Characterization of UV-cured gel polymer electrolytes for rechargeable lithium batteries

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Abstract

Novel ultraviolet (UV)-cured gel polymer electrolytes based on polyethyleneglycol diacrylate (PEGDA) oligomer and polyvinylidene fluoride (PVdF) are prepared and characterized. UV-curing of PEGDA oligomer containing PVdF and ethylene carbonate (EC)-based liquid electrolyte yields chemically and physically cross-linked PEGDA/PVdF blend gel electrolytes. PEGDA/PVdF blend films show much higher mechanical properties and electrolyte liquid retention than pure PEGDA film. The ionic conductivity (σ) of a PEGDA/PVdF (5/5) blend electrolyte reaches about $4 \, \mathrm{mS \, cm^{-1}}$ at ambient temperature and is as high as $1 \, \mathrm{mS \, cm^{-1}}$ at $0 \, ^\circ C$. All the blend electrolytes are electrochemically stable up to 4.6 V versus Li/Li$^+$. The cation transference number (t^+) measured by dc micropolarization exceeds 0.5 at room temperature. Li/(PEGDA/PVdF)/LiCoO$_2$ cells (2 cm × 2 cm) retains >91% of its initial discharge capacity after 50 cycles at the C/3 rate ($2 \, \mathrm{mA \, cm^{-2}}$) and delivers about 70% of full capacity with an average load voltage of 3.6 V at the C/1 rate. Cell performance is stable up to 80 $^\circ$C because PVdF chains might be stabilized by entanglement with the chemically cross-linked PEGDA network structure. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

World-wide research and development effort has been devoted to commercialize high performance lithium secondary batteries using solid polymer electrolytes because of their attractive potential applications to electric vehicles as well as portable electronic devices [1,2]. Polyether-based dry polymer electrolytes still suffer from poor conductivity at ambient temperature [3] while, to date, various gel polymer electrolytes plasticized by organic liquid solvents have been reported to display promising room temperature conductivity as high as $1 \times 10^{-3} \, \mathrm{S \, cm^{-1}}$ [4]. State-of-the-art PVdF gel electrolytes are usually prepared by dissolving polymer powders in highly permeable ethylene carbonate (EC)-based liquid electrolyte solutions at elevated temperature [5,6]. This procedure needs, however, long mixing and drying times to form free-standing polymer films owing to viscosity build-up in the polymer melt state. In addition, the polymer films may swell and dissolve in EC-based solvents by heat generated under abusive cell operation and may also undergo severe solvent exudation from the physically cross-linked polymer structure upon long storage [7].

On the other hand, ultraviolet (UV) or electron-beam curing methods are well established to obtain chemically cross-linked gel electrolytes at ambient temperature, and it is known that this technique produces more thermally stable electrolyte films [8]. To prepare UV-cured gel polymer electrolyte films, a liquid solution containing a UV-curable oligomer and a photo-initiator is mixed with liquid electrolytes. The mixture is then poured on to either lithium foil or release paper, and cured under UV irradiation within a few seconds to minutes [9]. Thus, the UV-curing process is able to facilitate scale-up and mass productivity of lithium-polymer batteries. There are, however, major drawbacks to large-scale commercial use, namely, weak mechanical strength and poor interfacial properties. Conventional UV-cured films cannot maintain mechanical integrity under external pressure or tensile elongation for practical battery assembly procedures [10]. Furthermore, their glassy surface has a small apparent contact area with the adjacent electrode as the physical contact occurs only at the asperity of the microscopically rough electrode surface, which tends to cause detrimental interfacial resistance between the electrode and the electrolyte film.

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To improve the mechanical and electrochemical properties of UV-cured polymer electrolyte systems, we first prepared a gel polymer electrolytes based on polyethyleneglycol diacrylate (PEGDA) oligomer and PVdF–hexafluoropropylene (HFP) copolymer. A series of PVdF–HFP polymer solutions was mixed with liquid PEGDA oligomer in the presence of EC-based liquid electrolytes and cured under UV irradiation at room temperature. The high miscibility of the PEGDA/PVdF blend solution might be attributed to homogeneous mixing between liquid-like PEGDA oligomer and dilute PVdF solution. As a result, this approach is expected to produce deformable and rubbery UV-cured gel polymer electrolyte films, while still maintaining the essential ionic conductivity. This paper presents the characterization of UV-cured PEGDA/PVdF blend gel polymer electrolytes and their application in rechargeable lithium batteries.

