fig. 1(b), the excess LiTFSI was added to a stirred solution of PDMIBr in water at room temperature. The mixture was then stirred for 5 h. The bottom layer was separated, dissolved in CH_2Cl_2, and washed several times with doubly distilled deionized water. After the CH_2Cl_2 was removed under reduced pressure, the resultant ionic liquid was dried under a vacuum at 70 °C for 24 h to produce a colorless solid form of PDMITFSI (12.2 g, yield 86%).

2.2. Characterization of the PDMITFSI ionic liquid

A Karl-Fischer titration showed that the PDMITFSI ionic liquid contained 10 ppm of water. A 0.45 g sample of the PDMITFSI ionic liquid was loaded on a sample holder and heated to 250 °C with dry nitrogen for the purpose of measuring the water content. Differential scanning data (Q200, TA Instruments) were obtained from a sealed aluminum pan with a cooling and heating rate of 10 °C/min under an N_2 purge.

2.3. Preparation of gel polymer electrolytes

A copolymer of vinylidene fluoride and hexafluoropropylene (P(VdF-co-HFP), Aldrich), which is widely used as a polymer matrix of gel polymer electrolytes, was dried in a vacuum at 80 °C for 24 h. The solvent tetrahydrofuran (THF) was distilled with a sodium–benzophenone complex prior to use. A liquid electrolyte was prepared by dissolving 1 M LiTFSI (PANAX E-Tec Co. Ltd.) in a mixture of ethylene carbonate (EC) and propylene carbonate (PC) (50/50, v/v). The EC/PC solvent and the PDMITFSI ionic liquid were mixed at various volume ratios, and a 1 M concentration of LiTFSI was dissolved in the resulting solution. Gel polymer electrolytes were fabricated by solution-casting a mixture of the polymer matrix and the liquid electrolyte with various amounts of the PDMITFSI ionic liquid dissolved in the THF solvent. The composition of each gel polymer electrolyte is listed in Table 1. A weight ratio of liquid electrolyte to a polymer matrix was 4:1. The polymer electrolyte films were approximately 150 μm thick.

2.4. Electrochemical measurements

We examined the characteristics of the interface of the gel polymer electrolyte and the lithium electrode by monitoring the impedance of the Li/gel polymer electrolyte/Li symmetrical cells. The lithium electrode had an area of 2 cm × 2 cm. The anodic limit of the gel polymer electrolyte was determined by means of linear sweep voltammetry; we used stainless steel as the working electrode and lithium electrode as the reference and counter electrodes.

