Synthesis, Characterization, and Ionic Conductivity of Novel Crosslinked Polymer Electrolytes for Li-Ion Batteries

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ABSTRACT: In this work, we communicate on the synthesis, characterization, and ionic conductivity of a series of novel crosslinked inorganic–organic gel polymer electrolytes. These crosslinked polymers were prepared by the modification of poly(vinyl alcohol) (PVA) with poly(ethylene glycol) (PEG) in the presence of borane-tetrahydrofuran (BH_3/THF) complex. During crosslinking, the polymer was doped with lithium trifluoromethane-sulfonate (CF_3SO_3Li) salt. Molecular weight of both PVA and PEG was varied, and the concentration of Li-salt was arranged for the ratio of lithium atoms to ether oxygen atoms. Boron-containing polymer electrolytes were produced and abbreviated as PVA1PEGX-Y and PVA2PEGX-Y. The reaction of PEG with PVA and the interaction of Li$^+$ ions with EO units were confirmed by FTIR. Thermal stability of these materials was measured with thermogravimetric analysis, and thermal behaviors were measured by differential scanning calorimetry. The ionic conductivity of these novel polymer electrolytes was studied by dielectric-impedance spectroscopy. Li-ion conductivity of these crosslinked polymer electrolytes depends on the length of the side units as well as the doping ratio. The maximum ionic conductivity was measured as 10^{-4} S cm^{-1} at room temperature. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 1193–1199, 2012

Key words: poly(vinyl alcohol); poly(ethylene glycol); borane-tetrahydrofuran; polymer electrolytes; Li-ion conductivity

INTRODUCTION

Solid polymer electrolytes (SPEs) have been extensively studied for application in many portable electronic devices, such as telecommunication equipment, smart credit cards, laptop computers, and cordless tools. It is expected that these materials should have high ionic conductivity, electrochemical stability, and good mechanical properties for application.[1]

In general, SPEs can be prepared by dissolving salt (LiX) in a suitable polymer matrix where there is an interaction between functional units of the host polymer and guest molecules.[2–4] Polyethylene oxide (PEO)-based electrolytes have been the most extensively studied systems.[5,6] Wright and Armand reported the earliest works on the ion conducting PEO/alkali metal salts.[7,8]

Polymer electrolytes based on poly(ethyleneoxide) and lithium salts were found to exhibit relatively low conductivity, and introduction of small molecule plasticizers (such as propylene carbonate) to such polymer-salt systems was found to improve the con-

ductivity of the materials.[9,10] Depending on the salt concentration, PEO-based polymer electrolytes have semicrystalline morphology, which may negatively affect the ionic conductivity especially at lower temperatures. In dry state, these dry polymer electrolyte systems (PEO-LiX) offer lower ionic conductivity of the order of $10^{-8} - 10^{-7}$ S cm^{-1} at ambient temperature.

To produce new polymer electrolytes with higher ion conductivity and good mechanical property, gel electrolytes such as PEO,[11,12] polyacrylonitrile,[13] poly(methylmethacrylate),[14] and poly(vinylidenflouride)[15] have been investigated.

Over the years, forming borate and boronate compounds using boron compounds with different alcohols has been studied.[16] Metha and Fujinami[17,18] have produced polymers by the condensation reaction of boron trioxide and poly(ethylene glycol)s (PEGs). Recently, several studies on electrolyte based on boron chemistry have been studied, indicating an increasing interest for electrolytes containing Lewis acids.[19–23]