2. Experimental

PVdF–HFP copolymer (Elf Atochem Co., Kynar2801) was dissolved in organic liquid solvents such as dimethylformamide (DMF, Aldrich), dimethylacetamide (DMA, Aldrich) and *N*-methyl-2-pyrrolidinone (NMP, Aldrich). Kynar2801 solutions were mechanically mixed with liquid PEGDA oligomer (Kyoeisha Chemical, MW = 742) and organic liquid electrolytes for 2 h, then a photo-initiator, 2-hydroxy-2-methyl-1-1-phenyl propane-1-on (Ciba Geigy, Darocuur 1173), and a curing accelerator, triethylamine (TEA, Junsei Chemical), were added to the solution mixture. The resultant solution was coated on to a Mylar film and cured under UV irradiation (Sankyo Denki GL 20, $\lambda = 375$ nm). A battery-grade electrolyte solution of EC and dimethyl carbonate (DMC) containing lithium salt (Merck, EC/DMC = 50/50 vol.%, 1 M LiPF$_6$) was used as a liquid electrolyte.

UV–VIS spectroscopy (Jasco V570 Spectrophotometer) was used to measure the optical transmittance of blend films. Tensile tests were carried out on an Instron Model 4201 tester, in accordance with ASTM D882-88. The test specimens were 6 mm in width and approximately 100 μm in thickness. The grip distance was 50 mm and the cross-head speed was 25 mm per min. The cross-sectional morphology of blend films was examined by scanning electron microscope (SEM, Hitachi S-2500C) at 10 kV. All samples were prepared by cryogenic fracture after immersion in liquid nitrogen for 5 min.

The ionic conductivity (σ) of blend films was determined by ac impedance spectroscopy (Zahner IM6 impedance analyzer). Sample films were sandwiched between two parallel stainless-steel (SUS) discs ($\phi = 1$ cm^2). The frequency ranged from 1 MHz to 1 Hz at a perturbation voltage of 5 mV. The cationic transference number (t^+) was measured by a micropolarization method. A bias of 10 mV was applied to Li|SPE|Li cell and the response of the cell current

was monitored until it reached a steady-state value. The electrochemical stability window was studied by linear sweep voltammetry using an EG&G Model 362 potentiostat. Lithium metal and a SUS disc ($\phi = 1$ cm^2) were used as electrodes.

Lithium cells using PEGDA/PVdF blend electrolytes were fabricated by sequential lamination of LiCoO$_2$ cathode (Seimi Co., 2 cm × 2 cm), PEGDA/PVdF film, and lithium metal anode (Chemmetal Co., 3 cm × 3 cm). The weight ratio of active material, carbon conductor (Lonza Co., graphite/acetylene black = 7/3 w/w) and polymer binder (Elf Atochem, Kynar 761) was 100:10:6. The cathode slurry was dip-coated on to an extended aluminum grid (Exmet Co.) current-collector and dried at 100 °C for 48 h in a vacuum oven. Cathode laminates (ca. 50 mg cm^{-2}) were roll-pressed under appropriate pressure to obtain uniform thickness. Finally, Li|SPE|LiCoO$_2$ cells were enclosed in a metallized plastic bag by a vacuum sealer. All procedures were carried out in a dry room where the moisture content is below 10 ppm. The galvanostatic charge–discharge performance was examined between 3.0 and 4.2 V (WonA Tech Co., Battery Cycler 3000).