2.5. Scanning electron microscopy observation

To investigate how the PDMITFSI ionic liquid affects the formation of dendritic lithium on a metallic lithium electrode, we swept the potential in a linear manner from 0 V to −1.0 V in relation to Li/Li$^+$ at a scan rate of 2 mV s^{-1} in Li/gel polymer electrolyte/Li symmetrical cells (1 cm × 2 cm). The pouch cells were then carefully disassembled in an Ar-filled glove box with less than 1 ppm each of oxygen and moisture, and the Li metal electrodes were subsequently rinsed in a mixed solvent of ethylmethyl carbonate and dimethyl carbonate to remove the residual electrolyte. The Li metal electrodes were dried in the glove box for 60 min. To investigate the surface morphology of the Li metal electrodes after the
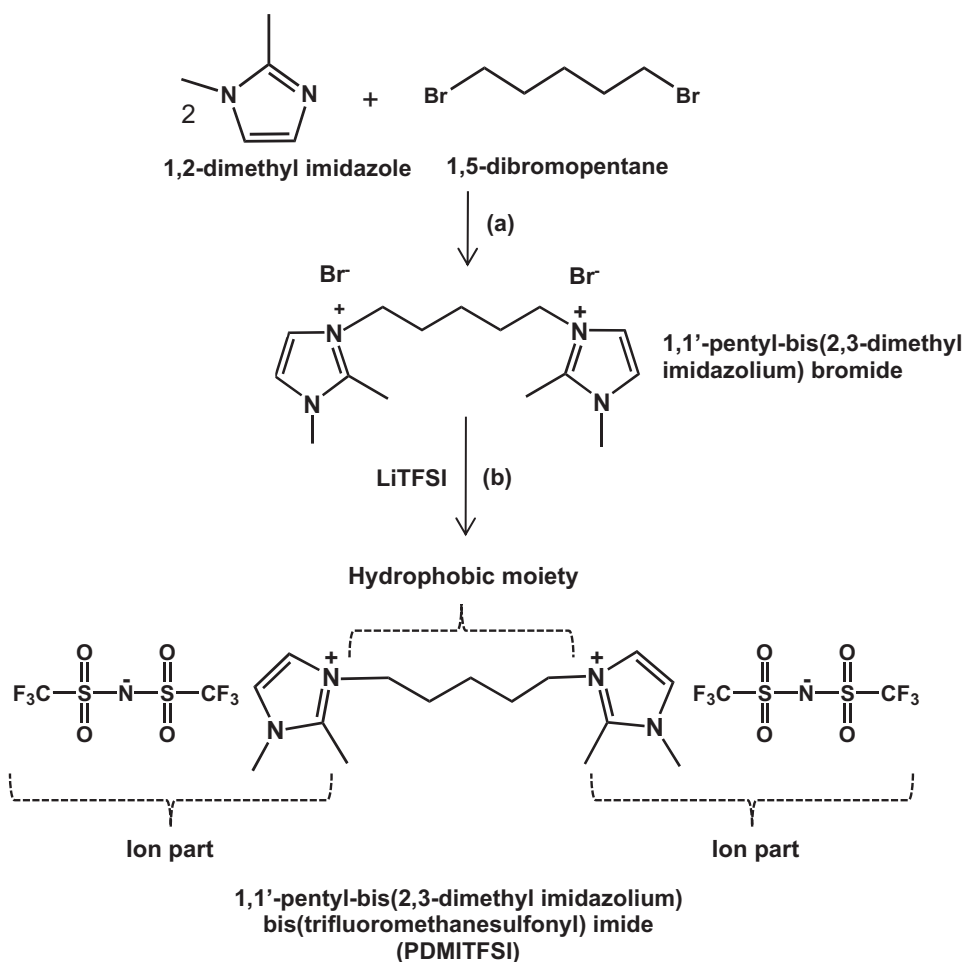
Fig. 1. (a) Reaction of 1,2-dimethyl imidazole and 1,5-dibromopentane for the formation of the 1,1′-pentyl-bis(2,3-dimethyl imidazolium) bromide (PDMIBr), (b) preparation of dimeric ionic liquid (PDMITFSI) via a metathesis reaction.

Table 1
Composition of gel polymer electrolytes.

Gel polymer electrolyte[a]	P(VdF-co-HFP) (wt%)	EC/PC[b] (wt%)	PDMITFSI (wt%)	LiTFSI[c] (wt%)
Gel 1	20	62.0	–	18.0
Gel 2	20	54.7	7.7	17.6
Gel 3	20	47.7	15.0	17.3
Gel 4	20	41.0	22.0	17.0

[a] P(VdF-co-HFP) copolymer was used as a matrix. A weight ratio of liquid electrolyte to a polymer matrix is 4:1.

[b] A volume ratio of EC and PC = 50:50.

[c] Mole concentration of LiTFSI dissolved in a solvent mixture is 1 mol l^{-1}.

first lithium deposition, we used a field-emission scanning electron microscope (FESEM, Quanta 200) at an operational voltage of 15 kV.

3. Results and discussion

Fig. 3 shows the results of differential scanning calorimetry (DSC): the PDMITFSI has a glass transition temperature (T_g) of −20.3 °C, a melting point (T_m) of 73.2 °C, and a heat of fusion (ΔH_m) value of 34 J/g. The DSC results confirm that the synthesized dimeric ionic liquid PDMITFSI is an ionic liquid because its melting point is below 100 °C [22,23].

