

Preparation and Characterization of Low Environmental Humidity Sensitive Ionic Liquid Polymer Electrolytes

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ABSTRACT: Methyl 3-(3-(2-hydroxyethyl)imidazole-1-yl)propanoate chloride salt (IL-Cl), methyl 3-(3-(2-hydroxyethyl)imidazole-1-yl)propanoate bromate salt (IL-Br), and their derivatives modified by polyethylene glycol (PEG) through ester-exchange reaction (IL-PEGs) were synthesized. First, the properties of those materials, especially their conductivity, have been extensively studied. Second, using the IL-PEG with the highest conductivity as a plasticizer and electrolyte, a series of gel polymer electrolytes were successfully fabricated from polyurethane, poly-1,4-butylene adipate glycol 2000, and IL-PEGs by melting blends with different mass ratios in a Haake torque rheometer. The surface morphology, thermal properties, and the surface resistivity of gel polymer electrolytes were studied by scanning electron microscopy, thermogravimetric analysis, differential scanning calorimetry, and surface resistivity test, respectively. Scanning electron microscopy pictures showed that the surface of polymer electrolyte is smoother than that without added IL-PEGs. Thermogravimetric analysis results revealed that the polymer electrolytes will not decompose when the processing temperature is below 275°C. It was found that the surface resistivity of polymer electrolytes can be below $10^9 \Omega$, showing a good antistatic property, and it changes slightly as the relative humidity decreases from 40% to 0.1%, indicating that the material has low humidity sensitivity. This study is a new demonstration and development in ionic liquid based polymer electrolyte. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 40675.

KEYWORDS: ionic liquids; polyurethanes; thermal properties

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INTRODUCTION

Polyurethane (PU) is a kind of versatile polymer materials because of its many outstanding advantages and processability, such as toughness, abrasion resistance, wearability, mechanical flexibility, chemical resistance, and biocompatibility.¹ Consequently, it is extensively used in coating materials, bonding agents, elastic fibers, biomaterials, and artificial leather.^{2,3} However, its excessive electrical insulation and high surface resistivity easily lead to static nature on the surface of materials. And this static nature may cause a series of damages, such as dust deposition, difficulty in spinning, decline of electrical performance, and even cause fire or explosion. Therefore, PU materials need antistatic modification.

The main methods to improve antistatic ability are the addition of antistatic agents,⁴ addition of conductive filler,^{5,6} permanent antistatic polymer,⁷ surface modification,⁸ etc. Antistatic agents are usually composed of an amphiphilic surfactant which can migrate to the surface and enhance the surface conductivity by attracting a layer of water to endow materials the antistatic abil-

ity. However, the surface resistivity is strongly dependent on the environmental relative humidity (RH). And the drawbacks limit the application of surfactant.⁹ Another measure is the addition of conductive fillers, including carbon black and metal powders. This method can endow the material with high antistatic ability. However, the material filled with carbon black has poor mechanical properties and single color. The permanent antistatic polymer has high processing requirements even though it has good antistatic ability under low RH. So it is very important to develop a novel antistatic agent with good and persistent antistatic ability even under low humidity. Meanwhile, the amount of additive should be decreased. Ionic liquids (ILs) refer to liquids composed entirely of ions that are fluid around or below 100°C.¹⁰ ILs have attracted much attention because they have many unique properties, such as intrinsic ionic conductivity, thermal stability, nonvolatility, strong solubilizing ability, and chemical stability.^{11,12} Furthermore, the ILs can be designed to have some specific characters by adjusting the cation and anion species.^{13–15} The intrinsic ionic conductivity of ILs under low environment RH, together with the low melting point and

light color make ILs a good antistatic agent.^{16,17} Ferrari et al.¹⁸ have used the ILs as electrolytes for lithium batteries.

Gel polymer electrolytes are functional polymer material composed of polymer, plasticizer, and electrolyte salt.^{19–21} In gel polymer electrolytes, polymer is the frame support and plasticizer can decrease the glass-transition temperature (T_g) of polymer to improve the ion conductive ability of the material. And the electrolyte salts provide ion carriers. In IL-based polymer electrolytes, the ILs can be used as plasticizer.^{22–24} IL-based gel polymer electrolyte is transparent and mechanically stable, and so it can be applied in lithium batteries.^{25,26} However, IL-based gel polymer electrolytes is usually in fluid state, which limits its wide application. Therefore, some solid-state IL-based polymer electrolytes are studied.^{27,28} But these methods use solvent, and it is known that solvent is not friendly to environment. So we use the melt blend method to prepare the “dry,” solvent-free ILs-based gel polymer electrolytes in a Haake torque rheometer.

In this study, we synthesized two ILs namely IL-Br and IL-Cl successfully, and the two ILs were used to prepare IL-polyethylene glycol (PEG). The IL-PEG is a kind of novel antistatic agent, which has good antistatic ability under low RH. Then the IL-PEG was blended with PU matrix in a Haake torque rheometer with an electrically heated mixing head and two noninterchangeable rotors to prepare IL polymer electrolytes. The surface morphology, thermal decomposition behaviors, and the surface resistivity were evaluated. The results show that the IL polymer electrolytes have low humidity sensitive antistatic property.

