Research Square

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Preparation of tetra-branched PEG network and its application to gel polymer electrolyte

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Abstract

Thiol-yne click reaction between pentaerythritol tetrakis(3-mercaptopropionate) and α, ω-dipropiolate-terminated polyethylene glycol was carried out in propylene carbonate containing lithium bis(trifluoromethanesulfonyl)imideto obtain gel polymer electrolyte with tetra-branched network structure. The ionic conductivity was found to depend on the polymer content in the gel polymer electrolyte and the chain length of the polyethylene glycol constituting the tetra-branched network structure. These experimental results indicated that lithium cations moved not only within the electrolyte solution but also along the PEG chains that form a network structure.

Introduction

Lithium-ion batteries are characterized by their high efficiency and high energy density, and are used in a wide range of applications, including not only power sources for small portable devices but also power sources for electric vehicles and aircraft. However, because they use flammable organic solvents as electrolytes, there are concerns regarding the risk of fire. Therefore, preventing electrolyte leakage and ensuring safety are critical practical challenges [1]. Solid-state electrolytes are a key technology for safe lithium-ion batteries. Inorganic solid electrolytes and polymer solid electrolytes are well-known as promising candidates for solid-state electrolytes [2–5].

Meanwhile, gel polymer electrolytes, which are solidified forms of liquid electrolytes, are also attracting attention as a means of preventing electrolyte leakage [6–8]. Gel polymer electrolytes are network polymers swollen with liquid electrolytes. They serve the dual roles of both electrolyte and separator, thereby eliminating the risk of electrolyte leakage. Gel polymer electrolytes offer an advantage over inorganic solid electrolytes in terms of forming a robust interface with the electrodes. Furthermore, regarding ionic conductivity, they can achieve levels comparable to those of liquid electrolytes. Gel polymer electrolytes combine the high diffusivity of liquids with the high affinity for electrodes.

Polymers to be used as matrices for gel polymer electrolytes include poly(vinylidene fluoride-hexafluoropropylene) (PVDF-HFP) [9], poly(methyl methacrylate) (PMMA) [10], and polyacrylonitrile (PAN) [11]. Cross-linked PEO is also a cost-effective matrix material for gel polymer electrolytes that possesses moderate mechanical and thermal stability. The CH_2CH_2O repeating units in the PEO main chain exhibit strong interactions with Li ions, enabling better charge transport. Cross-linked PEO as a gel electrolyte matrix is often prepared using a method that employs multifunctional acrylic acid derivatives containing PEG segments as cross-linking agents [12–16].

Sakai et al. prepared tetra-arm polyethylene glycol (tetra-PEG) hydrogels with ideally homogeneous network structure from two symmetrical tetra-arm macromonomers and found that these hydrogels exhibit high mechanical properties even at low polymer content in the hydrogel [17–19]. Tetra-PEG network can be applied to material as a matrix for gel polymer electrolytes. Since tetra-PEG gel possesses good mechanical properties due to its regular structure, a small amount (less than 10 wt%) of tetra-gel is enough to give a self-standing gel polymer electrolyte with mechanical toughness and good

ionic conductivity [20–26]. These reported tetra-PEG gels were synthesized by reacting two types of tetra-arm PEG macromonomers. Our idea for tetra-PEG network is to use a reaction between low molecular weight tetrafunctional compound as a cross-linking agent and telechelic linear polymer. In this work we report preparation of tetra-PEG gels by the click reaction between pentaerythritol tetra(3-mercaptopropionate) and α, ω-dipropiolate PEGs with various molecular weight, and their application to gel polymer electrolytes (Fig. 1).

Materials and Methods

Materials

Pentaerythritol tetrakis(3-mercaptopropionate) (**1**) was purchased from Tokyo Chemical Industry Co., Ltd (Tokyo, Japan). Polyethylene glycols (MW = 300, 1000, 2000) were purchased from Nakalai Tesque (Kyoto, Japan). Propiolic acid was purchased from Kanto Chemical Co., Inc. (Tokyo, Japan). Propylene carbonate and lithium bis(trifluoromethanesulfonyl)imide (LiTFSI) were purchased from Kishida Chemical Co., Ltd (Osaka, Japan). All other reagents were obtained from commercial sources and used as received.