Fig. 4 shows the impedance spectra of the Li/gel polymer electrolytes with and without synthesized PDMITFSI ionic liquid/Li symmetrical cells. The spectra were taken within 1 h of the cells being prepared. Gel polymer electrolytes with ionic conductivity levels greater than 10^{-3} S/cm can be considered for practical applications. Accordingly, we measured their interfacial resistance levels at 30 °C. The high polarization of the air cathode during the charging process is caused mainly by the precipitation of non-conducting lithium oxide deposits. During the discharge process, the performance of the air cathode is limited mostly by the diffusion of oxygen to the active sites. Furthermore, the low ionic conductivity of an electrolyte can retard the electrochemical reactions of lithium-air batteries as well as the high charge transfer resistance at the lithium–electrolyte interface. For these reasons, most of the experiments were conducted with carbonate-based electrolytes, even though Kuboki et al. achieved good results by using ionic liquids at low current densities of 0.01 and 0.05 mA cm^{-2} [17]. Cell impedance is affected by four components: a high-frequency intercept for the ohmic resistance of a cell (R_e); the physical contact between a gel and a Li electrode (R_f); the impedance associated
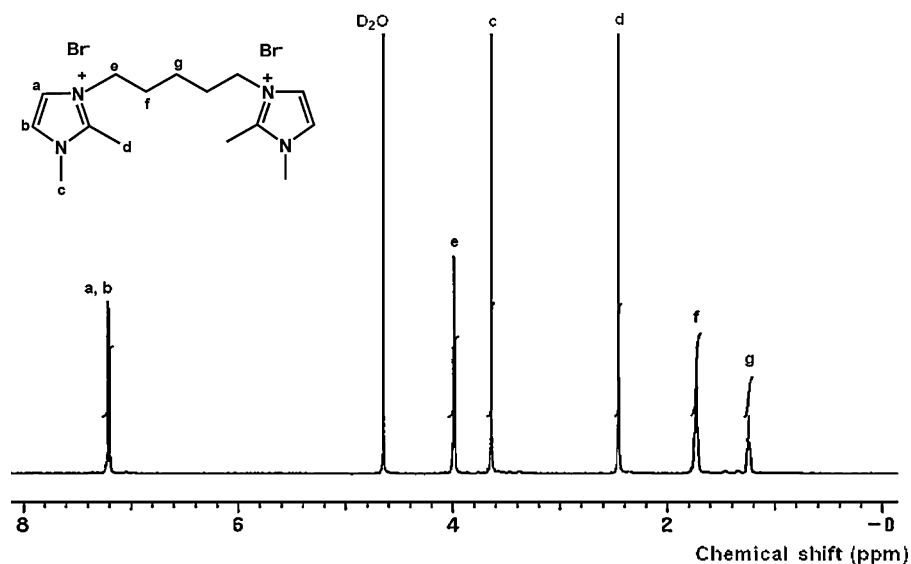
Fig. 2. ^{1}H NMR spectrum of the 1,1′-pentyl-bis(2,3-dimethyl imidazolium) bromide (PDMIBr).
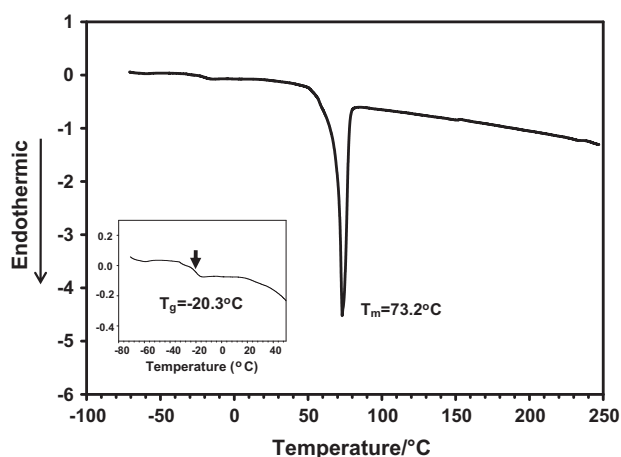