EXPERIMENTAL

Materials

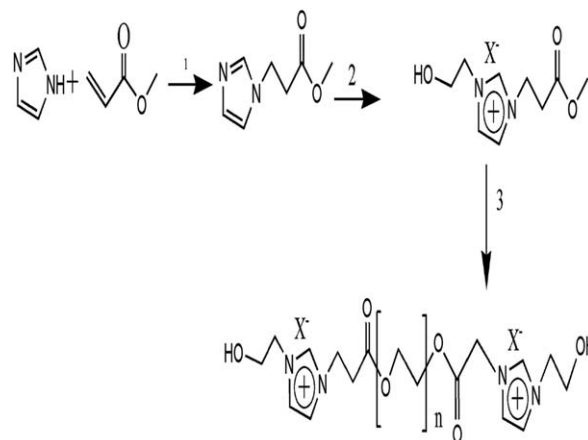
Analytically pure imidazole and PEG (PEG-300) were purchased from Kelong Chemical Reagent Co. (Chengdu, China). Methylacrylate (MA), anhydrous methanol, and dimethylacetamide were purchased from Tianjin Bodi Chemical Reagent Co. (Tianjin, China). PU was purchased from Yantai Wanhua Polyurethanes Co. (Yantai, China), and it was dried at 80°C for 4 h in an oven before use. 2-Chloroethanol and 2-bromoethanol were supplied by Nanxiang Reagent Co. (Shanghai, China). Poly-1,4-butylene adipate glycol (PBA2000) was purchased from Yutian Reagent Co. (Qingdao, China). All other chemicals used in this study were analytically pure and used as received.

Synthesis of IL-Cl and IL-Br

Imidazole and MA were allowed to react in anhydrous methanol at 80°C for 24 h. To the obtained methyl 3-(1-imidazolyl)propanoate, 2-chloroethanol and 2-bromoethanol were added in methylbenzene at 80°C for 48 h to yield the methyl 3-(3-(2-hydroxyethyl)imidazole-1-yl)propanoate chloride salt (IL-Cl) and methyl 3-(3-(2-hydroxyethyl)imidazole-1-yl)propanoate bromate salt (IL-Br), respectively.

Synthesis of IL-PEG

The IL-Br and IL-Cl (the molar ratio of IL-Br to IL-Cl was 4 : 1) reacted with PEG-300 at 140°C for 8 h under a pressure of 0.005 MPa. The polyether electrolyte IL-PEG was synthesized through the ester-exchange reaction. The main reaction is shown in Scheme 1.



Scheme 1. Synthesis route of main reaction.

Preparation of ILs/PU Gel Polymer Electrolytes

A series of ILs/PU gel polymer electrolytes were prepared by mixing PU and PBA2000 with different contents of IL-PEG in a Haake torque rheometer with an electrically heated mixing head and two noninterchangeable rotors. The temperature, rotor speed, and blending time were set at 160°C, 60 rpm, and 8 min, respectively. ILs/PU gel polymer electrolytes flakes with 1 mm thickness were prepared by compression molding at 160°C.

Characterization

Nuclear Magnetic Resonance Analysis. IL-Cl and IL-Br were characterized on Bruker AV II-400 MHz (Bruker, Germany) using D₂O as solvent and tetramethylsilane as internal standard.

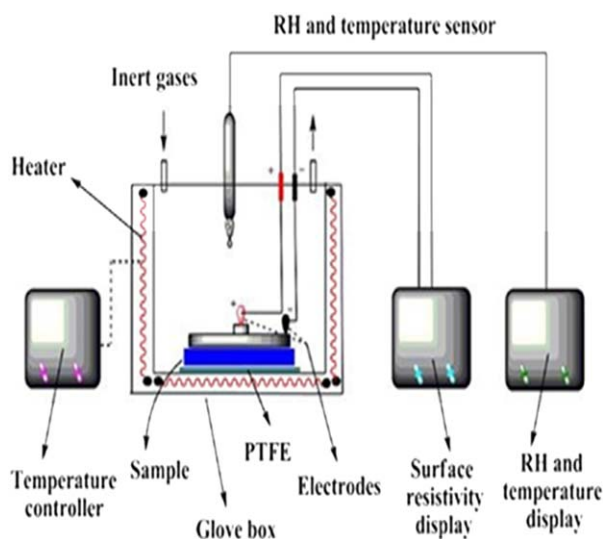
Fourier Transform Infrared (FTIR) Analysis. IL-Cl, IL-Br, IL-PEG, and PU were characterized on a Nicolet iS10 FTIR spectrometer (Thermo Fisher Scientific Co., Waltham, MA) on a resolution setting of 4 cm⁻¹. The scanning range was altered from 400 to 4000 cm⁻¹.

Electrical Conductivity Test. IL-PEG was characterized on a DDS-307 electrical conductivity tester (Shanghai Leici, Shanghai, China). The electrode used was platinum black electrode, and the electrode constant was 0.994/25°C.

Thermogravimetric Test. IL-PEG, PU, and gel polymer electrolyte were characterized on a SDT-Q600 thermo gravimetric analyzer (TA Instruments, New Castle, DE) at a heating rate of 10°C/min from room temperature to 600°C in an air atmosphere. Thermogravimetric (TG) curves were analyzed by a TA universal analysis program.

