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Effect of methylsisesquioxane filler on the properties of ionic liquid based polymer electrolyte

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Abstract

Composite electrolyte comprising methylsisesquioxane (MSQ) filler and 1-butyl-3-methyl-imidazolium-tetrafluoroborate ($BMImBF_4$) ionic liquid (IL) in poly(2-hydroxyethyl methacrylate) (PHEMA) matrix had been prepared. The polymer matrix was formed by free radical polymerization of HEMA macromer, and MSQ was produced in situ from methyl-trimethoxysilane by the sol–gel method. Infrared spectroscopy and dynamic mechanical analysis were employed to give insight into the interactions among the methylsisesquioxane filler, $BMImBF_4$ and the PHEMA polymer matrix. The PHEMA-IL-MSQ hybrids and the PHEMA-IL electrolyte without MSQ were investigated regarding their ionic conductivity and thermal and electrochemical properties. $BMImBF_4$ increased the thermal stability of the polymer and provided the ion conductivity; MSQ filler as the additive increased the mechanical strength of the polymer and provided the ion conductive pathway. The electrolyte with MSQ at the 10 wt% showed the highest ionic conductivity of 5×10^{-4} S cm^{-1} which was five times higher than that of the electrolyte without MSQ, and the electrochemical window was up to 3.6 V.

Keywords: $BMImBF_4$ ionic liquid; Methylsisesquioxane filler; Polymerization

1. Introduction

Room temperature ionic liquids (RTILs) have been extensively studied for their application as ‘green’ organic solvents and as safe electrolytes in solar cells, batteries, supercapacitors and other electrochemical devices [1–7]. Typical RTILs have wide liquid–phase range, non-flammability and very low vapor pressure at room temperature, wide electrochemical windows, high ionic conductivity, and excellent thermal and chemical stability. The alkyl imidazolium based ionic liquids have been investigated as possible electrolytes for lithium and lithium ion cells [8,9]. One of the interesting developments in the field of room temperature ionic liquids is the combination of the organic salts and polymers to form gel-like, polymer-salt composites for the applications to electrochemical devices [10–14].

Solid polymer electrolyte or gel polymer electrolyte has been widely studied due to its dimensional stability and its appliance in high energy batteries, but its ionic conductivity usually is lower than 10^{-5} S cm^{-1}. Most approaches to increase the ionic conductivity of, for example, PEO-based electrolyte, focused on lowering the degree of PEO crystallization or reducing the glass transition temperature. A variety of methods may be used, e.g. polymer structures modifications [15,16] and the incorporation of plasticizers [17,18] or ceramic fillers [19,20]. Adding of ceramic fillers to electrolyte has been proposed to be a good approach to an all solid-state design.

The preparation of hybrid organic–inorganic composites has attracted much attention because such hybrids may show controllable physical and chemical properties, such as optical, electrical and mechanical behaviors, by combining the properties of both organic polymers and inorganic compounds. The addition of inorganic fillers such as silica (SiO_2), alumina (Al_2O_3), copper oxide (CuO) or titania (TiO_2) particles into the polymer electrolyte results in the enhancement of physical strength as well as the increase in

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the absorption level of electrolyte solution [21–25]. In the case of the inorganic particles precipitated in situ in a regular manner in the polymer matrix, a strong interaction between the polymer and inorganic particle is expected to result in a hybrid with markedly improved mechanical properties and ionic conductivity [26,27]. To this date, polymer electrolytes containing both RTIL and dispersed ceramic fillers have not been presented, and the interaction between RTIL and the ceramic filler has not been proved yet.

The aim of this work to prepare ionic liquid based polymer electrolyte, we tried to polymerize 2-hydroxyethyl methacrylate (HEMA) in 1-butyl-3-methyl-imidazolium-tetrafluoroborate ($BMImBF_4$) ionic liquid as the polymerization solvent. $BMImBF_4$ was entrapped and used as both electrolyte and plasticizer [28] in this system, and methylsisesquioxane (MSQ) filler derived from methyltrimethoxysilane as the additive to increase the mechanical strength of the polymer. The ceramic filler was expected to provide desirable physical, thermal, mechanical, and electrochemical properties.