3. Results and discussion

3.1. Polymer blend characterization

PEGDA and PVdF have different glass transition temperatures, T_g, and thermal analysis is a valuable tool to determine whether PEGDA/PVdF blend is miscible or whether they exist in different phases. Unfortunately, it was difficult to find the T_g of cross-linked PEGDA in a gel state by DSC or DMA, and we measured the optical transmittance of blend films. The UV–VIS spectra of UV-cured PEGDA/PVdF (5/5) blend films are shown in Fig. 1. The existence of lithium salts in blend films had no effect on the transmittance pattern. UV-cured PEGDA films are highly transparent and the transmittance is usually above 93%. Only PEGDA/PVdF/DMF showed similar transmittance over the entire visible range. Kynar2801 solution
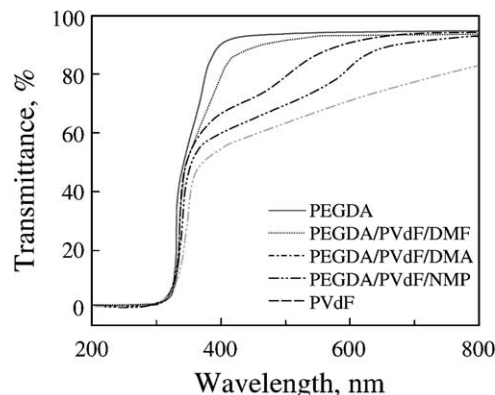

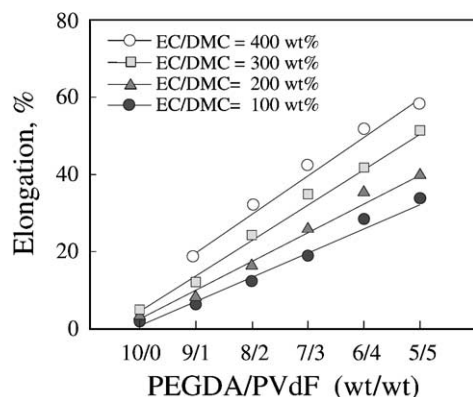Fig. 1. UV–VIS spectra of UV-cured PEGDA/PVdF blend films.

Fig. 2. Tensile elongation at breakage of UV-cured PEGDA/PVdF blend films.

Fig. 3. Electron micrographs of UV-cured polymer electrolytes: (a) PEGDA; (b) PEGDA/PVdF (5/5).

prepared from DMA and NMP was partially miscible with PEGDA at a blend ratio of 5/5 and sporadically whitish crystallites decreased the transmittance. Completely miscible polymer blends are less commonly found, but PEGDA oligomer is a low viscous liquid at room temperature when its molecular weight is <1000 and thus, it is easy to form a completely miscible solution blend with PVdF/DMF solution. Accordingly, UV-cured PEGDA/PVdF blend films prepared from PVdF/DMF solution were used as polymer electrolytes for mechanical and electrochemical studies.

The tensile elongation at break of PEGDA/PVdF blend gel films is given in Fig. 2 as a function of blend composition. In immiscible polymer blends, the interfacial adhesion is usually not sufficiently strong for stress to be efficiently transferred from one phase to another during yielding or fracture. This results in poor mechanical properties [11]. Therefore, measurement of the tensile properties of PEGDA/PVdF blends is another way to estimate the compatibility of PEGDA with PVdF. In particular, the tensile elongation at break is very sensitive to the strength of the interface and is commonly measured to examine the efficiency of the blend [12]. The tensile elongation of commercial Kynar2801 usually exceeds 200% but that of UV-cured PEGDA films is near zero due to their high brittleness. The elongation was improved by up to 60% in a PEGDA/PVdF (5/5) blend film by incorporating 400 wt.% of EC/DMC electrolyte solution (organic liquid electrolyte/dry polymer = 4/1 w/w). In general, UV-cured PEGDA gel electrolytes cannot be obtained as a free-standing films above EC/DMC = 300 wt.% even at high concentrations of photo-initiator because of the dilution effect, but UV-cured PEGDA/PVdF blend films show good mechanical integrity above EC/DMC = 400 wt.%. As shown in Fig. 3, the cross-sectional SEM image of the PEGDA/PVdF blend film is nearly same as that of the microporous PVdF gel electrolyte [13]. It seems that the structural change in the PEGDA/PVdF blend is responsible for uptaking a large amount of liquid electrolyte.