Differential Scanning Calorimetry Test. Differential scanning calorimetry (DSC) measurements were carried out in a DSC204 (NETZSCH, Germany) analyzer from -100°C to 150°C with a heating rate of 10°C/min. Approximately 5 mg of sample were placed in the aluminium pans for measurement under N₂ atmosphere. Glass transition temperature was calculated from the peak of the first derivative of the inflexion in the DSC curve.

Scanning Electron Microscopy. Polymer electrolyte films were characterized using a JEOLJSM-5900LV scanning electron microscope (Daojin, Japan). The samples were fractured in



Scheme 2. Scheme of the instrument for the surface resistivity test. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

liquid nitrogen and then covered with a thin gold layer to prevent charging.

Tensile Strength Test. For mechanical testing, dumbbell specimens were cut from the flakes. Uniaxial tensile testing of the composites was carried out with a tensile testing machine (Shanghai Yuanzhong, China) with a crosshead speed of 10 mm/min at room temperature.

Surface Resistivity Measurement. A surface resistivity meter (ZC46A, Shanghai, China) was used to measure the surface resistivity of the gel polymer electrolytes. The environments of different RHs and temperatures for the surface resistivity tests were obtained with a special glove box that could be infused with high-purity nitrogen. The scheme of the instrument is

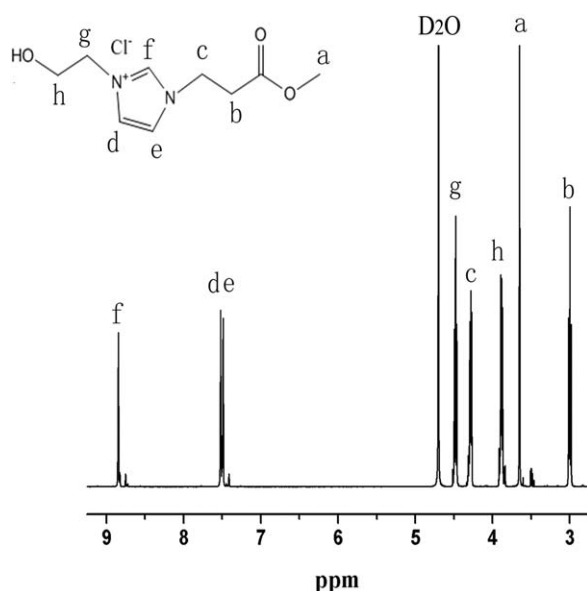


Figure 1. ^1H -nuclear magnetic resonance spectrum of IL-Cl.

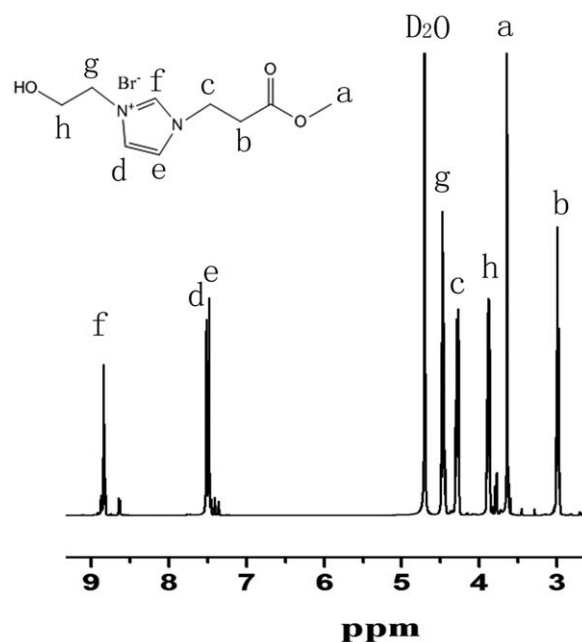


Figure 2. ^1H -nuclear magnetic resonance spectrum of IL-Br.

shown in Scheme 2. Every environment with different RH and temperature values was stabilized for more than 2 h before testing. The RH was detected by a thermo hygrometer (TRH-AZ, provided by Shinyei Co., Tokyo, Japan).

RESULTS AND DISCUSSION

Studies on ILs Properties

Synthesis of ILs. Figures 1 and 2 are the ^1H -nuclear magnetic resonance spectra of IL-Cl and IL-Br, respectively. As can be seen, the spectra are identical. The chemical shifts of all kinds of H atoms are listed in Table I. The result was in accordance with a recent report.²⁹ The resonance signals of protons from $-\text{CH}_2\text{CH}_2\text{COOCH}_3$ are observed at 3.03, 3.73, and 4.36 ppm, and protons in imidazole ring exist at 7.57, 7.60, and 8.93 ppm. The protons in 2-chloroethanol are observed at 4.56 and 3.96 ppm. From the spectra and analysis, it was found that the ILs were synthesized successfully.