2. Experimental

2.1. Chemicals

2-Hydroxyethyl methacrylate (HEMA) was purchased from Acros, purified by distillation. Poly(ethylene glycol) methyl ether methacrylate (PEGMA, $M_n=475$), 2,2′-azo-bis-isobutyronitrile (AIBN) were purchased from Aldrich. 1-Butyl-3-methyl-imidazolium-tetrafluoroborate ($BMImBF_4$, $\geq 98\%$) and methyl-trimethoxysilane (MTMOS) were purchased from Solvent Innovation and Sigma, respectively.

2.2. Preparation of the composite polymer electrolytes

Composite electrolytes were prepared by concurrent free radical polymerization of HEMA and PEGMA vinyl monomers and chemical polymerization of MTMOS [12, 29,30]. A typical procedure can be described as follows: HEMA and PEGMA were mixed with a volume ratio of 3:1, and AIBN in 0.5 wt% with respect of HEMA+PEGMA as initiator of the free-radical copolymerization. The mixture was sonicated and blended to make a homogeneous phase.

The sol–gel polymerization of MTMOS was catalyzed by adding a mixture of water and HCl respecting the following molar ratio: $MTMOS:H_2O:HCl=1:2:0.002$. The sol was stirred for 0.5 h then $BMImBF_4$ RTIL was added at the volume ratio of 1:2 to the total amount of monomers. The sol was stirred for 1 h before mixing with the monomers mixture and then stirred for another 1 h [13]. The resulted mixture was cast on glass plate or polytetrafluoroethylene mould and heated at 80 °C to initiate the free radical copolymerization of HEMA and PEGMA, and continue the sol–gel reaction to produce film material after 20 h. The obtained membrane was further treated at 60 °C in a vacuum for 5 h to remove the residual plasticizers (e.g. monomers, water and methanol).

The MSQ content in the resulting composites was adjusting up to 15 wt% by varying the weight ratio of MTMOS/monomers. With the content of MSQ above 15 wt%, particles were emerged from the surface of the membrane. Herein the electrolytes with different weight percentages of MSQ at 5, 10 and 15% were abbreviated as MSQ05, MSQ10, MSQ15, respectively. For comparison, the PHEMA-IL membrane without MSQ was prepared with the same procedures and labeled as 'blank' in this work.

2.3. Characterization

Scanning electron microscopy (SEM) was performed on JEOL-JSM5310 (JEOL, Japan) electron microscopy operating at 20 kV.

The dynamic elastic modulus and dissipation (tan δ) of the electrolyte materials were measured by Metravib MAK-04 Viscoanalyser (France) at 10 Hz in the temperature range from -140 to 120 °C at a heating rate of 3 °C/min under N_2 atmosphere.

Attenuated total reflection fourier transform infrared spectroscopy (ATR-FTIR) was conducted using a Bruker Vertex 70 FTIR spectrometer equipped with a single reflection germanium crystal to produce a single-pass ATR (Germany) in the frequency range of 4000–600 cm^{-1}. ATR–FTIR spectra of the electrolytes were obtained by forming thin films containing the interesting materials.

Electrochemical window of the electrolyte was checked by cyclic voltammetry using a three-electrode configuration. Both working electrode and counter electrode were stainless steels (SS), and an Ag wire (inside a frit containing $BMIMBF_4$) was used as a quasi-reference electrode. The potential of the Ag quasi-reference electrode was calibrated to be 0.61 V more positive than that of Ag/AgCl (saturated KCl) electrode [2]. The cyclic voltammetry was performed at room temperature by using a CHI 630A electrochemical station (CHI Inc., USA).

The thermal gravimetry analysis (TGA) was carried out using a Perkin–Elmer thermal analysis TGA system. Measurements were made heating from 20 to 700 °C, at a heating rate of 10 °C/min under N_2 atmosphere.

The ionic conductivity of the electrolyte was evaluated using a complex impedance method in the temperature range of 20 to 80 °C. The sample was sandwiched between two stainless steel blocking electrodes (1.13 cm^2) and was placed in a temperature-controlled furnace. The impedance measurements were carried out on a Solartron 1255B Frequency Response Analyzer and a Solartron 1470 Battery Test Unit (Solartron Inc., UK) coupled with a computer. The impedance spectra were recorded with the help of ZPlot/Z-View software (Scribner Associates Inc.) under an ac perturbation signal of 5 mV over the frequency range of 1 MHz to 1 Hz.

3. Results and discussion

Pure PHEMA-PEGMA films were transparent, hard but crisp. While adding RTIL, the membranes became flexible and brittle, and the membranes with MSQ at different contents were translucent and elastic.