The increase in mechanical properties might be associated with entanglement of the PEGDA network structure and the PVdF chains. In this blend system, unravelled PVdF chains
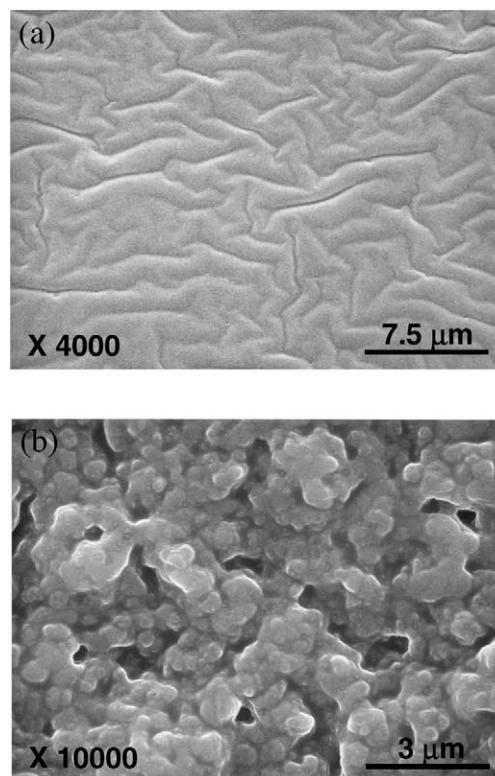
are randomly mixed with liquid PEGDA oligomer, then PVdF chains are just trapped in the PEGDA network frame formed by UV cross-linking of PEGDA oligomer. This cross-linking is eventually accompanied by physical cross-linking of PVdF with liquid electrolyte. Both chemical and physical cross-linking makes it possible to form rubbery and deformable PEGDA/PVdF blend gel electrolytes with a fully amorphous structure. PVdF-based gel polymer electrolytes are known to be one of the best candidates for lithium-polymer batteries, but they locally dissolved in organic liquid electrolytes above 60 °C and also leak liquid electrolyte solution from the structure with time [14]. Therefore, a PEGDA/PVdF blend prepared by chemical cross-linking simultaneously with physical cross-linking may be an unique approach to preventing the syneresis effect of PVdF and the brittleness of PEGDA.

3.2. Electrochemical properties of blend films

Arrehenius plots of the ionic conductivity of UV-cured PEGDA/PVdF blend gel electrolytes at different blend compositions are presented in Fig. 4. The ionic conductivity of a pure PEGDA film is below 1×10^{-3} S cm^{-1} until the temperature reaches about 60 °C. The relatively rapid increase in conductivity at high temperatures is partly due to the large flexibility of ethylene oxide units between cross-links in the PEGDA network structure [15]. The ionic
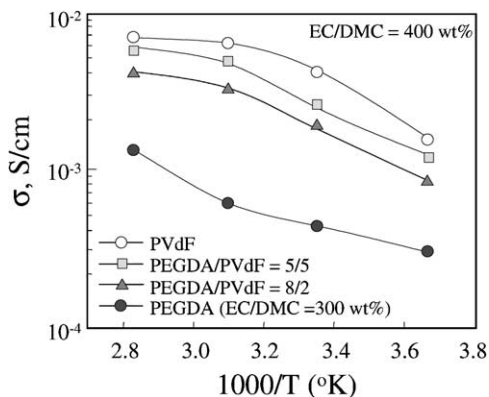
Fig. 4. Ionic conductivity of PEGDA/PVdF blend electrolytes.

conductivity of the PEGDA/PVdF (5/5) blend is approximately 4×10^{-3} S cm^{-1} at room temperature and exceeds 1×10^{-3} S cm^{-1} even at 0 °C. These conductivities are much higher than that of a pure PEGDA film. Also, it can be noticed that the increase in ionic conductivity at higher PVdF content is related to better electrolyte liquid retention of PVdF.

Typical current–voltage curves of Li|(PEGDA/PVdF)|SUS cells at room temperature are given in Fig. 5. The electrochemical stability window of a given SPE is generally determined by linear sweep voltammetry of an inert electrode in the selected electrolyte [16]. The onset of the current in the anodic high-voltage range is assumed to result from a decomposition process associated with the electrode [17], and this onset voltage is taken as the upper limit of the electrolyte stability range. This voltage is generally located at the point of intersection of the extrapolated linear current in the high-voltage region with the voltage axis. For all blend electrolytes, the onset of the current increase is above 4.7 V versus Li/Li$^+$, and PEGDA/PVdF blend electrolytes have suitable electrochemical stability to allow the use of high-voltage electrode couples such as LiCoO$_2$, LiNiO$_2$ and LiMn$_2$O$_4$.