Figure 3 shows the FTIR spectra of IL-Cl and IL-PEG due to the similarity on FTIR spectra between IL-Cl and IL-Br. As illustrated in Figure 3, both the samples had absorption band around 1560 and 1450 cm^{-1} . It indicates that ester-exchange

Table I. Chemical Shift Analysis of ILs Hydrogen Atom

Chemical shift	Intensity	Proton type
3.08	2H,m	$\text{CH}_2\text{CH}_2\text{CO}$
3.73	3H,S	CH_3
3.96	2H,m	$\text{CH}_2\text{CH}_2\text{OH}$
4.36	2H,m	$\text{CH}_2\text{CH}_2\text{CO}$
4.56	2H,m	$\text{CH}_2\text{CH}_2\text{OH}$
7.57 and 7.60	1H,m	$-\text{N}-\text{CHCH}-\text{N}-$
8.93	1H,m	$-\text{N}-\text{CH}-\text{N}-$

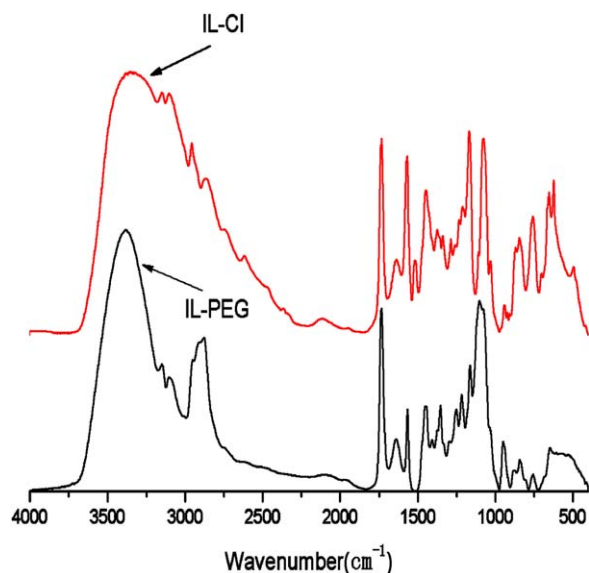


Figure 3. FTIR spectra of IL-Cl and IL-PEG. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

reaction does not destroy the structure of imidazole ring.²⁹ In comparison with IL-Cl, the stretching vibration of the C—H around 2877 cm^{-1} appears, and the absorption peak of C—O—C around 1100~1200 cm^{-1} broadens and strengthens. Besides, the characteristic absorption band of —CH₃ of IL-Cl end groups vanished. It reveals that the ester-exchange reaction has occurred successfully. According to the previous analysis,²⁹ it obviously demonstrates that the IL-PEG was successfully synthesized by ester-exchange reaction.

Ionic Conductivity of ILS. Figure 4 shows the effect of ILS ratio on ionic conductivity of PEG. The curve reveals that the ionic conductivity is highest when the molar ratio of IL-Br : IL-Cl is

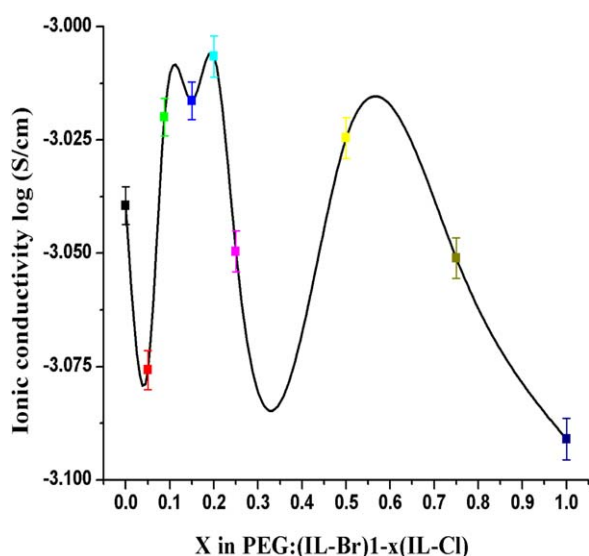


Figure 4. Effect of ILS ratio on the conductivity of PEG. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

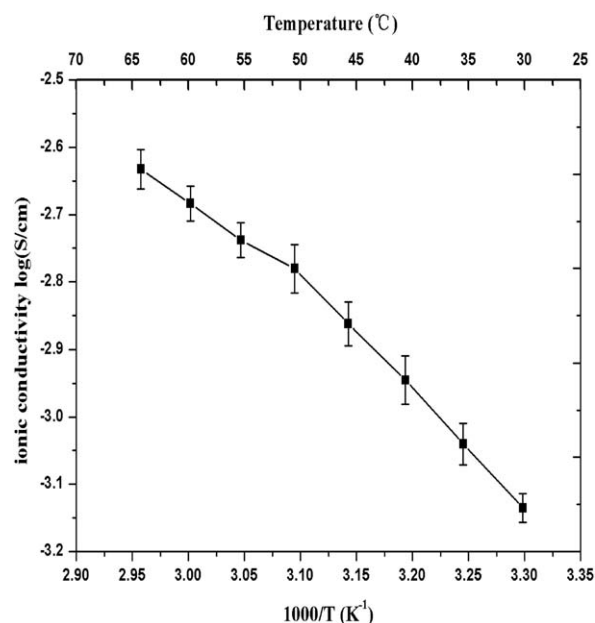


Figure 5. Effect of temperature on the conductivity of IL-PEG.

4 : 1. Thus, this molar ratio was chosen to synthesize IL-PEG to get good ionic conductivity ability.