Preparation of α, ω-dipropiolate-terminated polyethylene glycol (MW = 300) (2a)

Propiolate esters of polyethylene glycol (PEG) were prepared according to the reported procedure [27]. Into a round-bottomed flask equipped with a Dean Stark apparatus were added PEG (MW = 300) (4.5 g, 15 mmol), propiolic acid (4.2 g, 60 mmol), p-toluenesulfonic acid monohydrate (0.7 g, 3.7 mmol), and toluene (50 mL). The reaction mixture was heated under reflux with azeotropic removal of water for 6 h. The rection mixture was cooled to room temperature and diluted with dichloromethane. The organic layer was washed with an aqueous potassium carbonate solution, dried over magnesium sulfate, and placed under reduced pressure to remove the solvent to obtain 3.7 g (62%) of PEG **2a** as a pale yellow waxy solid; ^{1}H NMR (400 MHz, $CDCl_3$, δ): 4.34 (t, *J* = 4.6 Hz), 3.74 (t, *J* = 4.6 Hz), 3.67–3.65, 2.98; IR (NaCl, cm^{-1}): 2875, 2115, 1714, 1110.

Preparation of α, ω-dipropiolate-terminated polyethylene glycol (MW = 1000) (2b)

PEG **2b** was prepared according to the same procedure for **2a** using PEG (MW = 1000) (10 g, 10 mmol), propiolic acid (2.8 g, 40 mmol), p-toluenesulfonic acid monohydrate (0.5 g, 2.6 mmol), and toluene (70 mL). PEG **2b** was obtained as a pale yellow solid (9.2 g, 83%); ^{1}H NMR (400 MHz, $CDCl_3$, δ): 4.30 (t, *J* = 4.8 Hz), 3.74 (t, *J* = 4.8 Hz), 3.66–3.64, 2.97; IR (NaCl, cm^{-1}): 2871, 2111, 1714, 1107.

Preparation of α, ω-dipropiolate-terminated polyethylene glycol (MW = 2000) (2c)

PEG **2c** was prepared according to the same procedure for **2a** using PEG (MW = 2000) (5.0 g, 2.5 mmol), propiolic acid (0.7 g, 10 mmol), p-toluenesulfonic acid monohydrate (0.12 g, 0.65 mmol), and toluene (50 mL). PEG **2c** was obtained as a pale yellow solid (4.2 g, 79%);^{1}H NMR (400 MHz, $CDCl_3$, δ): 4.35 (t, *J* = 4.8 Hz), 3.74 (t, *J* = 4.8 Hz), 3.66–3.64, 2.98; IR (NaCl, cm^{-1}): 2881, 2112, 1715, 1120.

Preparation of tetra-branched PEG gel for IR analysis

A mixture of tetrathiol **1** (93 mg, 0.19 mmol), PEG **2b** (0.42 g, 0.38 mmol), and propylene carbonate (PC) (0.5 g) was heated at 100°C for 8 h to obtain a yellow gel. The solvent captured in the polymer network was removed by Soxhlet extraction using ethanol as a solvent for 24 h. The residue in the extraction thimble was dried and analyzed by IR.

Preparation of gel polymer electrolyte

A given amount of tetrathiol **1** and propiolate-terminated PEG (**2a**, **2b** or **2c**) were dissolved in 1.0 M LiTFSI in PC. The resulting solution was put into a Teflon petri dish and heated at 100°C for 48 h. The obtained gel polymer electrolyte was peeled from the Teflon petri dish. All preparation procedures were carried out in an argon-filled glovebox.

Ionic conductivity measurement

Ionic conductivities of the gel polymer electrolytes were measured by the two-probe method after the samples had been fixed inside a Teflon O-ring spacer with known thickness and sandwiched between two stainless steel (SS) electrode disks acting as ion-blocking electrodes and set in a thermostat oven chamber. The measurements were carried out using a Solartron 1260 impedance/gain-phase analyzer over a frequency range of 10^6 to 1 Hz and a temperature range of 80 to 10°C with an amplitude of 10 mV. All samples were first kept at 80°C for at least 12 h and then measured during the cooling cycle. The measurements were carried out after keeping the samples at each temperature for 1 h to attain thermal equilibration. The data were processed by using an appropriate fitting program.

Instrumentation

^{1}H spectra were recorded at room temperature on a JEOL JNM-ECZ 400 nuclear magnetic resonance spectrometer. Samples were dissolved in deuterated chloroform ($CDCl_3$) and tetramethylsilane (TMS) was used as the internal standard. Differential scanning calorimetry (DSC) measurement was carried out on an SII EXSTER 6000 thermal analysis instrument DSC 6220 under nitrogen gas flow at a heating rate

of 10 K/min. Fourier-transform infrared (FTIR) spectra were recorded on a Thermo Scientific NICOLET iS50R FT-IR equipped with ZnSe ATR (DuraSample II). Raman spectra were measured using a laser Raman spectrophotometer (Lambda Vision, Micro-RAM 300) with a 532 nm (30 mW) laser at room temperature.