Impedance analysis is used to clarify the compatibility of PEGDA/PVdF blend electrolytes with the lithium electrode. The ac impedance spectra of Li|PEGDA|Li cell as a function
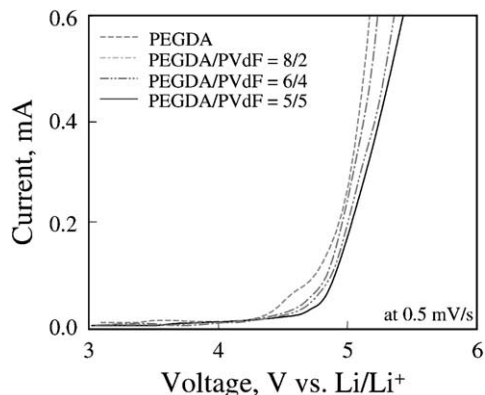
of ageing time under open-circuit conditions at room temperature are shown in Fig. 6(a). There is a progressive expansion of the semicircle in the middle frequency range, and eventually it is almost stabilized at a constant value of about 115 Ω because of the immobilization of liquid solvent in the bulk of the polymer electrolyte [18]. This semicircle is a measure of the continuous growth of a resistive layer on the lithium metal surface [18], and the growth of arcs implies that lithium metal electrodes are readily passivated in contact with PEGDA/EC/DMC/LiPF$_6$ electrolytes.

A PEGDA/PVdF (5/5) blend film also shows similar impedance behavior, as shown in Fig. 6(b), but the rate of growth of the resistive layer on lithium metal is significantly reduced. The possible reason for the improved interface is the blend film being more rubbery and viscous than the glassy PEGDA film. This physical stability could decrease the resistive layer growth by suppressing the flow of corrosive solvents to the interface [19]. It is obvious from impedance results that the PEGDA/PVdF blend electrolyte exhibits good compatibility with lithium metal and it is a better reservoir for EC-based liquid electrolyte solution than pure PEGDA electrolyte.

Since the electrochemical process in lithium batteries involves the intercalation and de-intercalation of lithium cations throughout the host compound lattice, solid polymer electrolytes with cation transference numbers (t^+)
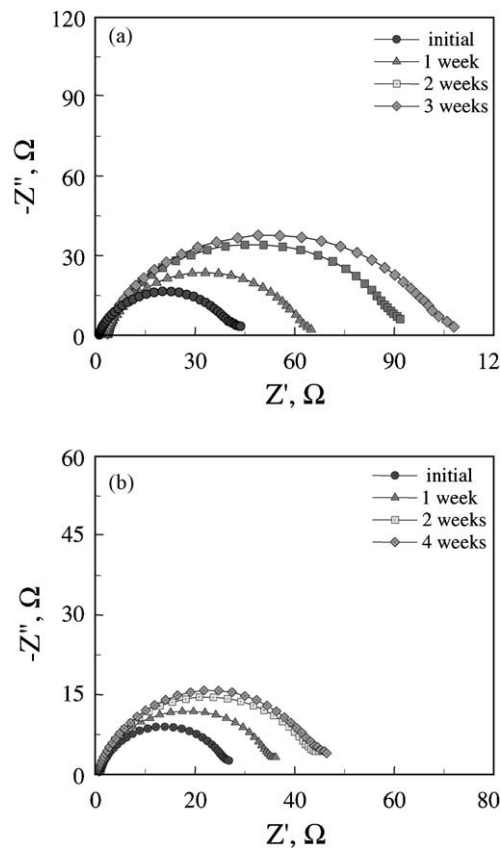

Fig. 5. Linear sweep voltammetry of Li|(PEGDA/PVdF)|SUS cells.