Fig. 1 shows the SEM images of the blank and the hybrid films, showing the MSQ particles embedded in the PHEMA matrix. In the present method of preparation, MTMOS was converted into MSQ via a sol–gel process that took place within a growing polymer network [31]. The rapid condensation of the MTMOS sol at the polymerization temperature made most MSQ existed as belt aggregations that were non-uniformly dispersed in the polymer matrix. The more aggregations were present with increasing the content of MSQ. The crosslinked network strengthened the mechanical property of the hybrid electrolyte, as discussed below.

Dynamic mechanical analysis was performed on hybrid electrolytes and the results of MSQ10, and blank samples are shown in Fig. 2. The main feature observed during mechanical analysis from low to high temperatures was the strong relaxation starting around −70 °C (Fig. 2(A)). At the lower temperatures, the membranes showed glassy properties with the storage modulus of 2000 MPa. With the increase of the temperature, at the range of −70 to 30 °C, the membrane changed from glassy to elastomeric, and the modulus dropped from 2000 to 1 MPa at room temperature. Although the content of MSQ was low (10 wt%), a significant change in storage modulus was observed. Above T_g, the hybrid membranes showed excellent elasticity and tenacity reflecting the crosslinked character of the MSQ materials.

The temperature corresponds to tan δ peak is usually an indication of glass transition temperature (T_g). In Fig. 2(B) the peak around 0 °C was ascribed to the glass transition temperature and the peak around 90 °C was the intenerate temperature. For the electrolyte without MSQ, the glass transition temperature was 4 °C. In the case of the hybrid electrolytes T_gs were 1, −2 and −5 °C for MSQ05, MSQ10, MSQ15, respectively. As the MSQ was doped into, the tan δ peak shifted to lower temperature, that is, the addition of the in situ formed MSQ caused a slight decrease in T_g. This was rationalized in terms of hydrogen bonding between the surfaces –OH groups of MSQ particles and the oxygen atoms of PEGMA branches that could skew the polymer chains and result in additional free volume [27]. Increasing the volume fraction of the amorphous domains and decreasing the glass transition temperature appeared to be the main rules to obtain better ionic conductivity.