The temperature dependence of ionic conductivity of the IL-PEG is shown in Figure 5. As can be seen, the relevant curve is divided into different linear parts. Both of the two parts are in good accordance with Arrhenius relationship:

$$\sigma = \sigma_0 \exp(E_a/kT) \quad (1)$$

where E_a is the activation energy, σ_0 is the pre-exponential factor, and k is the Boltzmann constant. This relationship suggests an ionic conduction mechanism,³⁰ and the determinant of the ion conductivity is activation energy. The curve has a point of inflection at 50°C. It illustrates that the activation energy of IL-PEG is different between low temperature ($E_a = 14.41$ KJ/mol) and high temperature ($E_a = 10.01$ KJ/mol), and it is higher when the temperature is below 50°C. That is because there exist hydrogen bonds in the ILS as C—H...Cl, O—H...Br, and O—H...O=C at low temperature.^{31,32} The hydrogen bonds increase the viscosity and decrease the ion mobility. The low ion mobility results in high activation energy at low temperature. The hydrogen bonds rupture as the temperature rise. So the activation energy is lower at high temperature.

Studies on IL/PU Gel Polymer Electrolytes

PU and IL-PEG mixed in a Haake torque rheometer to prepare the gel polymer electrolytes at 160°C. At the beginning of the study, set contents PU was added to the torque rheometer. IL-PEG was added to the system after PU melting. The torque of system dropped to zero immediately when the IL-PEG added. The phenomenon can contribute to the IL-PEG has good lubricity.^{33,34} However, the torque rose again when PBA added surpass 6 g. It indicated the IL-PEG has poor lubricity with PBA.

FTIR Analysis of PU. Figure 6 shows the FTIR spectra of PU. The absorption peak around 3328 cm^{-1} belongs to the stretching vibration peak of N—H bond, and that around 2940 and

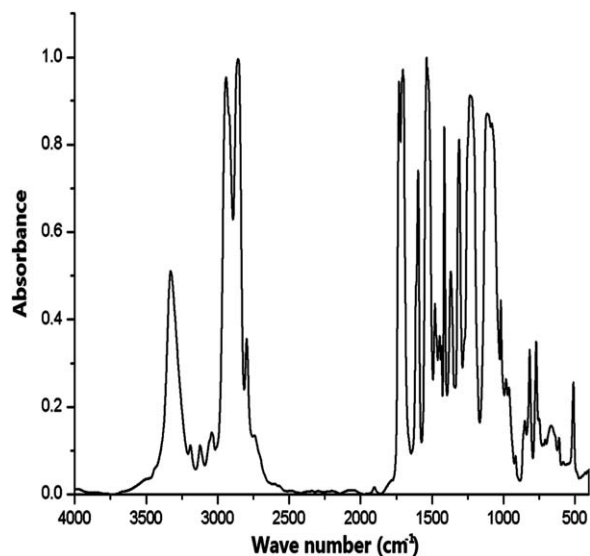


Figure 6. FTIR spectra of PU.

2885 cm^{-1} correspond to the stretching vibration peak of C—H bond. The peaks at 1597 and 1538 cm^{-1} are the characteristic skeletal vibration of benzene ring. The characteristic stretching vibration peak of C=O bond around 1731 cm^{-1} is detected. And the absorption peaks of 1233 and 1113 cm^{-1} belong to stretching vibration peak of C—O—C bond. According to the previous analysis, it indicates that PU matrix has both ester bond and ether bond.³⁵

Surface Resistivity of Polymer Electrolyte Films. Figure 7 shows the effect of temperature on surface resistivity of the polymer electrolyte film. The surface resistivities decline as the temperature increases like the curve in Figure 5. When the temperature increases, the ion movement ability and the mobility

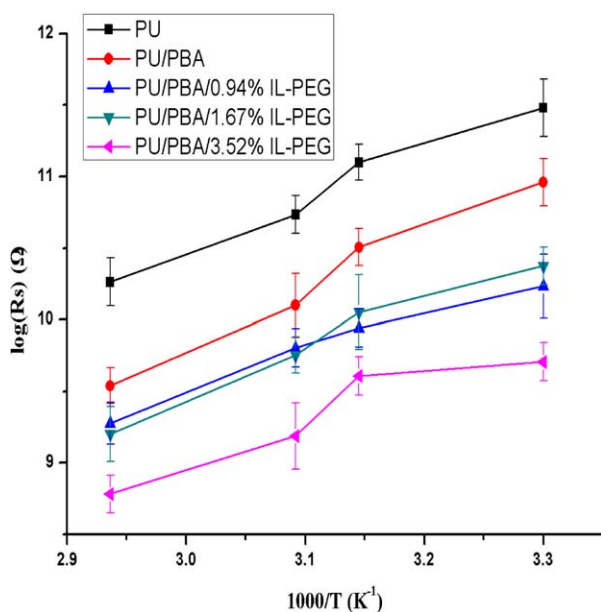


Figure 7. Effect of temperature on surface resistivity of the polymer electrolyte film. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