Fig. 6. The ac impedance spectra of Li|SPE|Li cells: (a) PEGDA; (b) PEGDA/PVdF.

approaching unity are desirable for avoiding a concentration gradient during repeated charge–discharge cycles. Thus, the evaluation of t^+ is of great importance for the characterization of polymer electrolytes. Various methods have been used to determine t^+ in polymer electrolytes [20,21]. For example, Bruce et al. have proposed a reproducible and reliable method based on a combination of dc polarization and ac impedance. This method involves of measuring the resistance and current across a symmetrical Li|SPE|Li cell polarized by a dc voltage pulse. In this condition, t^+ is given by the following expression [22]:

$$t^+ = \frac{I_s(V - I_0 R_0)}{I_0(V - I_s R_s)} \tag{1}$$

where V is the dc voltage applied to the cell; R_0 and R_s the initial and steady-state resistances of the passivating layer; I_0 and I_s the initial and steady-state currents.

The impedance response of a Li|(PEGDA/PVdF)|Li cell, inset of Fig. 7(a), was monitored at initial time and at a steady-state current condition. The decrease in current may result from the growth of passivating layers to a limiting thickness at the electrode [23]. The current response is

completely stabilized after 20 h. The t^+ values of PEGDA/PVdF blend electrolytes at different compositions are shown in Fig. 7(b). PEGDA/PVdF (5/5) blend films show t^+ as high as 0.5 above EC/DMC = 300 wt.%. The reported t^+ values for dry polymer electrolytes range from 0.06 to 0.2 [24]. For a gel polymer system, t^+ values of order 0.4–0.5 have been found for poly(bis-methosxy ethoxy ethoxy)phosphazene by Abraham and Alamgir [25], and Mastuda et al. reported a value of $t^+ = 0.56$ for PEO/PMMA systems [26]. Hence, a t^+ of about 0.5 for the PEGDA/PVdF blend system is an acceptable level for a gel polymer electrolyte.

3.3. Battery performance

Li|LiCoO$_2$ cells of 2 cm × 2 cm area were fabricated with PEGDA and PEGDA/PVdF blend electrolyte, respectively. The cycleability of Li$_x$CoO$_2$ is generally limited to $x = 0.5$, giving a practically specific capacity of 137 mAh g^{-1}. For a value x of lower than 0.5, the oxidation of Co^{3+} to Co^{4+} becomes possible which may also transform the crystallinity of the LiCoO$_2$ cathode and affect the crystal structure to cause a decrease in reversibility [27]. The performance of Li|LiCoO$_2$ cells using pure PEGDA film is shown in Fig. 8(a). This cell was charged and discharged at the C/3
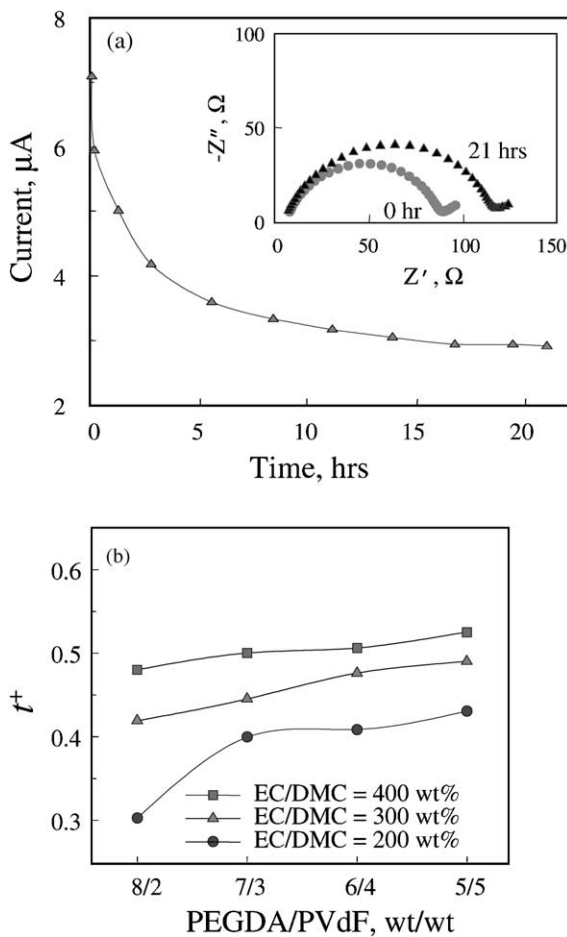