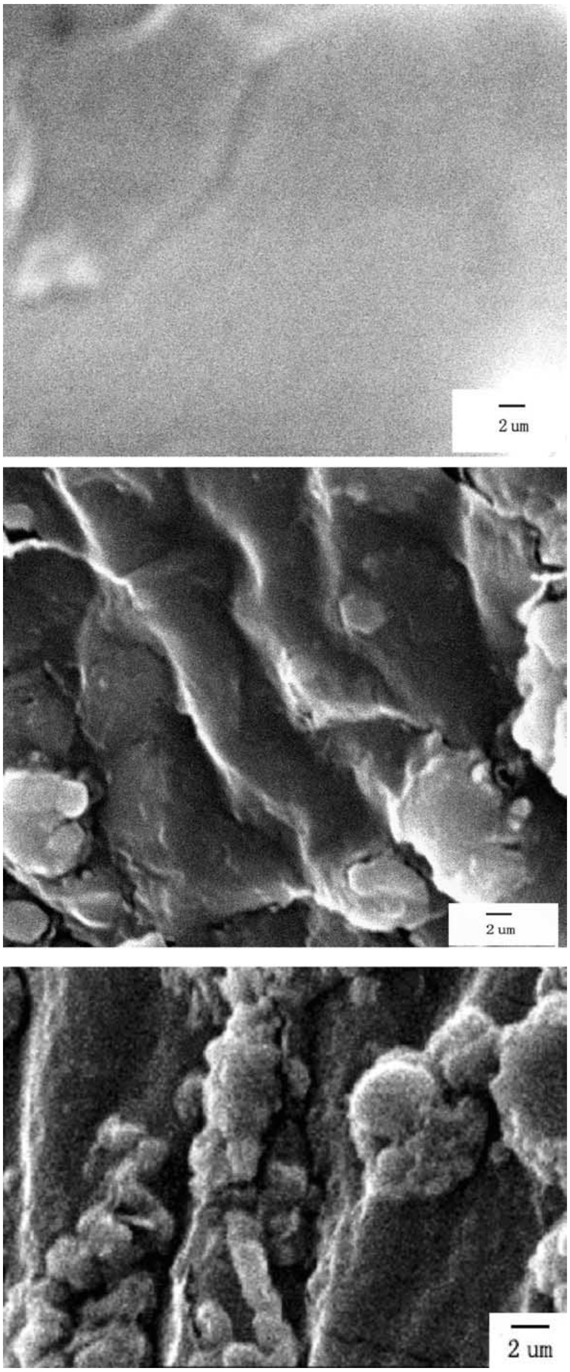

Fig. 1. SEM micrographs of the polymer electrolytes. From top to bottom were referred to the blank electrolyte, the hybrid electrolytes with MSQ at 5 and 10 wt%, respectively.

ATR-IR spectra of the polymer matrix PHEMA, PHEMA-RTIL (blank), and electrolytes with different percentages of MSQ are shown in Fig. 3. Assignments of the main bands were based on the literature values [27,32–34]. The matrix PHEMA sample showed a characteristic ester absorption band at 1723 cm^{-1} (–CO–O–), a broad band occurring at around 1026–1144 cm^{-1} (the C–O–C stretch mode of the ester group), and a broad band around 3443 cm^{-1} arose from the hydroxyl group of PHEMA. No band at 1635 cm^{-1} assigned to the C=C stretch vibration of the monomer was present indicating that C=C was

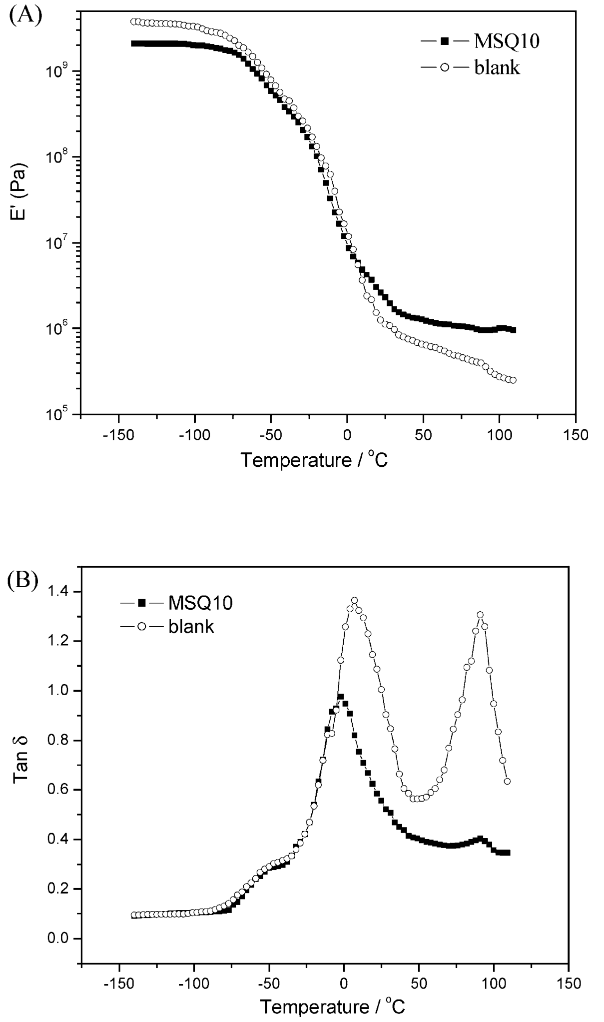

Fig. 2. Temperature dependence of (A) storage modulus and (B) tan *δ* of the blank electrolyte (open circles) and the hybrid electrolyte with MSQ at 10 wt% (squares). Heating rate, 3 °C/min; frequency, 10 Hz.