Results and discussion

Preparation of tetra-branched PEG network

Tetra-branched PEG gel was first prepared by the reaction between tetra-arm amine-functionalized PEG and tetra-arm N-hydroxysucsinimide-functionalized PEG in 2008 [17]. The synthesis method involved end-linking reaction of the two tetra-armed PEGs with reactive functional groups at their ends (Fig. 2a). Most reported tetra-gels have been synthesized using this star-polymer-based approach. Our idea for simpler preparation method for tetra-gels is to use a coupling reaction between a low-molecular-weight tetrafunctional cross-linking agent and a difunctional telechelic polymer (Fig. 2b). In this work we chose pentaerythritol tetra(3-mercaptopropionate) and α, ω-dipropiolate PEG as a tetrafunctional cross-linking agent and a telechelic difunctional polymer, respectively. Because thermal thiol-yne click reactions are robust methodologies to proceed nucleophilic conjugated addition of thiol to C ≡ C in excellent yields [28, 29].

As a preliminary experiment we carried out a thermal click reaction between tetrathiol **1** and propiolate-terminated PEG **1b** in PC. The obtained gel was washed with ethanol using Soxhlet extractor to remove PC captured in the polymer network. Figure 3 shows IR spectra of the product after removing the solvent together with those of tetrathiol **1** and propiolate-terminated PEG **2b**. Tetrathiol **1** and polymer **2b** show characteristic peaks at 2549 cm^{-1} (SH groups) and 2111 cm^{-1} (C ≡ C groups), respectively. These two peaks disappeared completely after reaction, indicating that thiol-yne click reaction between **1** and **2b** proceeded quantitatively to give a tetra-branched network structure.

Preparation of gel polymer electrolyte based on tetra-branched PEG network

Gel polymer electrolytes were prepared by carrying out thermal thiol-yne click reaction between **1** and **2** in the presence of 1.0 M LiTFSI in PC. Composition of the gel polymer electrolytes are summarized in Table 1. The glass transition temperatures (T_gs) determined by DSC measurement are also summarized in Table 1.

Table 1
Composition and T_g of Gel Polymer Electrolytes

entry	1, mg (mmol)	PEG, g (mmol)	1.0 M LiTFSI in PC, g	Polymer Content, wt%	T_g, °C
1	39 (0.08)	**2b**, 0.18 (0.16)	0.5	30	−75.0
2	59 (0.12)	**2b**, 0.26 (0.24)	0.5	40	−60.4
3	93 (0.19)	**2b**, 0.42 (0.38)	0.5	50	−41.0
4	137 (0.28)	**2b**, 0.62 (0.56)	0.5	60	−38.3
5	190 (0.39)	**2a**, 0.31 (0.78)	0.5	50	−32.6
6	54 (0.11)	**2c**, 0.46 (0.22)	0.5	50	−48.1

Effect of polymer content in the polymer gel electrolyte

We prepared four gel polymer electrolytes with different polymer content (entries 1–4 in Table 1). Figure 4 shows the effect of polymer content in the gel polymer electrolytes on the ionic conductivity. In the low-temperature region, ionic conductivity increased as the polymer content increased. If ionic conduction occurred entirely within the liquid electrolyte, ionic conductivity would be expected to decrease as the polymer content increases because the liquid electrolyte content would decrease; however, in fact, ionic conductivity increased with increase of polymer content. This suggests that the PEG chains forming the network structure are also involved in ionic conduction. On the contrary, ionic conductivity decreased with increase of polymer content in high-temperature region although the difference was not significant. This suggests that the ion conduction mechanism may differ between high-temperature and low-temperature regions.

Effect of PEG length on ionic conductivity

Then, we examined the effect of PEG length in the network structure on ionic conductivity. We prepared three gel polymer electrolytes (polymer content, 50%) having different molecular weight of PEG (entries 3,5 and 6 in Table 1). Figure 5 shows the effect of the chain length of the PEG segments composed of the network on ionic conductivity. It was found that the chain length of the PEG segments had a significant effect on ionic conductivity. Ionic conductivity increased with the chain length of PEG constituting network. Gel polymer electrolytes based on shortest PEG chain (**2a**) with molecular weight of 300 exhibited very low ionic conductivity. As shown in Table 1, T_g value of the gel polymer electrolyte increased as the molecular weight of the PEG decreased. The effect of PEG chain length on ionic conductivity could be explained by the mobility of the PEG chains involved in ionic conduction. In other words, these experimental results suggest that ionic conduction occurs not only in the liquid electrolyte but also along the PEG chains of the matrix.