In a previous work, we have presented electrolyte systems based on boronate ester polymers and LiCF$_3$SO$_3$ for Li-ion batteries.[24] A novel comb-branched boron-containing copolymers were prepared by using borane-tetrahydrofuran (BH_3/THF) to link different poly(vinyl alcohol)s PVAs and PEGMEs via the formation of boronate esters. Maximum ionic conductivity was measured for the
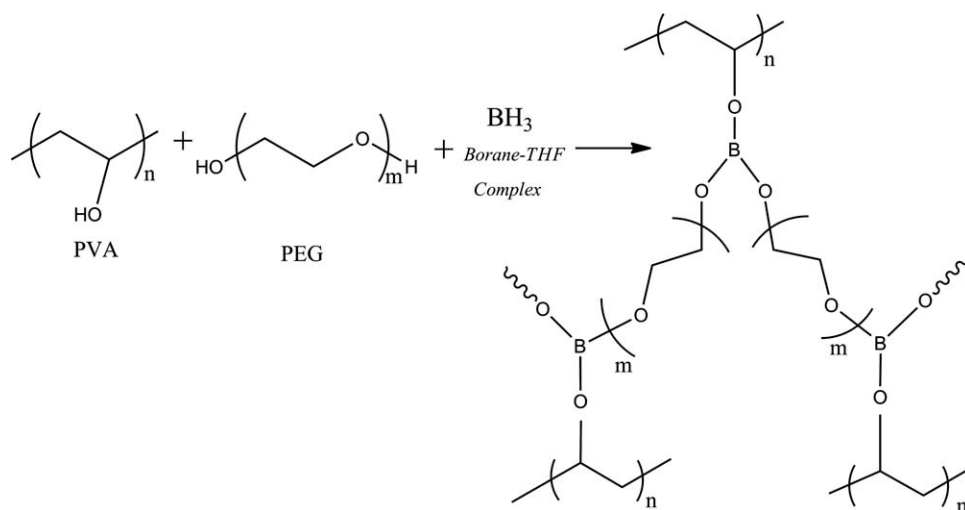
Figure 1 Synthesis scheme of PVAPEGX crosslinked polymers.

polymer electrolyte, PVA2PEGME550-25 as 8.9×10^{-5} S cm^{-1} at 20°C and 3.9×10^{-3} S cm^{-1} at 100°C.

In the present study, PVA was modified with PEG using BH$_3$/THF with different molecular weights of PVA and PEG to produce boron-containing crosslinked polymers. Meanwhile, polymer electrolytes were produced by doping with the CF$_3$SO$_3$Li according to EO/Li ratio. The crosslinked polymer electrolytes were characterized via FTIR, TG, and differential scanning calorimetry (DSC). Li-ion conducting properties of the materials were investigated by dielectric-impedance analyzer, and the results are discussed.

EXPERIMENTAL

Materials

PVA (degree of hydrolization $\geq$ 98%, $M_w = 72,000$ and 145,000 g/mol) was provided from Merck, BH$_3$/THF (1.0M solution in THF) and PEG (average $M_n = 200$, 600, and 1000 g/mol) were purchased from Aldrich. Dimethylsulfoxide (DMSO, analytical reagent) was obtained from Merck. CF$_3$SO$_3$Li 97% was received from Alfa Aesar and stored in the glove box.

Synthesis of the crosslinked polymer electrolytes

The crosslinked polymers were prepared by using BH$_3$/THF to link different PVAs and PEGs via the formation of boronate esters (Fig. 1). The general procedure is as follows: 2.3 mmol (0.1 g) of PVA was taken in a flask and dissolved in the solvent DMSO. To this, 2.3 mmol of PEG (ex: 0.46 g PEG200) admixed and the solution were stirred under nitrogen for 1 h at a temperature around 80°C. Then, 2.3 mmol of BH$_3$/THF was added slowly to the above mixed solution while increasing the temperature from 80 to 100°C and then continually stirring for 24

h under nitrogen atmosphere. Because the crosslinked polymers are not soluble in common organic solvents, lithium trifluoromethane-sulfonate (CF$_3$SO$_3$Li, 97%) was added into the reaction medium. The amounts of crosslinked polymer and salts were adjusted for the different levels of salt concentration corresponding to the ratios [EO] : [Li] = 10, 20, and 30. The synthesized PVA1PEGX-Y and PVA2PEGX-Y were precipitated by using diethyl ether and washed several times with excess diethyl ether to remove unreacted PEGs. Then, polymer electrolytes were dried in a vacuum oven at 80°C for 7 days to remove residual solvent.

The synthesis and the handling of the polymer electrolytes were made under protective atmosphere of nitrogen, and, finally, the highly transparent, light yellow, and sticky polymer was stored in a glove box under nitrogen atmosphere (moisture content below 1 ppm).