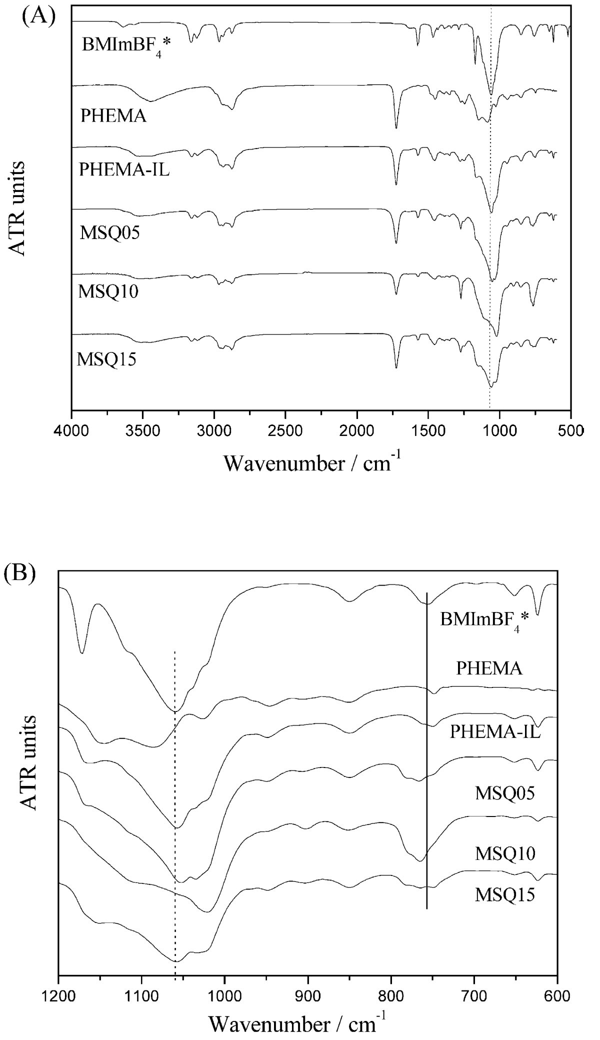

Fig. 3. ATR-FTIR spectra of the polymer electrolyte systems in the 4000–600 cm^{-1} regions (A) and the enlarged part in the 1200–600 cm^{-1} regions (B). $BMImBF_4$*, spectra of neat $BMImBF_4$ measured in transmittance for comparison; PHEMA, the matrix without RTIL and MSQ; PHEMA-IL, the blank with RTIL at the volume ratio of 2/1; MSQ05, MSQ10 and MSQ15 were hybrid electrolytes with MSQ at the weight percent of 5, 10 and 15%, respectively.

transformed to C–C completely. As for the RTIL incorporated, the bands of the PHEMA-IL electrolyte showed a few changes giving some interesting information. Compared with the infrared spectra of pure $BMImBF_4$ and PHEMA, the aromatic C–H stretching absorption bands at 3121, 3162 cm^{-1} shifted to 3118, 3157 cm^{-1}, respectively, and the hydroxyl groups band at 3443 cm^{-1} became wider. This means that the hydrogen bond between BF_4^- and –OH of the PHEMA was present [35].

As shown in Fig. 3(B), the B–F asymmetry stretching band at 1059 cm^{-1} shifted to 1056 cm^{-1} when $BMImBF_4$ was dispersed in PHEMA (dotted line in Fig. 3), and the B–F symmetry stretching band at 756 cm^{-1} shifted to around 765 cm^{-1} when the MSQ was incorporated (solid line in Fig. 3(B)). Moreover, the relative intensity of 765 cm^{-1} band increased sharply with respect to that of the 1056 cm^{-1} band in the MSQ10 electrolyte. As we know, due to the strong electrostatic interactions of $BMImBF_4$ molecules, ion pairs or larger ion clusters might present in the neat $BMImBF_4$. As the IL was dispersed into the polymer matrix, the electrostatic interactions weakened and the hydrogen bonding between $BMImBF_4$ and –OH group in PHEMA was present, thus the vibration frequency changed. In the case of the sample with MSQ, the absorption bands at 900–1200 cm^{-1} (the asymmetric Si–O–Si stretch) were overlapped with those of BF_4^- asymmetry stretching and the C–O–C stretching mode in PHEMA polymer matrix. So it was hard to elucidate the interactions between the MSQ filler and the PHEMA matrix by the change of the C–O–C stretching mode of polymer chain [36]. However, the Si–O–Si peak was seen to shift to lower wavenumber, and the intensity increased as the content of MSQ increased. It was possible that the change of the relative intensity of 765 cm^{-1} band resulted from the grain boundary effect of the MSQ particles [36].

Note that a structurally significant presence of water can be excluded by IR measurements. More –OH groups are available from the surface of MSQ prepared from sol–gel process, hydrogen bonding interactions among the Si–OH group, the –OH end group of polymer and BF_4^- group as well as the ether oxygen could occur. So the broad bands between 3100 and 3700 cm^{-1} related to the presence of (i) C–OH (from PHEMA) and Si–OH (from MSQ), (ii) inter- and intra-molecular hydrogen bonds, and (iii) small amounts of residual water in the samples.