Fig. 3. DSC thermogram of PDMITFSI ionic liquid. The inset presents a glass transition temperature of the ionic liquid.
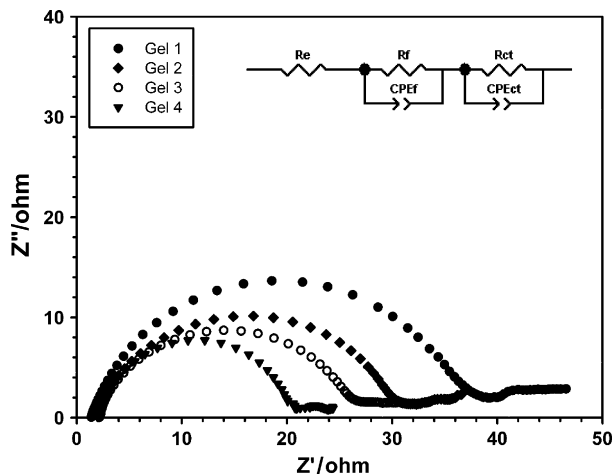

Fig. 4. Impedance diagram for Li/gel polymer electrolyte/Li symmetrical cells at 30 °C.

with Li$^+$ migration across the SEI layer (R_f); and the resistance of a low-frequency Faradaic charge transfer reaction (R_{ct}). The impedance data have been fitted with an ordinary equivalent circuit shown in Fig. 4. Two RC parallel elements in series describe the SEI film on the surface, the physical contact (R_f and CPE$_f$) and Li$^+$ charge transfer at interface (R_{ct} and CPE$_{ct}$), respectively. The circuit includes, in addition, the electrolyte resistance (R_e). The experimental data could be satisfactorily fitted using this equivalent circuit with only slight differences. The simulation results are summarized in Table 2. The gel polymer electrolyte without an ionic liquid has the highest interfacial resistance ($R_f + R_{ct}$) of 41.5 Ω at the initial state. When the PDMITFSI ionic liquid was added, the interfacial resistance of the gel polymer electrolytes decreased. Gel 4 has the lowest interfacial resistance of 21.5 Ω. The difference in the interfacial resistance of all the gels is closely linked to several factors: the physical contact between the gel polymer electrolyte and the Li electrode; the SEI layer formed by the electrolyte decomposition; and the charge transfer resistance. The impedance of Li symmetrical cells was measured within 1 h of the cells being prepared. Therefore, there is no significant difference between the SEI layers formed from gel polymer electrolytes with and without PDMITFSI ionic liquid because the SEI layer is composed mostly of the decomposition products EC and PC, which form the main solvent. A semicircle at a low frequency represents the charge transfer resistance and is analogous among gels. Therefore, the main reason why Gel 4 with the PDMITFSI ionic liquid has a lower level of interfacial resistance is probably related to the better physical contact between Gel 4 and the Li electrode compared to other gels. Previously, we reported that the P(VdF-co-HFP) polymer matrix has poor compatibility with polar organic solvents such as EC and PC [24]. This result suggests that an unwanted phase separation occurs between the liquid electrolyte solution (EC/PC/1 M

Table 2
Impedance parameters obtained from the simulation.