Characterizations

Before Fourier transform infrared (FTIR) spectra measurements, samples were dried under vacuum and stored in a glove box. The IR spectra (4000–400 cm^{-1} and resolution 4 cm^{-1}) were recorded at room temperature using a Bruker α-P in ATR system coupled to a computer.

Thermal stabilities of the obtained polymer electrolytes were examined by thermogravimetry analysis (TGA) with a Perkin–Elmer Pyris 1. The samples ($\sim$ 10 mg) were heated from room temperature to 750°C under nitrogen atmosphere at a heating rate of 10°C min^{-1}.

The thermal behavior of the obtained polymer electrolyte samples was investigated by differential scanning calorimetry (DSC) using a Perkin–Elmer Pyris 1 instrument. The samples were first cooled
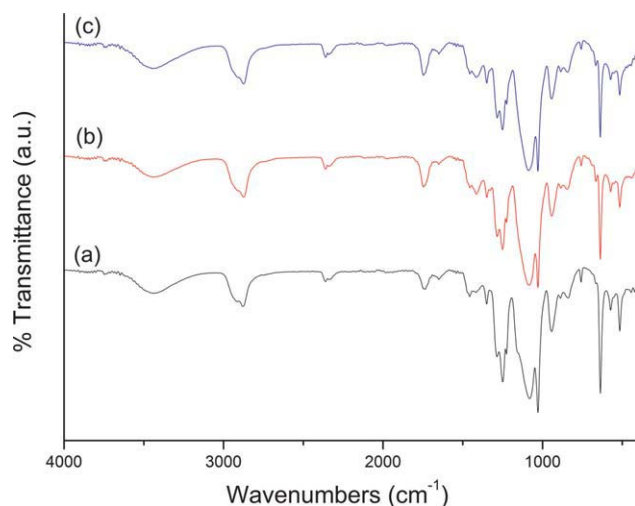
Figure 2 Comparison of FTIR spectra of PVA2PEG600-Y with $Y = 10$ (a), 20 (b), and 30 (c). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

from room temperature to $-40°C$, kept isothermally for 10 min, and then heated up to $180°C$. After, the samples were cooled to $-40°C$ and then finally heated to $200°C$. All thermograms were recorded at a rate of $10°C$ under nitrogen flow. All DSC experiments were done in duplicate, and the thermograms shown refer to the final heating.

The alternating current (ac) conductivities of the copolymer samples were measured using a Novocontrol dielectric-impedance analyzer in the frequency range from 0.1 Hz to 3 MHz as a function of temperature. The samples with a diameter of 10 mm and a thickness of ~ 0.5 mm were sandwiched between two gold-coated electrodes, and their conductivities were measured at 10 K intervals under a dry nitrogen atmosphere.

RESULTS AND DISCUSSION

FTIR study

The FTIR spectra of PVA have been reported in a previous work. The characteristic absorptions of the pure PVA are located at 3300, 2934, and 1100 cm^{-1}, corresponding to ν O—H, ν C—H, and ν—C—C—O—, respectively. The peak at 1145 cm^{-1} (C—O, $\nu = 1090$–1150 cm^{-1}) can be attributed to C—O stretching.[24]

The FTIR spectra of the polymer electrolytes PVA2PEG600-Y are shown Figure 2. In all the samples, there is a peak at 3400 cm^{-1}, which corresponds to the absorption of hydroxyl units. Comparing to pure PVA, the intensity of this peak is lower, which illustrates high conversion of the hydroxyl groups into boronate esters.[22] In addition, polymer electrolytes show strong bands at 2910–2870 cm^{-1} due to C—H stretching and weak peaks at 667 cm^{-1}

due to B—O units.[25,26] The absorption bands at $\sim$ 1029 and $\sim$ 1350 were assigned to the vibration of B—O—C bond and to the antisymmetric stretching vibration of B—O, respectively,[23] which confirms the reaction of BH$_3$ with hydroxyl units of both PVAs and PEGs. After increasing the doping ratio, there is a broadening of the C—O—C peaks indicating a higher degree of interactions by co-ordination between Li$^+$ and EO.[27] The peak at 640 cm^{-1} belongs to CF$_3$SO$_3$Li. The peak at 1030 cm^{-1} can be attributed to stretching vibration of SO$_3$ unit of the dopant and increases with the dopant ratio. Consequently, the FTIR spectra reveal that crosslinked polymers were successfully synthesized.