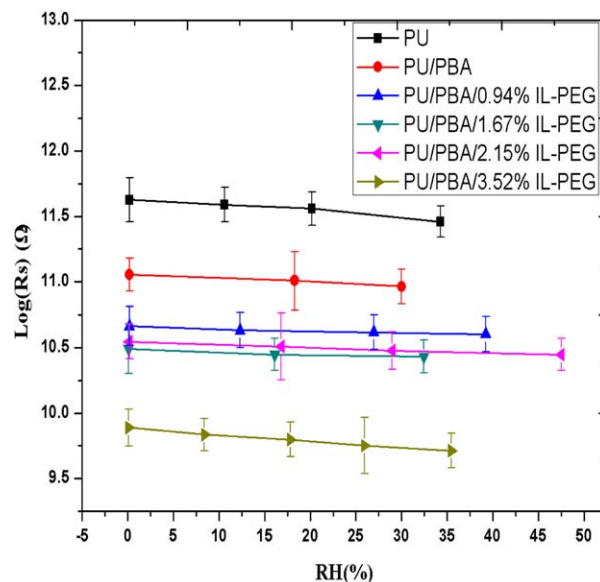


Figure 8. Effect of relative humidity (RH) on surface resistivity at room temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

of segment strengthen³⁶ resulting in increase in ion conductivity. So the surface resistivities decrease. Similar to the electrical conductivity of IL-PEG, the gel polymer electrolytes have a point of inflection at 50°C. It favors formation of hydrogen bonds at low temperature, leading to poor antistatic property. The linear relationship suggests an Arrhenius-type activated process. Some studies used the Arrhenius relationship to explain the temperature dependence of ionic conductivity in solid and gel polymer electrolytes.^{32,37}

Figure 8 demonstrates the effect of RH on surface resistivity at room temperature. The surface resistivities slightly slow down as the RH decreases, indicating that the gel polymer electrolytes

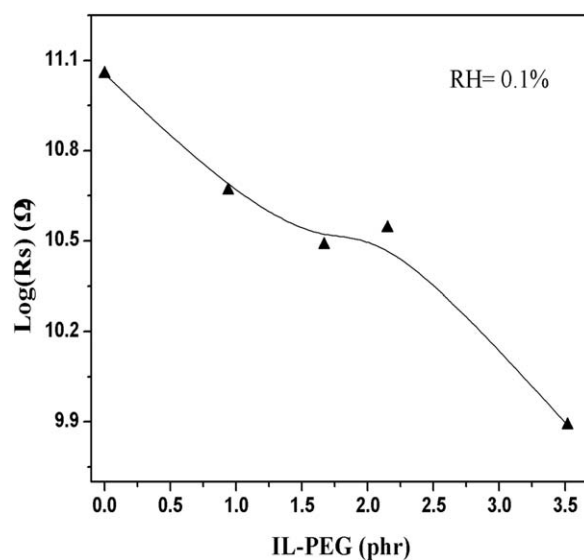


Figure 9. Effect of IL-PEG content on surface resistivity under an RH of 0.1% at room temperature.