Raman spectroscopy

Raman spectroscopy is an effective tool to obtain useful information about the coordination state of lithium ions. In Raman spectra, a peak originating from the S-N-S stretching vibration of the TFSI anion is observed in the range between 730 and 760 cm^{-1}, and it is known that the position of this peak changes depending on the coordination state of the TFSI anion [30–32]. When the lithium ions are directly bonded to the TFSI anions to form contact ion pairs and ionic aggregates, the peak corresponding to the S-N-S stretching vibration is observed between 745 and 755 cm^{-1}. On the other hand, when the lithium ion is not coordinated to the TFSI anion, this peak shifts to the lower wavenumber, appearing between 739 and 742 cm^{-1} [31, 32]. Figure 6 shows the Raman spectra of electrolyte solution (1.0 M LiTFSI in PC) and gel polymer electrolytes with two different network polymer content of 30 and 60wt%. The peak from the S-N-S stretching vibration of the electrolyte solution was observed at 749 cm^{-1}, corresponding formation of contact ion pairs between lithium cations and TFSI anions. On the other hand, this peak shifted towards lower wavenumber for gel polymer electrolytes. Furthermore, the higher the content of network polymer in the gel polymer electrolyte, the more pronounced the shift toward lower wavenumbers. These results suggest that lithium cations coordinated with the ether oxygen atoms in the PEG polymer chain in the network, leading to the formation of free TFSA anions. In other words, this suggests that as the polymer content in the gel polymer electrolyte increases, the contribution of ion conduction by the PEG chains becomes greater.

Conclusions

We prepared gel polymer electrolytes based on a network polymer with tetra-branched structure composed of ion-conductive PEG. Thermal thiol-yne click reactions were carried out in the presence of an electrolyte solution between a tetrathiol cross-linking agent and propiolate-terminated PEG with different molecular weight. The thiol-yne click reaction proceeded quantitatively to give gel polymer electrolytes. As the network polymer content in the gel polymer electrolyte increased, ionic conductivity improved in the low-temperature region. Furthermore, ionic conductivity decreased significantly as the chains of the telechelic polyethylene glycol became shorter. These experimental results suggest that ionic conduction occurs not only in solution state but also along the PEG chains forming the network. Raman spectroscopy also suggested that lithium cations are coordinated to the PEG polymer chains from the solution. By using a network polymer with a regular structure, known as a tetra-gel, as the matrix for the gel polymer electrolyte, we were able to gain interesting insights into the coordination structure of lithium ions.

Declarations

Supplementary Information The online version contains supplementary material available at ....

Author contributions Tomoya Enoki: writing–original draft, methodology. Ryodai Kagami, formal analysis and data curation. Takahiro Uno: writing–review and editing. Nanami Sasaki: data curation. Nobuyuki

Imanishi: project administration. Masataka Kubo: writing–review and editing and supervision.

Data availability

No datasets were generated or analyzed during the current study.

Competing interests The authors declare no competing interests.

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Figures

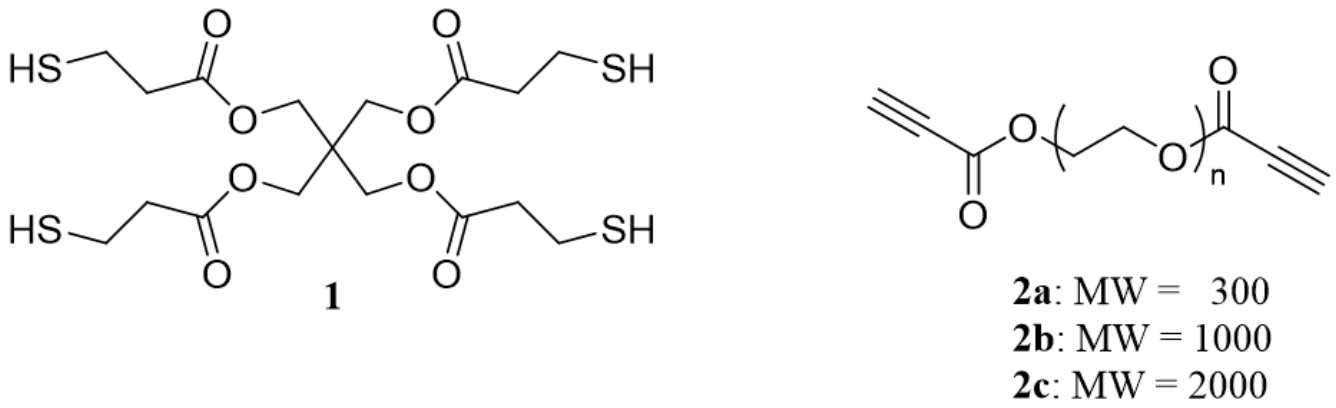


Figure 1

Chemical structures of the compounds used in this study.