Thermal properties

The thermal stability is an important property for electrolytes composed of polymers and lithium salts during application in rechargeable lithium-ion polymer batteries. The thermal stability was evaluated by TGA for the neat polymer and the electrolytes with various Li-salt for further application in technological devices.

Table I and Figure 3 show the TGA results of the pure PVA ($M_w = 145,000$ g/mol) and the polymer electrolytes PVA2PEG600-Y. As seen in the figure, the onset of degradation temperature of PVA2 is $\sim 300°C$. After LiCF$_3$SO$_3$ is introduced into the polymer, the degradation temperature (T_d) clearly shifts to $200°C$, and decomposition occurs in two steps. This behavior may be attributed to the weakness of the C—O bond caused by a reduction in electronic density due to coordination between the Li ions and

TABLE I
The Composition, T_d, and Ionic Conductivity of PVAPEGX-Y Samples

Sample PVAPEGX-Y	Y (EO : Li mol ratio)	T_d (°C)	Max. ion conductivity (S/cm) at 100°C
PVA1PEG200–10	10 : 1	200	3.7×10^{-4}
PVA1PEG200–20	20 : 1	200	–
PVA1PEG200–30	30 : 1	200	8.9×10^{-5}
PVA1PEG600–10	10 : 1	200	5.2×10^{-4}
PVA1PEG600–20	20 : 1	200	3.1×10^{-3}
PVA1PEG600–30	30 : 1	200	6.4×10^{-4}
PVA1PEG1000–10	10 : 1	250	1.6×10^{-3}
PVA1PEG1000–20	20 : 1	250	6.6×10^{-4}
PVA1PEG1000–30	30 : 1	250	–
PVA2PEG200–10	10 : 1	200	5.0×10^{-4}
PVA2PEG200–20	20 : 1	200	–
PVA2PEG200–30	30 : 1	200	2.3×10^{-4}
PVA2PEG600–10	10 : 1	200	1.1×10^{-3}
PVA2PEG600–20	20 : 1	200	1.1×10^{-3}
PVA2PEG600–30	30 : 1	200	7.9×10^{-4}
PVA2PEG1000–10	10 : 1	300	1.5×10^{-3}
PVA2PEG1000–20	20 : 1	250	0.3×10^{-3}
PVA2PEG1000–30	30 : 1	300	8.3×10^{-4}
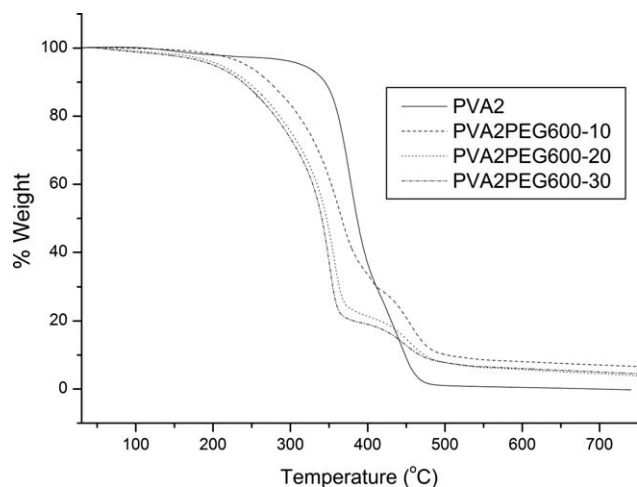
Figure 3 Comparison of the weight loss of PVA2 and PVA2PEG600-Y with Y = 10, 20, and 30.

the ether oxygen of the polyether.[28] The decomposition temperature is higher for the samples containing PEG with molecular weight of 1000. All the samples can be said to be thermally stable up to 200°C, which is far higher than the operating temperature of lithium ion batteries.