Fig. 7. Cationic transference number (t^+) measurement of PEGDA/PVdF blend electrolytes: (a) dc micropolarization test; (b) t^+ as a function of PVdF content.
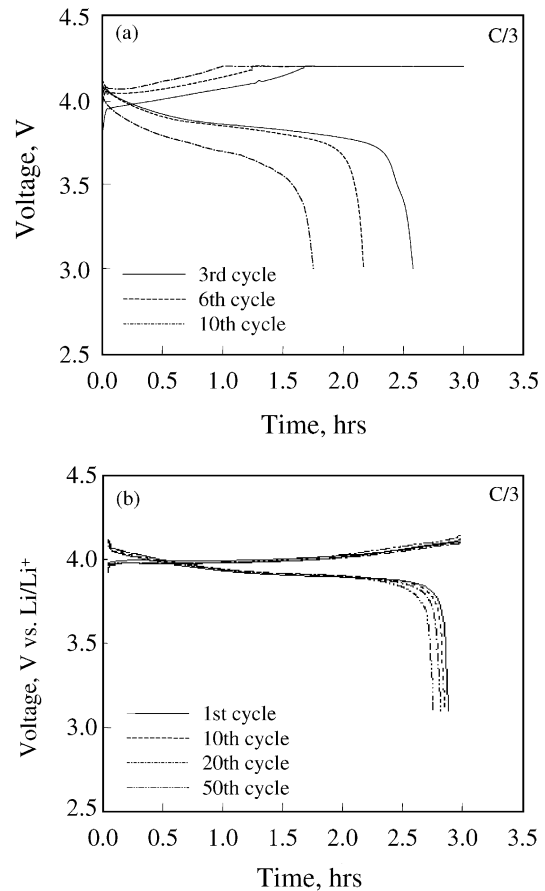

Fig. 8. Battery performance of Li|LiCoO$_2$ cell: (a) PEGDA; (b) PEGDA/PVdF (5/5).

rate (ca. 2 mA cm^{-2}) between 3.0 and 4.2 V. The initial capacity was slightly increased up to 120 mAh g^{-1} during the first few cycles because of the initial poor interfacial contact between the electrolyte and the electrodes. Nevertheless, the discharge capacity decreases rapidly during the following several cycles. The large irreversible capacity, short cycle-life and poor columbic efficiency are primarily ascribed to low ionic conductivity and poor interfacial properties. As a result, conventional UV-cured gel electrolytes are inadequate for practical applications despite their high production rate.

The charge–discharge profiles of Li|(PEGDA/PVdF)|LiCoO$_2$ cells at the C/3 rate are given in Fig. 8(b). The intercalation of lithium cations in LiCoO$_2$ gives rise to a voltage plateau at about 3.8 V versus Li/Li$^+$. It is evident from Fig. 8(b) that very good cycleability is realized for a PEGDA/PVdF (5/5) blend electrolyte when cycled between 3.0 and 4.2 V. The capacity decreases more slowly during cycling and this cell retains >92% of the initial discharge capacity after 50 cycles, as shown in Fig. 8(b). The small capacity loss could be primarily associated with passivation of the lithium metal anode by its reaction with liquid

electrolyte [28]. The good capacity retention at higher PVdF content is in good agreement with the ionic conductivity results, as shown in Fig. 4.

The rate capabilities of Li|LiCoO$_2$ cells using PEGDA/PVdF (5/5) blend electrolytes are given in Fig. 9(a). The Li|(PEGDA/PVdF)|LiCoO$_2$ cell at the C/5 rate reached close to 100% nominal capacity. At the C/3 rate, this cell delivered about 95% of the nominal cathode capacity at a load voltage of 3.8 V. The reduced capacity at high rates is due to the low value of the chemical diffusion coefficient of lithium ions in the lattice of LiCoO$_2$, and the lower diffusion rate of lithium ions in a solid polymer electrolyte compared with that in liquid electrolyte. At the C/1 rate, this cell delivers about 70% of the full capacity at an average load voltage of 3.5 V.