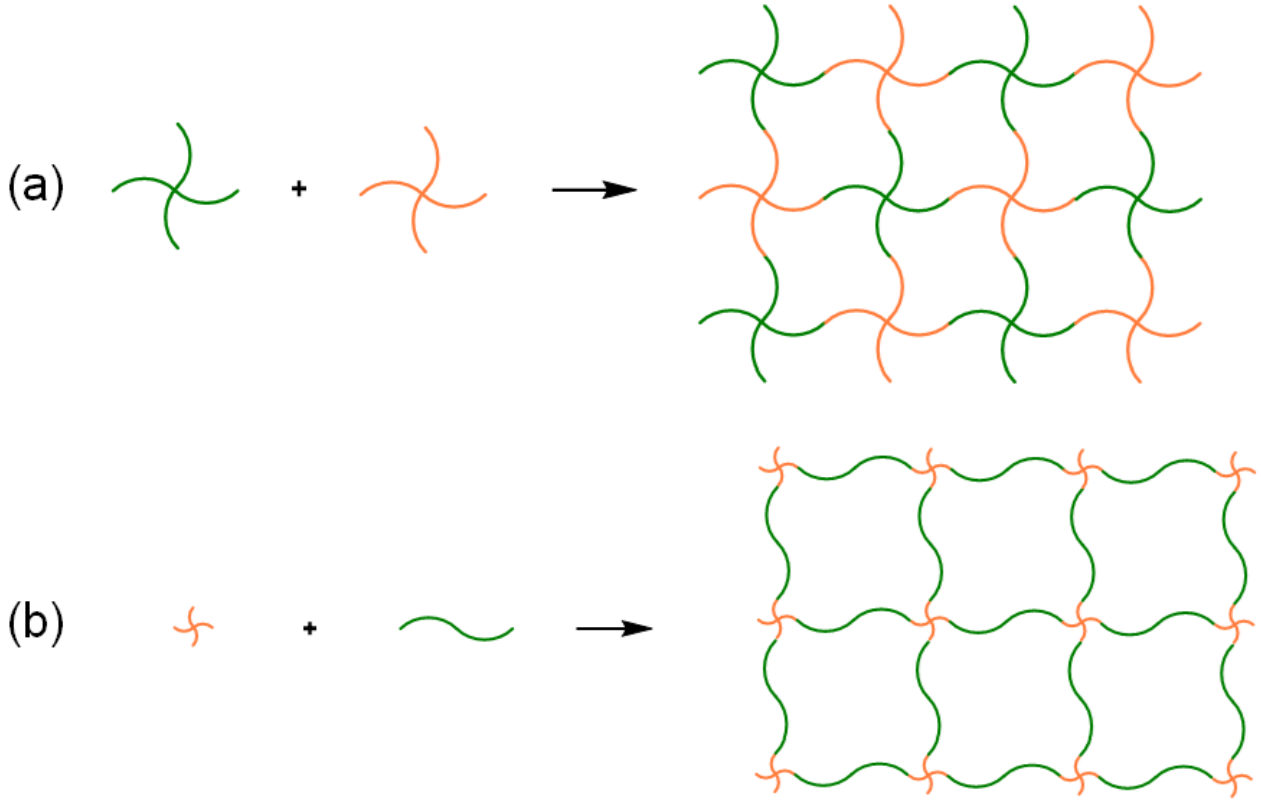


Figure 2

Preparation of tetra-branched network by (a) star-polymer-based approach and (b) tetrafunctional cross-linking agent approach.