The thermal properties of the polymer electrolytes with various Li-salt were investigated using DSC as well. The melting temperatures of PVA1 and PVA2 are 200°C. In addition, PEG200, PEG600, and PEG1000 have melting points at around −50, 20, and 34°C, respectively.

Figure 4(a,b) shows the melt endotherms of semi-crystalline crosslinked polymer and the doped samples. For PVA2PEG600-Y series, there is no definite T_g or melting point. The PVA2PEG600-Y electrolyte series and the PVA1PEG1000-10 electrolyte were fully amorphous, while the PVA1PEG1000-20 and PVA1PEG1000-30 electrolytes displayed melting transitions at 29°C and 33°C, respectively. A cold crystallization was observed at −2°C and −24°C in the thermogram of the electrolytes PVA1PEG1000-10 and PVA1PEG1000-30 before the melting peak. Clearly, the material showed more crystalline behavior with increasing PEG molecular weight, and the addition of more lithium salts leads to an amorphous character.

Ionic conductivity

Lithium ion conductivity of crosslinked polymer electrolytes PVA1PEGX-Y and PVA2PEGX-Y with different interchain PEG spacers units was measured by the ac impedance analyzer. The polymer electrolyte films were cut into disks of 10 mm in diameter sandwiched between Pt electrodes and subjected to the impedance analyzer in a completely water-free environment. The ion dynamics in the polymer elec-

trolytes and the frequency dependence of the conductivity at various temperatures have been analyzed for all samples.

The frequency-dependent ac conductivity curves show a general trend for all samples and at all temperatures in the log–log plot. Figure 5 shows the frequency dependence of the ac conductivity (σ_{ac}) at various temperatures for PVA1PEG600-20 [Fig. 5(a)] and PVA2PEG1000-20 [Fig. 5(b)]. It can be observed that the ac conductivity versus frequency for various temperatures exhibits almost linear increasing at low frequencies, which are assigned to the polarization of the blocking Pt electrodes, while well-developed frequency independent plateaus are observed at higher frequencies.

The switchover from the frequency independent to frequency dependent region marks the onset of conductivity relaxation that gradually shifts to higher frequencies as temperature increases Figure 5(a,b). The frequency-independent conductivity is, in general, identified with the dc conductivity (σ_{dc}). The dc conductivities were derived from the plateaus by linear fitting of the frequency-independent region of the σ_{ac}. A similar kind of frequency dispersion was observed for all other compositions as well.

Figure 6(a,b) shows the obtained σ_{dc} variation of the PVA1PEGX-Y and PVA2PEGX-Y as a function of temperature. It is obvious from the plots that the temperature dependence of ion conductivity did not follow the Arrhenius behavior. The curved lines indicates that the ion conduction in these crosslinked polymer electrolytes can be explained by the concept of the free volume,[29,30] which is expressed by the Vogel–Tamman–Fulcher (VTF) equation (eq. 1). To have better insight into the temperature dependence of σ_{dc}, the conductivity data have been fitted to the equation.

$$\sigma = \sigma_0 \exp[-B/k(T - T_0)] \tag{1}$$

where σ_0 is the prefactor, T is the absolute temperature, k is the Boltzmann constant, B is an apparent activation energy, and T_0 is the equilibrium glass transition temperature at which the "free" volume disappears or at which configuration free entropy becomes zero (i.e., molecular motions cease).