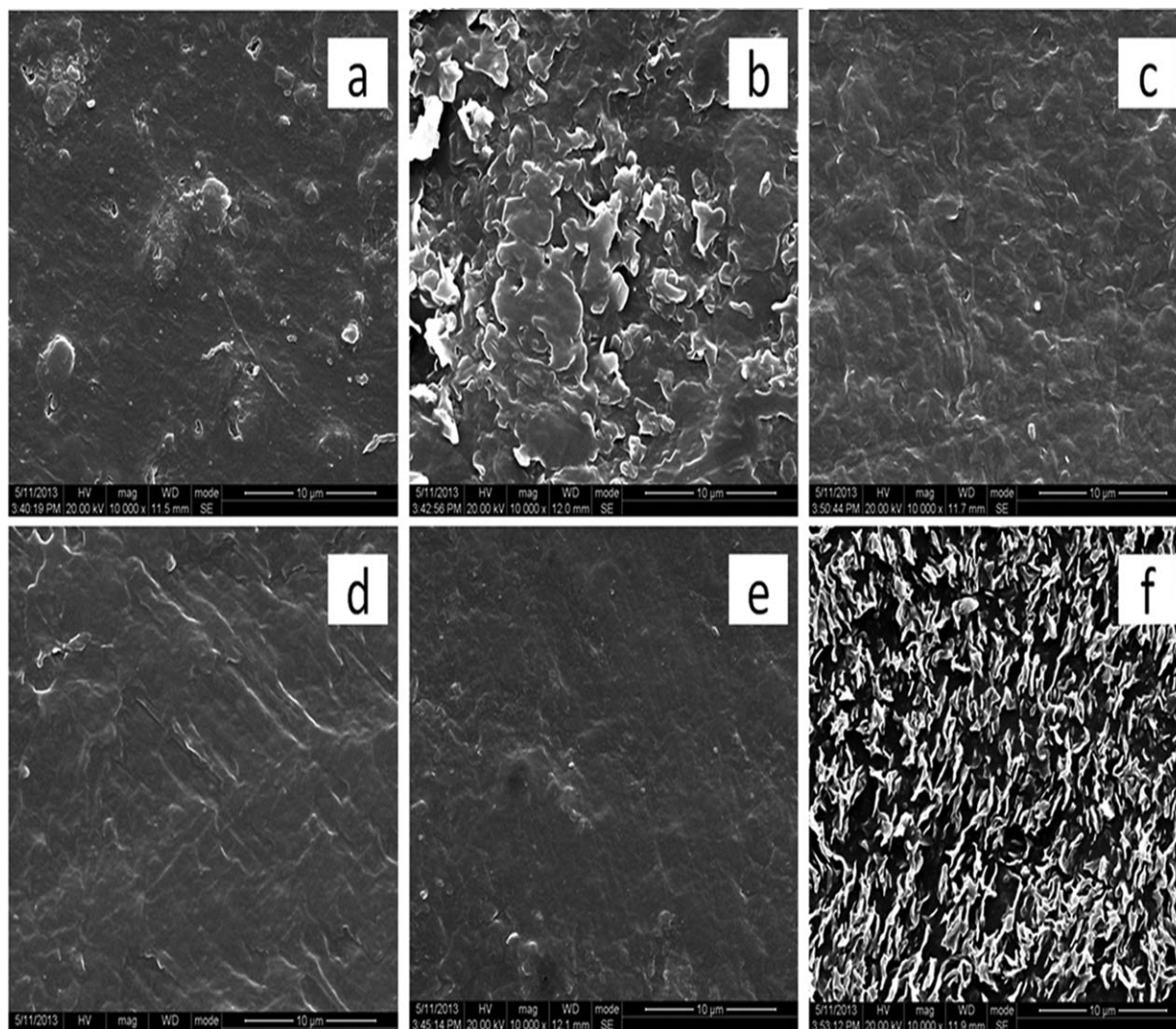


Figure 10. SEM pictures of PU and gel polymer electrolytes: (a) PU; (b) PU : PBA = 86 : 14; (c) PU : PBA : IL-PEG = 86 : 14 : 0.97; (d) PU : PBA : IL-PEG = 86 : 14 : 1.67; (e) PU : PBA : IL-PEG = 86 : 14 : 3.52; and (f) PU : PBA : IL-PEG = 86 : 12 : 3.75.

have low humidity sensitivity. The IL is composed entirely of ions; so, it need not dissociate in water or organic solvent like salt. So the gel polymer electrolytes are not humidity sensitive. Also, the materials have good antistatic ability even at low RH.

Figure 9 shows the effect of IL-PEG content on surface resistivity under an RH of 0.1% at room temperature. The surface resistivity decreases as the IL-PEG content increases. Because the conductive mechanism of IL-PEG is ionic conduction, the number of conductive ions increases as the content of IL-PEG increases. And the more conductive ions result in reduced surface resistivity. When the content of IL-PEG is 3.52 wt %, the surface resistivity is below $10^9 \Omega$ at high temperature. The gel polymer electrolytes have remarkable antistatic ability.

Surface Morphology of Polymer Electrolytes Films. Figure 10 shows the scanning electron microscopy (SEM) pictures of PU and gel polymer electrolytes. Figure 10(a) is the SEM picture of PU film, and it shows that the surface of PU is not very flat.

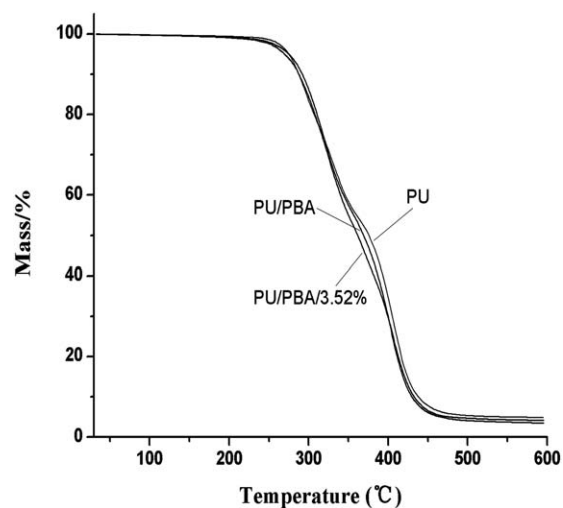


Figure 11. Thermogravimetric (TG) curves of PU and modified PU.

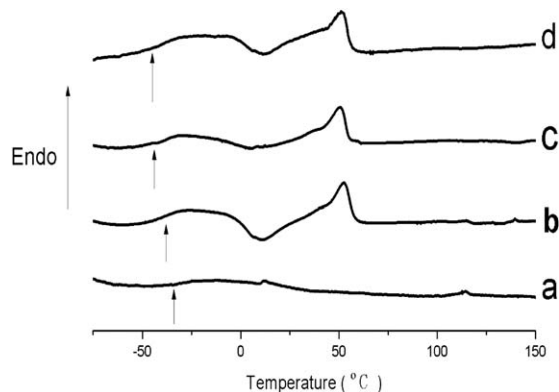
Table II. Thermal Degradation Data of Polymer Electrolytes

Sample	A		B		C		Grayish char %
	T_0 (°C)	T_{max} (°C)	T_0 (°C)	T_{max} (°C)	T_0 (°C)	T_{max} (°C)	
PU	267.5	295.5	-	329.1	-	405.1	4.84
PU/PBA	276.8	-	-	317.7	-	401.7	3.46
PU/PBA 7 g/3.52% IL	275.5	-	-	323.8	-	406.3	4.12

Figure 10(b) is the SEM picture of PU and PBA (86 : 14 by wt). The PBA is crystalline in the PU/PBA composite; so, the surface of the composite is uneven because of the existence of the two different phases in the material. Furthermore, Figure 10(c) is the SEM picture of gel polymer electrolytes whose proportion is PU : PBA : IL-PEG = 86 : 14 : 0.94. The surface smoothness of material greatly increased when compared with Figure 10(b). A comparison of Figure 10 subparts (b), (c), (d), and (e) shows that surface smoothness of material increases as the content of IL-PEG increases. The phenomenon can contribute to the fact that IL-PEG destroys the crystallinity of PBA and enhances the compatibility of PBA and PU to eliminate phase separation and make the surface more even. However, when the content of PBA is redundant, the content of IL-PEG is not enough to destroy PBA crystallinity. So the phase separation of material results in uneven surface. Therefore, it is found that the optimal content of PBA is 14 portions.