The TGA thermograph curves for the PHEMA-IL-MSQ composites are shown in Fig. 4. For the polymer matrix without IL and MSQ (PHEMA), a sharp and complete weight loss between 180 and 350 °C was seen, assigned to the decomposition of the polymer matrix. With the addition of the RTIL, the blank electrolyte retained its thermal stability well up to 220 °C. Above 220 °C, there was a weight loss of 97% attributing to the decomposition of the polymer matrix, along with the decomposition of $BMImBF_4$ [37]. That is, the thermal stability of the polymer matrix increased due to the interaction between $BMImBF_4$ and the matrix. In the case of the hybrid electrolytes with MSQ filler, their thermal properties showed no obvious change compared with that of the blank electrolyte and the weight remaining depended on the content of the inorganic component. These results suggested that the introduction of MSQ had little influence on the thermal stability of the given hybrid materials.

Fig. 5 shows the cyclic voltammograms of the different electrolytes. For the composite electrolyte with MSQ, the irreversible reduction started from ca. −1.8 V versus Ag reference, and the irreversible oxidation appeared at ca. 2.0 V versus Ag reference. As a result, the polymer electrolyte exhibited an electrochemical potential window wider than 3.6 V. Compared with the blank electrolyte, the electrochemical window showed almost equal windows. This meant that the MSQ filler had no influence on the electrochemical stability of the $BMImBF_4$ significantly.

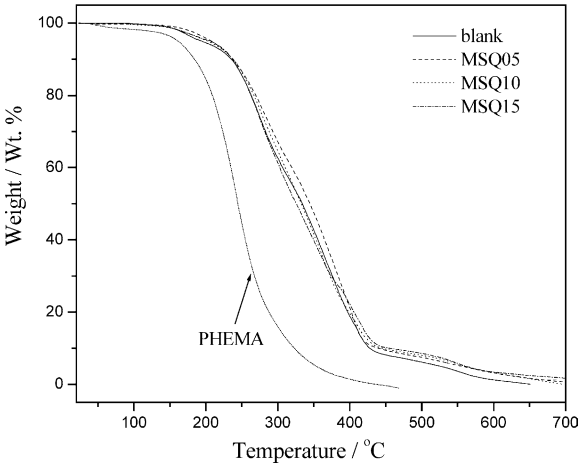

Fig. 4. TGA curves of the polymer electrolyte systems. Blank (solid line), the PHEMA-IL electrolyte with the volume ratio of monomer/IL 2/1; MSQ05 (dashed line), MSQ10 (dotted line), MSQ15 (dash-dotted line) were the hybrid electrolytes with MSQ at the weight percent of 5, 10 and 15%, respectively; PHEMA (dotted line), the matrix without RTIL and MSQ.

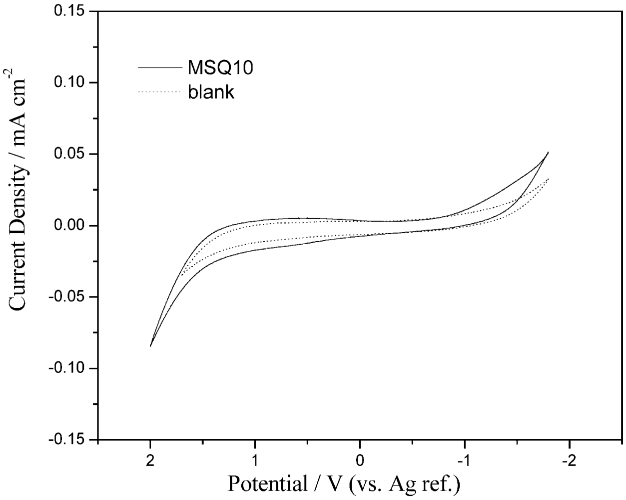

Fig. 5. Cyclic voltammograms of the blank electrolyte (broken line) and the hybrid electrolyte with MSQ at 10 wt% (solid line). Working and counter electrodes, stainless steels; quasi-reference electrode, Ag wire (inside a frit containing $BMIMBF_4$); scan rate, 10 mV/s.