The polymer electrolytes have crosslinked EO units, and, as a result, the crosslinked EO spacers have a special structure, which can lead to the lithium ion conductivity. A comparison between the conductivities of the electrolytes with different chain lengths is shown in the Figure 6(a,b). It indicates that at a given temperature, the conductivities of the polymer electrolytes increase with the length of PEG incorporated in the polymer main chain, which acts as a plasticizer mainly to the matrix polymer.[31] It is clearly seen from the conductivity isotherms that the
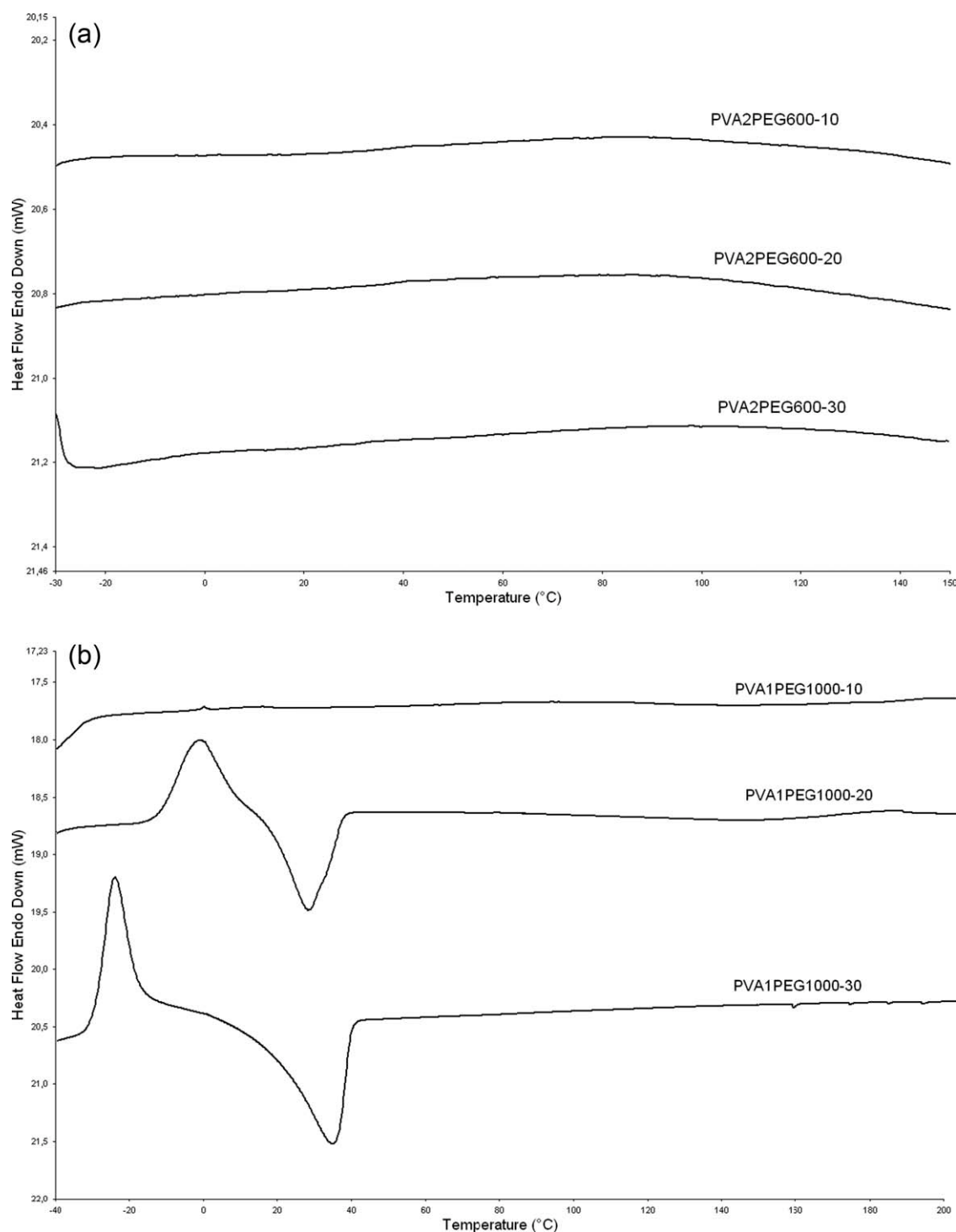
Figure 4 (a) DSC curves of PVA2PEG600-Y (Y = 10, 20, and 30) polymer electrolytes. (b) DSC curves of PVA1PEG1000-Y (Y = 10, 20, and 30) polymer electrolytes.