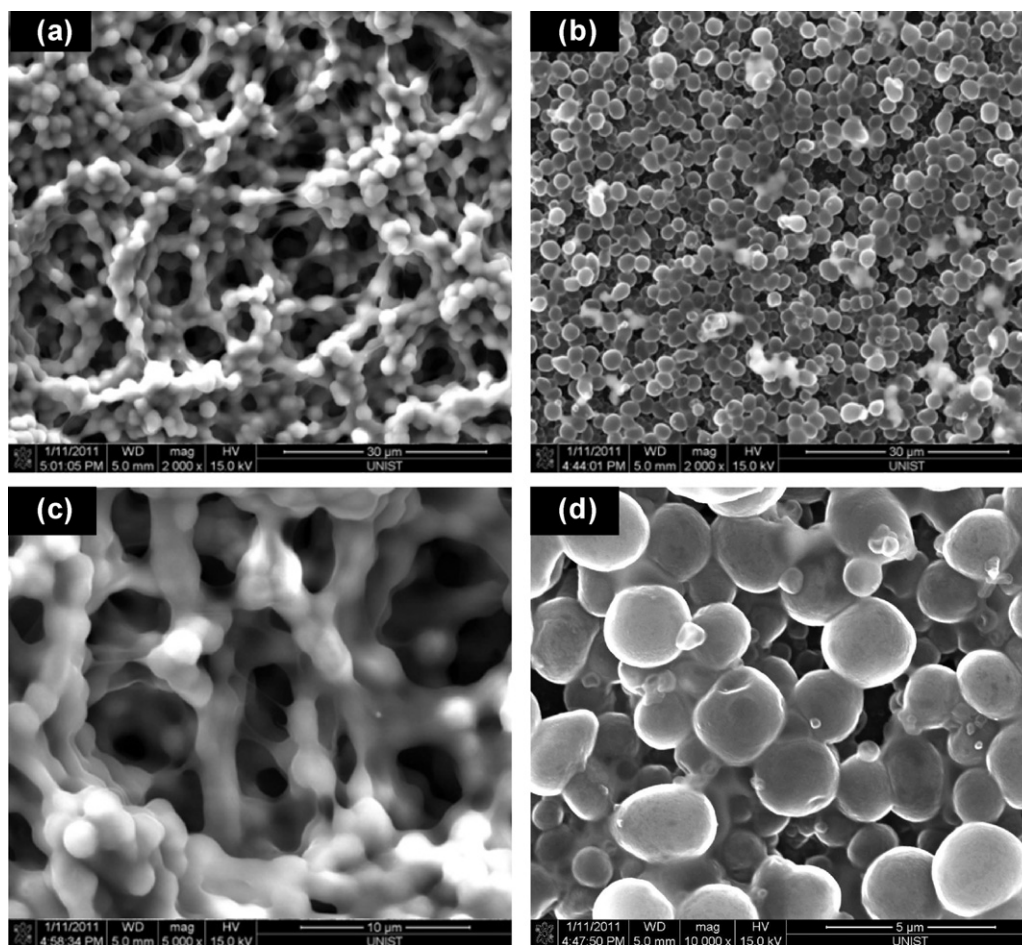|	R_e (Ω)	R_f (Ω)	R_{ct} (Ω)	Total (Ω)
Gel 1	1.38	35.63	5.87	42.88
Gel 2	2.01	27.81	5.49	35.31
Gel 3	2.16	22.89	4.75	29.80
Gel 4	2.3	17.49	4.01	23.8

Fig. 5. SEM images for the surface of lithium metal anodes after the first lithium deposition: (a) Gel 1, (b) Gel 4, (c) enlarged image of (a), and (d) enlarged image of (b).

LiTFSI) and the polymer matrix and causes uneven physical contact between the gel polymer electrolyte and the Li electrode. In the case of gel polymer electrolytes with an ionic liquid, the polar ionic part of the PDMITFSI ionic liquid preferably interacts with the liquid electrolyte, and the nonpolar hydrocarbon moieties are compatible with the P(VdF-co-HFP) polymer matrix. Thus, the PDMITFSI dimeric ionic liquid can be found at the interface of an electrolyte-rich phase and a polymer-rich phase with a small amount of liquid electrolyte; the domain size of the dispersed liquid electrolyte can be effectively reduced in gel polymer electrolytes. This indicates that the liquid electrolyte domain is uniformly distributed when PDMITFSI ionic liquid is added to a gel polymer electrolyte. The uniform distribution of the liquid electrolyte facilitates effective physical contact and allows a low level of interfacial resistance.

During the lithium deposition from 0 V to −1.5 V versus Li/Li$^+$, Gel 1 without the PDMITFSI ionic liquid suddenly generates enormous currents and eventually explodes. This behavior is probably the result of the dendritic lithium that was formed by the nonuniform lithium deposition and the occurrence of an internal short circuit. Because the Li deposition morphology is strongly dependent on the scan rate, the dendritic lithium formation can be mitigated at a low scan rate. To clarify the effect of the PDMITFSI ionic liquid on the dendritic lithium formation, we applied a relatively high scan rate (2 mV s^{-1}) to the Li symmetric cells. Surprisingly, the Gel 4 with the PDMITFSI ionic liquid shows no sign of generating a large current or exploding.