The ionic conductivity of the composite polymer system has been derived from complex impedance plot. The impedance plots of different compositions for SS/electrolyte/SS sandwiched structures are shown in Fig. 6. Each spectrum showed a small arc in the high frequency range and an inclined straight line in the low frequency range. The disappearance of the semicircle in impedance spectra at high frequency is explained with a good contact between electrodes and electrolyte and the straight line is due to the migration of ions and the surface roughness of the electrodes. According to the literature [38,39], the change of the slope of the straight line at low frequency could be explained by the alteration of the microstructure of polymer electrolyte and the contacting state on the electrolyte/electrode interface, and the lower value of slope correlating to the high roughness of the electrolyte/electrode interface.

The bulk electrolyte resistance R_b can be estimated from the low frequency end of the small arc and the high frequency end of the straight line. And then the ionic conductivity (σ) of the gel electrolyte is calculated by

$$\sigma = \frac{l}{AR_b} \qquad (1)$$

where l is the thickness of the electrolyte and A is the area of the SS electrode. It was found that the conductivity of PHEMA-RTIL polymer could be enhanced by the addition of MSQ particles.

The conductivities of the electrolytes with different MSQ contents as the function of temperature are shown in Fig. 7. The dependence of the conductivity with the temperature exhibited positively curved profiles, indicating that the

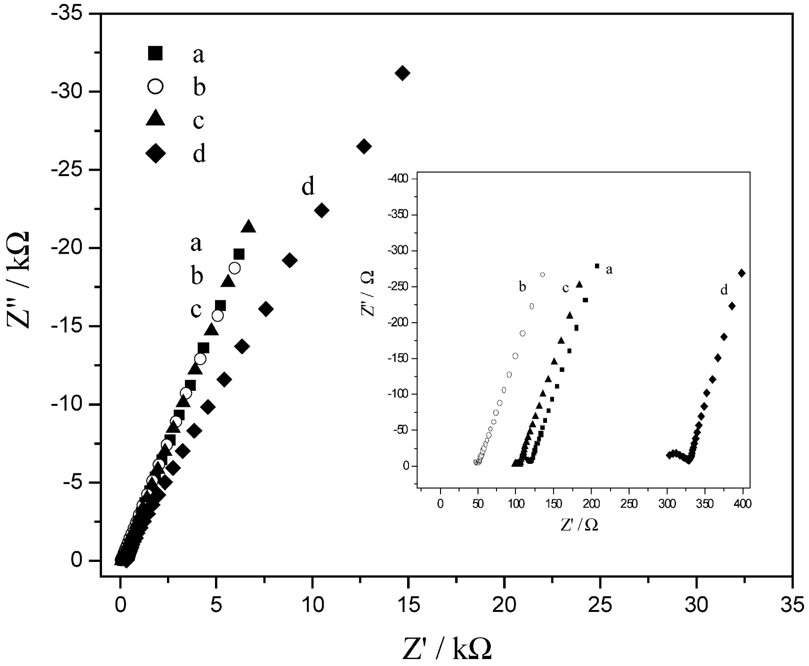

Fig. 6. Complex impedance plots of the polymer electrolyte systems. Inset is the enlarged of the impedance plots. Symbols Z' and Z'' refer to the real and imaginary components; data were collected at open circuit with ac perturbation signal of 5 mV and the frequency range of 1 MHz to 1 Hz; a, the blank electrolyte of PHEMA-IL; b–d, the hybrid electrolytes with MSQ at the weight percent of 5, 10 and 15%, respectively.

conductivity–temperature behavior cannot be correlated by the Arrhenius equation [40] (Fig. 7(A)):

$$\sigma(T) = A \exp\left(\frac{-E_a}{RT}\right) \tag{2}$$

On the other hand, the data can be fitted well to the Vogel–Tamman–Fulcher (VTF) equation [41–43] (Fig. 7(B)):

$$\sigma(T) = AT^{-1/2} \exp\left[\frac{-E_a}{R(T - T_0)}\right] \tag{3}$$

where A is a pre-exponential factor, E_a is the activation energy for ionic conduction and T_0 is the temperature at which the transport function ceases and may be considered as the ideal glass transition temperature, $T_0 = T_g - 50$. It is well known that when the ion transport was dominated by the segmental motion of an amorphous polymer electrolyte, the conductivity can be shown to depend on the free volume of the polymer and may be correlated by VTF equation [42, 43]. The incorporation of MSQ filler resulted in a conductivity enhancement in comparison to the electrolyte free of MSQ and showed no influence on the conduction mechanism. The more interesting result was an increase of the conductivity by five times (up to 5×10^{-4} S cm^{-1} at 30 °C for MSQ10) when the MSQ content reached 10 wt%, and we attributed this to the interactions between PHEMA matrix and BMImBF$_4$ as well as the MSQ particles influenced the conductivity.