similar comprising low molecular weight EO spacers has lower conductivity, due to crosslinking (they have no melting temperatures), but PVAPEG1000 has high conductivity due to melting transition of PEG units in the samples (even they are cross-linked). It is generally considered in polymer electrolytes that ionic conduction occurs by segmental motion of polymer chains in amorphous region.[32–34]

T_g is correlated with flexibility of EO chains, that is, T_g is correlated with segmental motion of EO chains. Accordingly, mobility of carrier ions in polymer electrolytes is correlated with T_g.[35–37] The temperature range for the ionic conductivities of the present study [Fig. 6(a,b)] is above T_g's. In other words, the ionic conductivities of polymer electrolytes whose phase is almost amorphous were measured in the
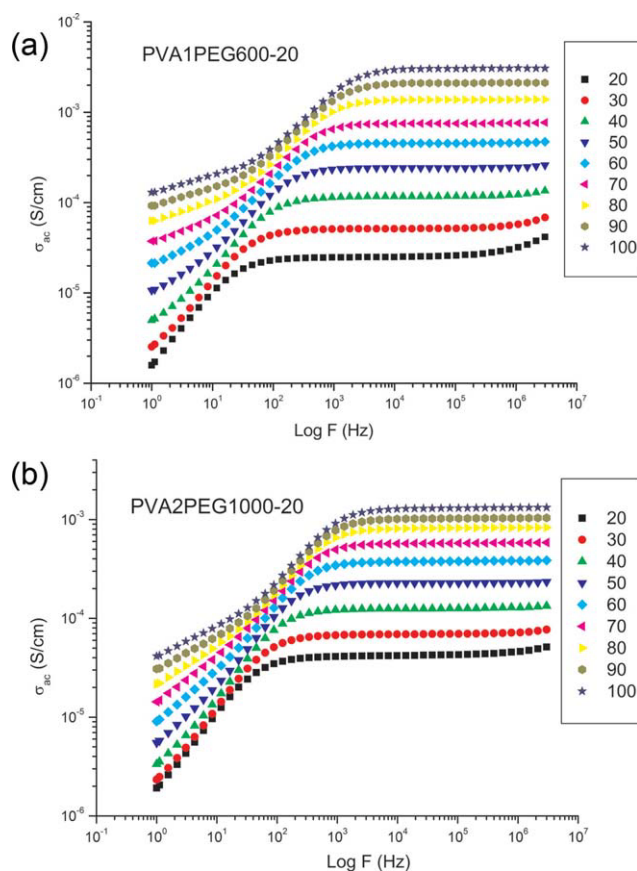
Figure 5 (a) Alternating current (ac) conductivity versus frequency of anhydrous PVA1PEG600-20 at various temperatures. (b) ac conductivity versus frequency of anhydrous PVA2PEG1000-20 at various temperatures. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

present study. Maximum ionic conductivity was measured for the polymer electrolyte, PVA1PEG600-20 as 3.1×10^{-3} S cm^{-1} at 100°C. The conductivity increased gradually with increasing salt concentration and reached a maximum for EO/Li = 20. This maximum ion conductivity value may be explained by the increase in the concentration of charge carrier when salt is added to the polymer until a maximum value is reached, after which the ionic conductivity increases.[38] The decrease in ionic conductivity with salt concentration can be attributed to the formation of ionic clusters, reducing the number and the overall mobility of the ions.[39] A further reason for the high conductivities of the electrolytes having low salt concentrations is the relatively low level of ion-polymer interaction. Indeed, it has been proposed that one Li$^+$ is coordinated by four ethylene oxide oxygens in PEO, leading to "transient crosslinks," which reduce the segmental mobility of the polymer.[40]