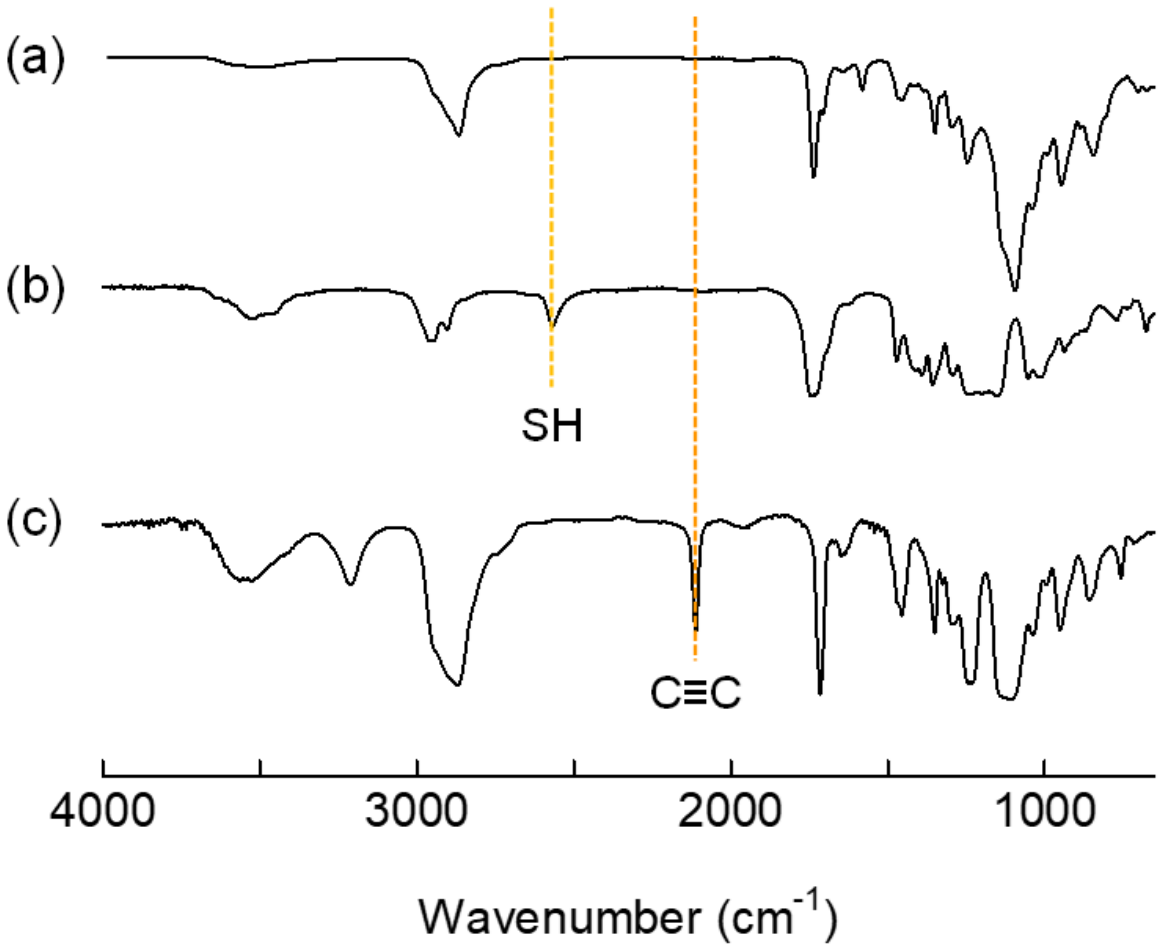


Figure 3

IR spectra of (a) tetra-branched PEG network, (b) tetrathiol **1**, and (c) PEG **2b**.