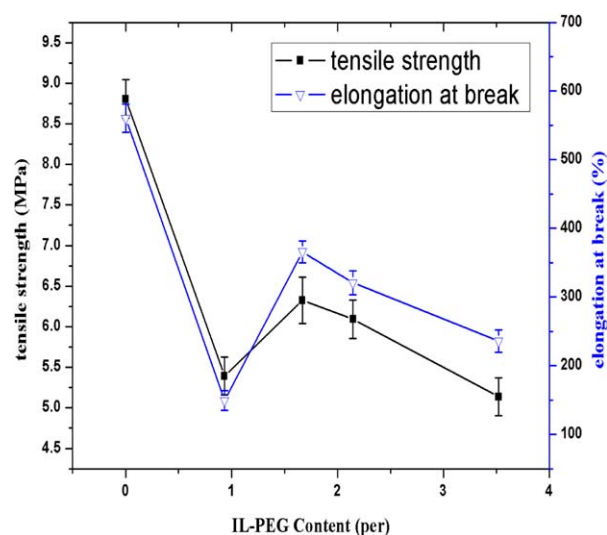
Thermal Properties of PU and Polymer Electrolytes. Figure 11 and Table II show the TG curves and the thermal degradation data of PU and modified PU. Initial decomposition temperature of PU is 268°C. The PU has good thermal stability. When PBA is added to PU matrix, the initial decomposition temperature increased to 277°C. Almost no weight loss is observed for PU/PBA/IL complex up to 276°C. The TG curve has little difference with PU/PBA material when IL-PEG is added to the material. It is noted that the addition of plasticizers decreases the initial decomposition temperature due to their volatility.²⁶ On the contrary, the nonvolatility of the IL is of special importance for their application.

DSC curves of pure PU and modified PU are shown in Figure 12. In Figure 12(b–d), the characteristic endothermic

**Figure 12.** Differential scanning calorimetry (DSC) curves of (a) pure PU, (b) PU/PBA, (c) PU/PBA/1.7% IL-PEG, and (d) PU/PBA/3.52% IL-PEG.

peak around 52°C corresponds to the T_m of PBA. A characteristic transition in DSC curve of PU are observed at around -35°C corresponding to the glass transition (T_g) value. A decrease in T_g value from -35°C to -37°C has been observed, when PU is blended with PBA. This indicates a slight increase in the flexibility of PU chains when PBA is added. On addition of different amounts of IL, T_g values around -40°C has been observed, which indicates a further increase in the flexibility in the PU backbone.^{25,32} The IL has the function of plasticizer.

Mechanical Properties of Polymer Electrolyte Films. Figure 13 demonstrates the effect of IL-PEG content on tensile properties. As shown in Figure 13, the tensile strength and elongation at break of PU/PBA composite material were 8.8 MPa and 620%, respectively. When the content of IL-PEG is 0.97%, the tensile strength and elongation of polymer electrolytes sharply decrease. The reason is that IL-PEG destroys a part of PBA crystalline. So the system has phase separation, which resulted in the decline of the tensile strength and elongation at break. When the content of IL-PEG is 1.7%, the tensile strength and elongation at break of polymer electrolyte increase. Because the content of IL can totally destroy the crystalline of PBA, the PU and PBA are in a complex amorphous phase. The tensile strength and elongation at break decrease as the content of IL-PEG further increase. The IL-PEG is liquid in the material and is the weakest component in the system; so increase in IL-PEG content decreases the tensile strength and the elongation at break.

**Figure 13.** Effect of IL-PEG content on tensile properties. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

CONCLUSION

IL-Cl and IL-Br were synthesized by addition reaction between imidazole and MA, and further quaternized by 2-chloroethanol and 2-bromoethanol, respectively. Then, IL-PEG was synthesized through ester-exchange reaction between prepared ILs and PEG. Finally, a series of gel polymer electrolytes were prepared by PU/PBA and with different content of IL-PEG in a Haake torque rheometer with the method of melt blending. The surface resistivity of the prepared PU/PBA/IL-PEG gel polymer electrolytes remarkably decreased, and it was below $10^9 \Omega$ when the temperature was above 60°C. The surface resistivity changed slightly when the RH changed from 40% to 0.1%. The polymer electrolytes had smoother surface and could be used without any decomposition when the processing temperature was below 270°C. The tensile strength and elongation at break were not obviously destroyed with the addition of IL-PEG. Based on the results, it can be concluded that the material can be used as antistatic material in environment with low RH. Also, the material is easily prepared by melt blending without solvent in Haake torque rheometer; so, this material has potential industrial application value.

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