Kato et al.[41] showed that the high degree of dissociation of CF$_3$SO$_3$Li might be induced by the Lewis acidic PEG-borate ester. In other words, the PEG-

borate ester with Lewis acidity interacts with the anions, leading to improved dissociation of Li-salts and transport number of lithium ions. Therefore, the interaction between the PEG-borate ester and the CF$_3$SO$_3^-$ should be higher, which can be related to order of "hardness" of the Lewis basic anions, CF$_3$SO$_3^-$ > ClO$_4^-$ > N(CF$_3$SO$_2$)$_2^-$, estimated by *ab initio* calculations.[42] As a consequence, the PEG-borate ester in polymer electrolytes improves ionic conductivity as a plasticizer and Lewis acid. Additionally, Lewis acidity of the PEG-borate ester is particularly effective for the enhancement of the degree of Li-salt dissociation on CF$_3$SO$_3$Li.[41] PEG-boronate ester polymers were studied by Jannasch for electrochromics, and it was generally found that, at a given temperature and salt concentration, the conductivity of the electrolytes increased with the length of the side chain segment.[25] Poly(vinyl borate) (PVBO) and lithium derivative of PVBO, which was prepared by homogeneous esterification of PVA with boric acid,
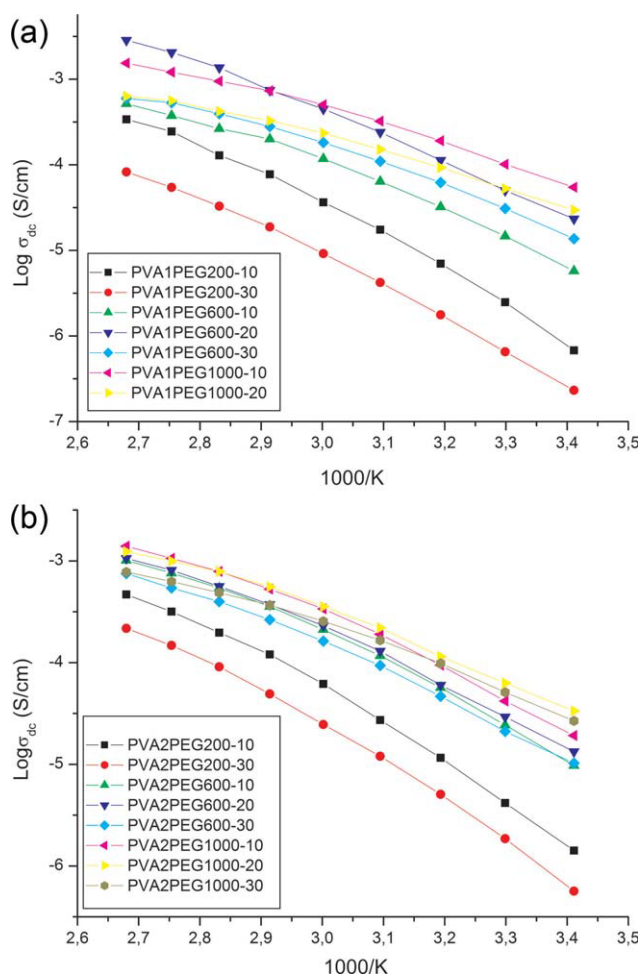
Figure 6 (a) Temperature dependence of the DC conductivities of PVA1PEGX-Y based materials. (b) Temperature dependence of the DC conductivities of PVA2PEGX-Y-based materials. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

was studied in literature. It was showed that there is more than hundred time increase of conductivity in the case of PVBO-Li, and so it behaves as moderately good polyelectrolyte and become comparable with literature.[26]

CONCLUSIONS

In the present work, a novel crosslinked boron-containing polymer with different molecular weights PVA and PEG was produced. Then, polymer electrolytes were synthesized after doping the host material with lithium salt at several molar ratios, that is, EO/Li. The EO units of the polymer are capable of forming donor: acceptor-type bonds by complexing with the lithium ion. These polymer electrolytes were characterized using FTIR, TGA, DSC, and dielectric-impedance spectroscopy. The samples are thermally stable up to 200°C. The conductivity studies illustrate that the ionic conductivity of these polymer electrolytes was found to be dependent on side-chain length as well as the Li^+ content. The polymer electrolyte PVA1PEG600-20 illustrated a satisfactory ionic conductivity of 3.05×10^{-3} S cm^{-1} at 100°C. The polymer electrolytes with higher Li^+ content show VTF behavior, indicating that the Li^+ ion motion is depended on the flexibility of the polymer side chain. These materials have high ion conductivity and can be suggested for application to lithium ion batteries.

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