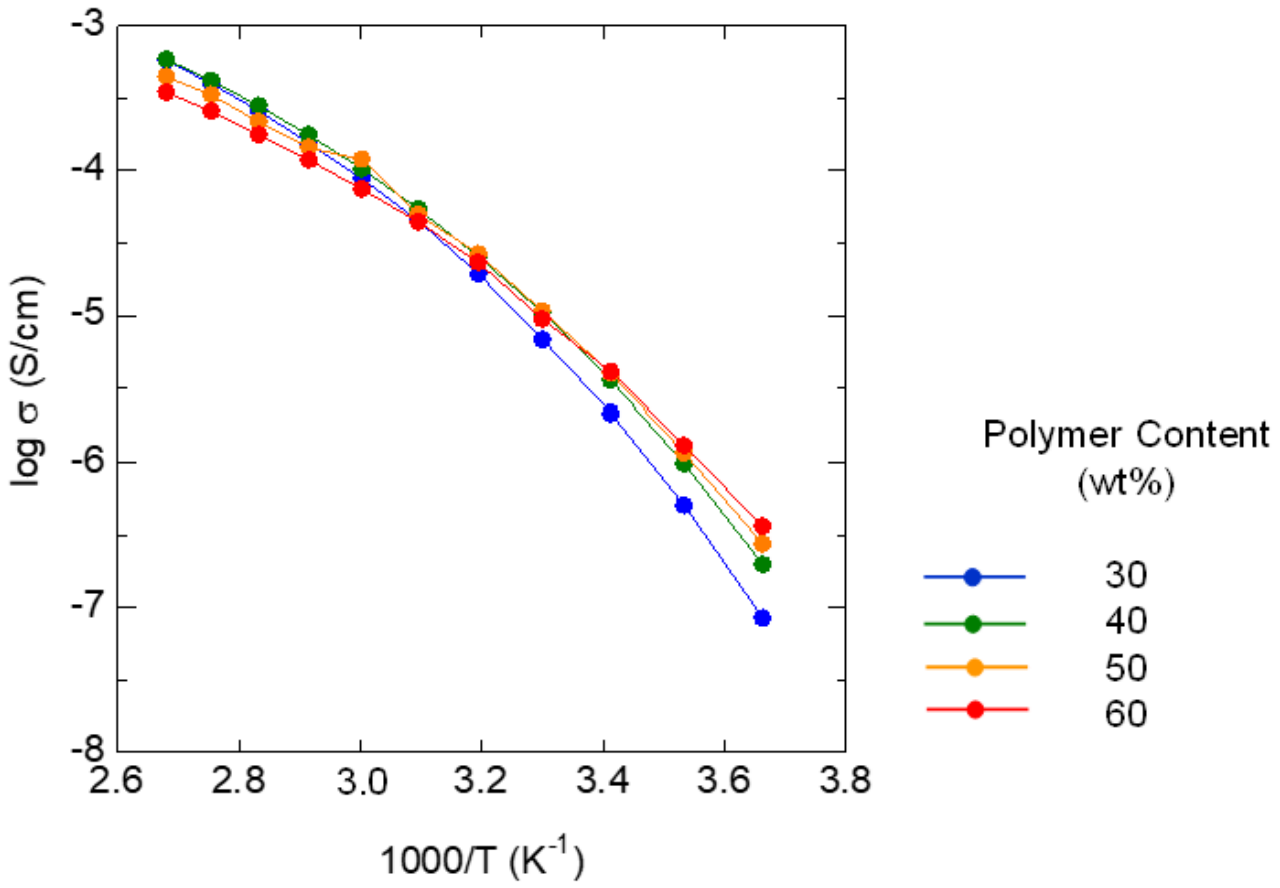


Figure 4

Effect of Polymer Content in Gel Polymer Electrolyte on Ionic Conductivity.