Fig. 5 shows SEM images of the lithium surface morphology; the images obtained after the initial lithium deposition during the linear sweeping of the potential from 0 V to −1.0 V versus Li/Li$^+$ (before any explosion of the Li/gel 1/Li cell). Fig. 5(a) and (c) shows the morphology of the lithium deposition on a Li metal electrode without the PDMITFSI ionic liquid: the dendritic lithium has a reticulated pattern and the morphology is very porous. Viscoelastic gel polymer electrolytes reportedly inhibit the formation of lithium dendrite. The mechanical strength of the gel appears to increase as the poly(acrylonitrile) content increases, and this tendency mitigates the formation of dendritic lithium [25]. In contrast, Gel 1, which is based on the P(VdF-co-HFP) matrix, fails to suppress the formation of lithium dendrite. Essentially, the dendritic lithium can be cut and isolated from the anode substrate. The isolated lithium causes a loss of the anode material and leads to a shorter life cycle. Moreover, lithium-air batteries capable of repeated cycling cannot be developed because of the difficulty of uniformly dissolving lithium from the dendritic lithium. However, as shown in Fig. 5(b) and (d), when PDMITFSI ionic liquid is added to the gel polymer electrolyte, the deposited lithium is formed with a particle-like shape. It is so difficult that lithium ions readily migrate through a polymer-rich phase because this phase serves as a barrier to the lithium deposition. Lithium is thought to be mainly deposited through an electrolyte-rich phase, which serves as an appropriate pathway of ion conduction. Even though lithium is preferential to deposit through an electrolyte-rich phase, Fig. 5(b) shows no sign of the formation of lithium dendrite. The discernable difference in the deposited lithium of Gel 1 and Gel 4 is most likely related to the morphological change and the increase in ion concentration induced when PDMITFSI ionic liquid is added to the gel polymer electrolytes.
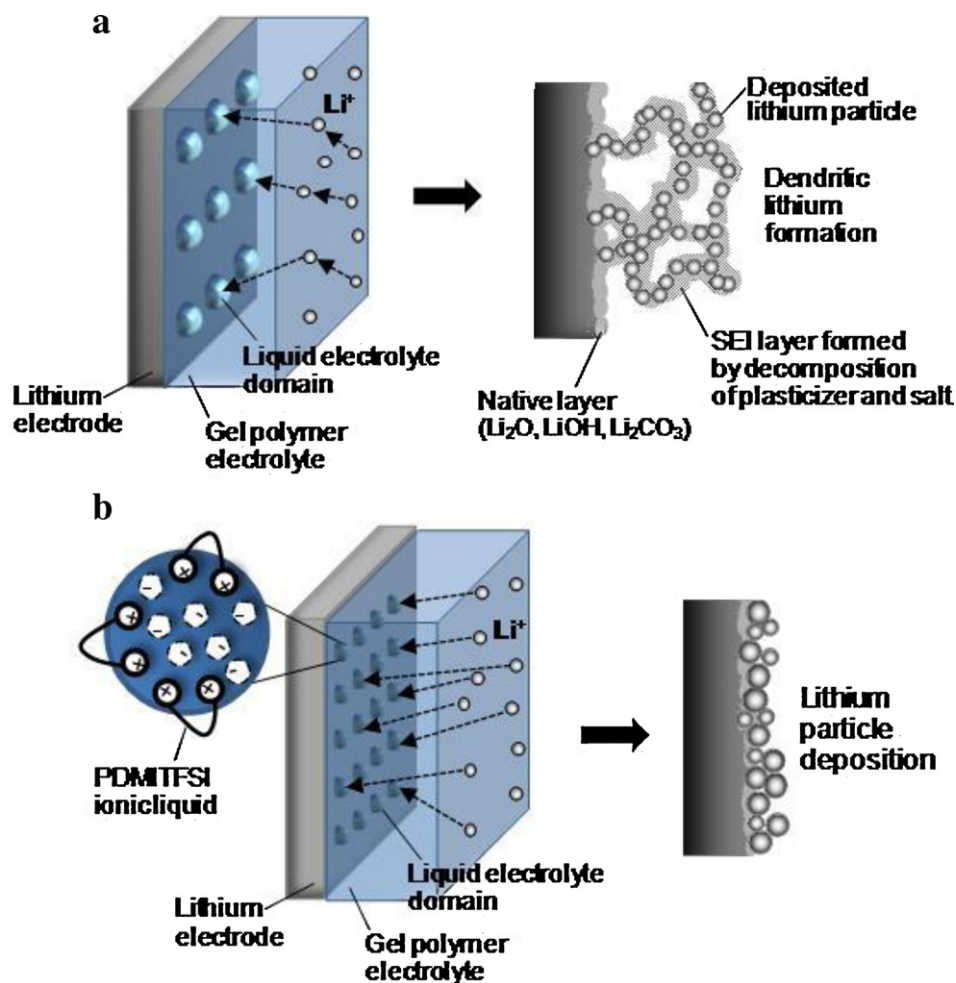
Fig. 6. Schematic presentation for functional role of PDMITFSI ionic liquid on lithium deposition: (a) Gel 1, (b) Gel 4.