The discharge capacity of Li/LiCoO$_2$ cells using a PEGDA/PVdF (5/5) blend electrolyte was also tested at different temperatures. After assembly, each cell was stored at the test temperature condition for 3 days prior to charge–discharge reaction. The Li|(PEGDA/PVdF)|LiCoO$_2$ cell retained 85% of the room temperature discharge capacity at −4 °C, as illustrated in Fig. 9(b). The capacity loss at low temperature is generally caused by large polarization of the lithium ions at the electrode/electrolyte interface [29]. It is interesting to note that a slightly higher capacity is obtained at 80 °C and that the capacity loss is less than that at room temperature. The slight increase in discharge capacity is partially due to the large flexibility of (ethylene oxide) units between cross-links in the PEGDA network given the effect of temperature on the ionic conductivity of a pure PEGDA film. PVdF chains are physically stabilized by entanglement with the chemically cross-linked PEGDA network frame, and the physically and chemically cross-linked PEGDA/PVdF polymer blend structure may account for the observed improvement in thermal stability.
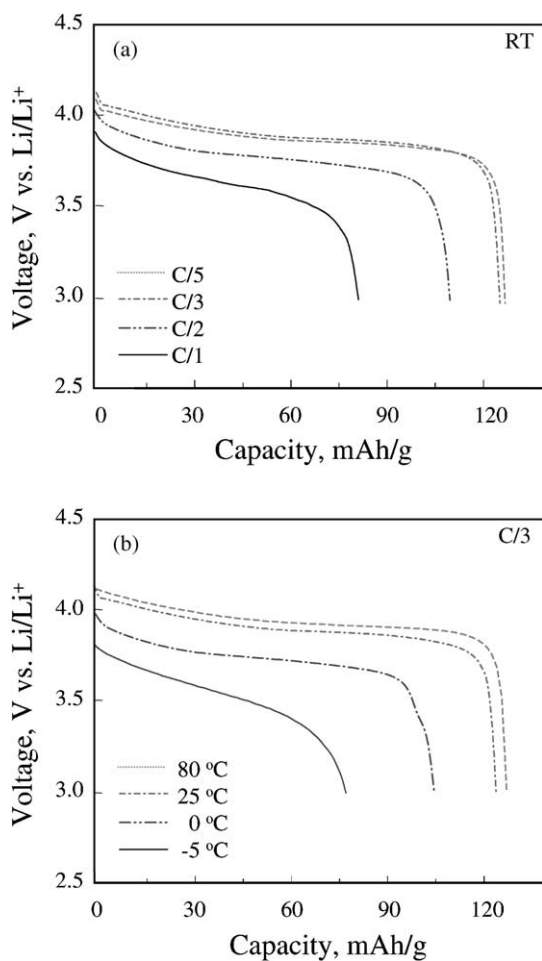

Fig. 9. Battery performance of Li|(PEGDA/PVdF)|LiCoO$_2$ cell: (a) rate capability; (b) temperature dependency.

4. Conclusions

Despite highly desirable mass productivity, the mechanical properties of conventional UV-cured PEGDA films appear to be a major limitation for these materials to become commercially viable. It is found, however, that liquid PEGDA oligomer is completely miscible with PVdF/DMF solution. A rubbery and deformable UV-cured film has been obtained by chemical and physical cross-linking of PEGDA/PVdF solution in the presence of EC-based liquid electrolytes. The addition of PVdF into the PEGDA network improves the tensile elongation of a UV-cured PEGDA/PVdF blend gel by up to 60% from near zero. PEGDA/PVdF (5/5) blend gel electrolytes have an ionic conductivity of 4 mS cm^{-1} at room temperature without any inorganic additives. The conductivity is higher than 1 mS cm^{-1} even at 0 °C. The PEGDA/PVdF blend electrolyte is electrochemically stable up to 4.7 V versus Li/Li$^+$. In addition, impedance studies confirm that the PEGDA/PVdF blend electrolyte has better compatibility with lithium metal and a

good cationic transference number as a solid polymer electrolyte. Li|(PEGDA/PVdF)|LiCoO$_2$ cells show comparable cycleability and rate capability to common lithium ion cells. Furthermore, the thermal instability of PVdF can be minimized by means of entanglement of the PVdF chain with the PEGDA network frame. Consequently, the synergetic performance of the UV-cured PEGDA/PVdF blend system appears to be highly suitable for large-scale, commercial lithium polymer batteries.

Acknowledgements

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