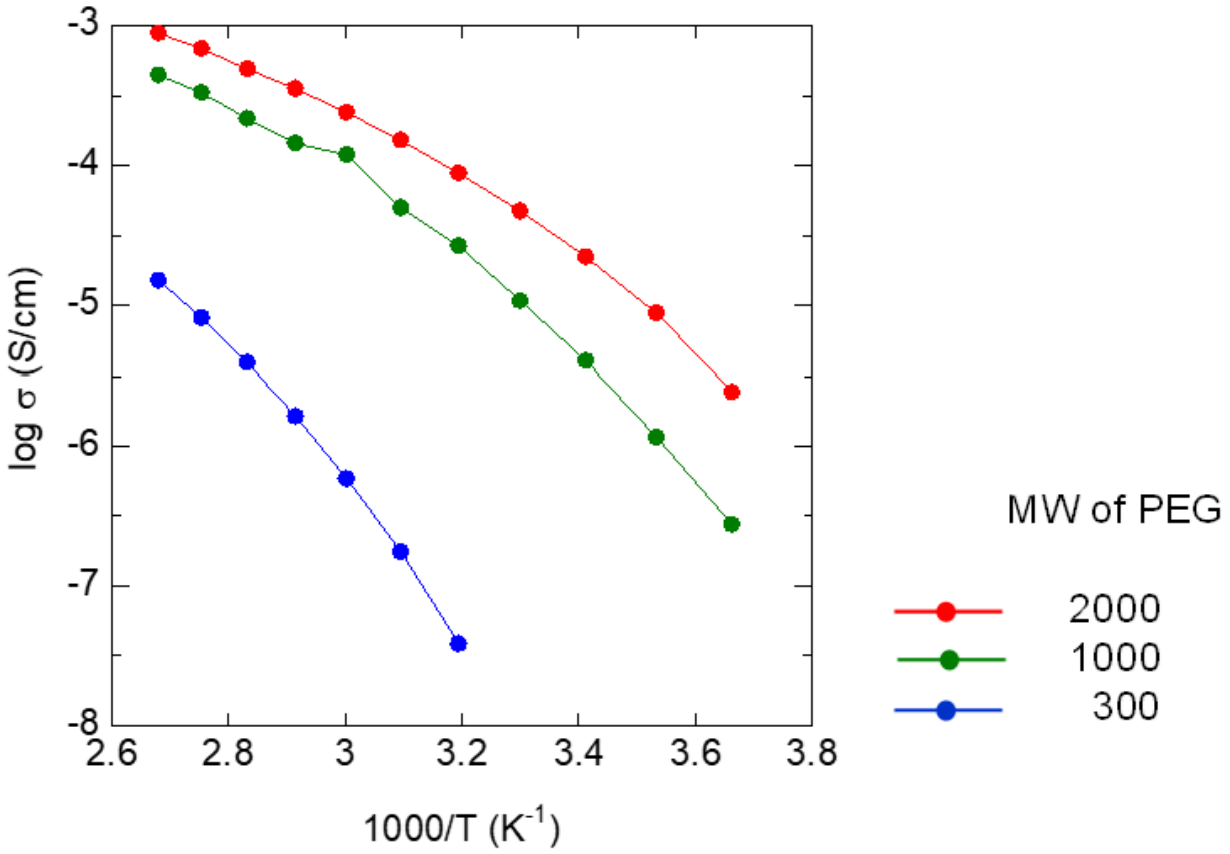


Figure 5

Effect of PEG Chain Length on Ionic Conductivity.

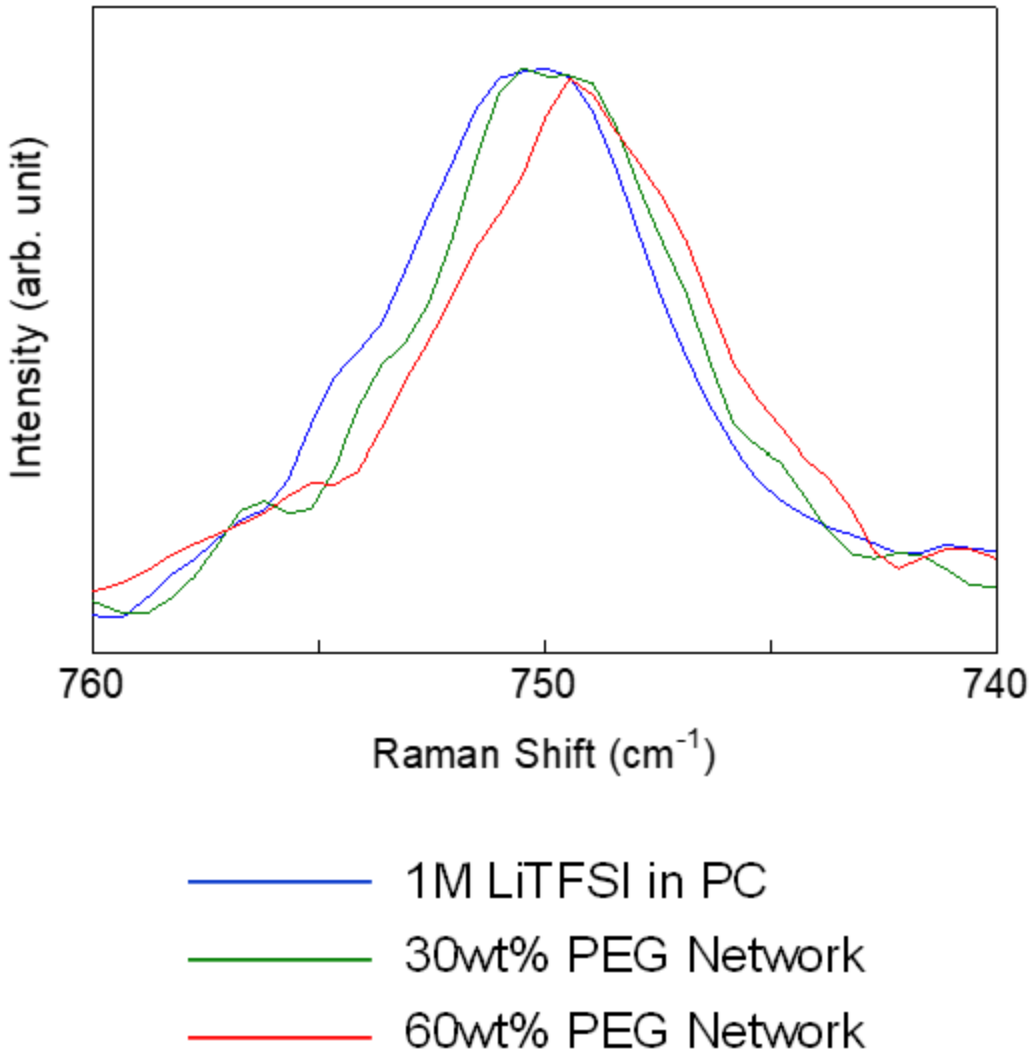


Figure 6

Raman spectra for 1M LiTFSI in PC (blue) and polymer gel electrolytes of 30wt% PEG network (green) and 60wt% PEG network (red).

Supplementary Files

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