Fig. 6 depicts the possible mechanism of Li deposition on a Li electrode. When the PDMITFSI ionic liquid is present at the interface of the polymer-rich phase and the liquid electrolyte-rich phase, the domain size of the dispersed liquid electrolyte can be drastically reduced and the number of electrolyte-rich phases can be increased. This behavior occurs because the hydrophobic alkyl moieties and the polar ionic parts of the PDMITFSI ionic liquid serve as a compatibilizer which attenuates the phase separation between the polymer matrix and the electrolyte solution. Fig. 6(b) shows an increase in the number of dispersed electrolyte-rich domains. This increase indicates that there are many sites into which lithium can be deposited uniformly, especially in comparison with the Li/Li symmetric cells without the PDMITFSI ionic liquid. Moreover, as illustrated in Fig. 6(b), the ionic parts (PDMI$^+$ cations and TFSI$^-$ anions) of the PDMITFSI ionic liquid are mostly present in the electrolyte-rich phase due to the ion-dipole interaction between PDMITFSI and the EC/PC solvent. This behavior is a likely cause of the increase in the ion concentration of the liquid electrolyte domain and the reason why the lithium is deposited in a particle-like shape. The formation of dendritic lithium produces a porous structure, which provokes a colossal volume expansion of cells. In contrast, when the lithium deposition is uniform in terms of the particle shape, there is likely to be a dense structure with no significant change in volume. It is therefore crucial to control the interface of dendrite-free lithium metal anodes.

Fig. 7 presents typical linear sweep voltammograms for Li/gel polymer electrolytes/stainless steel cells. The results confirm that Gel 4 with the PDMITFSI ionic liquid is not electrochemically oxidized by 5.25 V versus Li/Li$^+$; it also has a slightly better anodic limit than Gel 1.
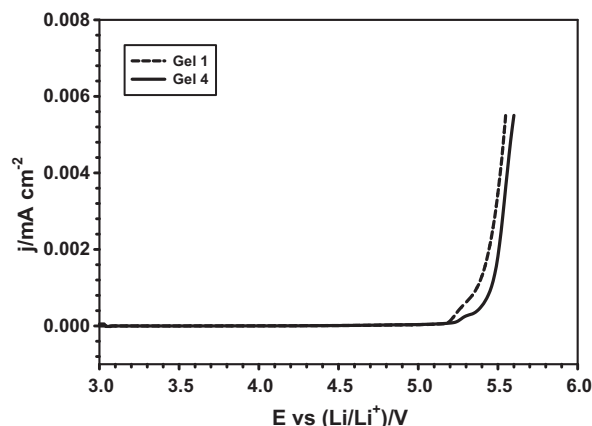

Fig. 7. Linear sweep voltammograms of Gel 4 at a scan rate of 2 mV s^{-1}.

4. Conclusions

A dimeric ionic liquid with hydrophobic moieties, two imidazolium cations, and two TFSI$^-$ anions was successfully synthesized. The addition of PDMITFSI ionic liquid instead of EC/PC solvents reduced the ionic conductivity levels, lowered the interfacial resistance levels of gel polymer electrolytes, and makes the lithium deposition and dissolution processes reversible in lithium symmetric cells. Moreover, the dendritic lithium formation was significantly mitigated when PDMITFSI ionic liquid was used as a compatibilizer to modulate the electrolyte-rich phase for a lithium deposit pathway on the lithium metal anode. These findings provide guidance on the efficient design of viable electrolytes; specifically, they helped prevent the formation of dendritic lithium on the lithium anode of a lithium-air battery.

Acknowledgements

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