In our case, this enhancement of conductivity was tentatively interpreted by a contribution of the MSQ surface that would provide an alternative path for the ionic conduction. Due to the large surface area, the MSQ particle would act as a sort of ‘solid plasticizer’ for the polymer chains by kinetically inhibiting their reorganization [44] and separating the polymer chains which provided a large free volume for ions to diffuse, that is, MSQ might provide a suitable channel for the transitions of ions. The decreased intensities of the only lump in X-ray diffraction patterns (data not shown) of the system (ascribed to PHEMA) also proved that the reduction of PHEMA crystallinity. Additional, the lower conductivity of the blank electrolyte might ascribe to the existence of the ion clusters (ion pairs, ion triple, etc.) of BMImBF$_4$. As the MSQ was incorporated into, the interactions between filler and BMImBF$_4$ and the grain boundary effect reduced ion clusters and assisted the dissociation of BMImBF$_4$, thus the hybrid electrolytes showed higher ionic conductivities.

But above 10 wt% MSQ content, the conductivity decreased slightly. The reason might be that the MSQ particles were easily aggregated, and the aggregated MSQ blocked ion-conducting pathways and slowed ion movement by causing ions to travel around MSQ particles.

4. Conclusions

Organic–inorganic hybrids based on PHEMA-BMImBF$_4$-MSQ composite electrolytes were prepared using sol–gel process for the in situ formation of the MSQ particles. The chemical polymerization of MSQ sol and the free radical polymerization of the monomers were carried out concurrently to enable the simultaneous formation of the

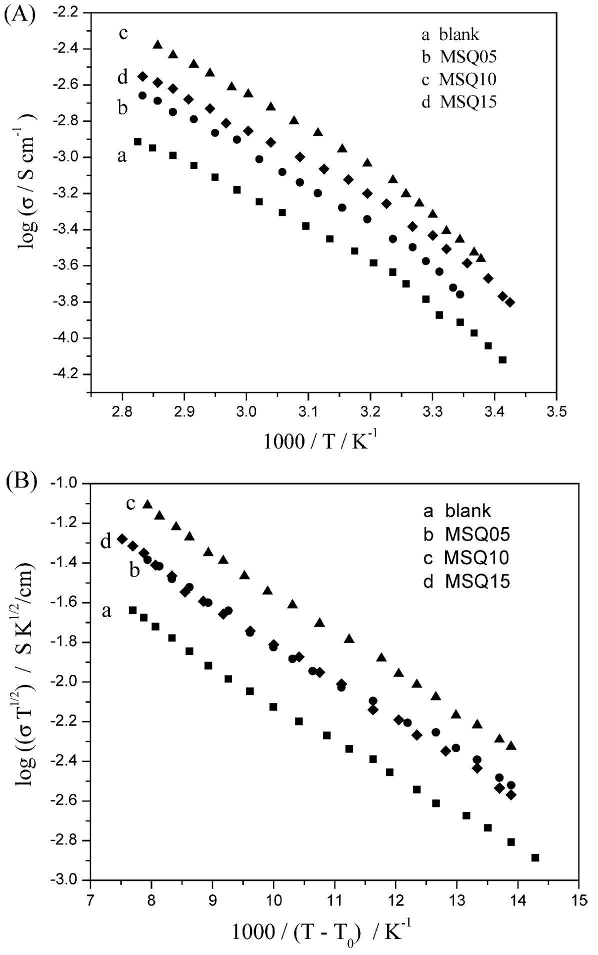

Fig. 7. Arrhenius plots (A) and VTF plots (B) for the polymer electrolyte systems; a, the blank electrolyte of PHEMA-IL; b–d, the hybrid electrolytes with MSQ at the weight percent of 5, 10 and 15%, respectively.

polymer network and the inorganic particles. In this system, $BMImBF_4$ was used both as a plasticizer and as an ionic source. $BMImBF_4$ provided the ionic conductivity and increased the thermal stability of the polymer matrix. The MSQ filler increased the mechanical strength of the polymer and provided the ion conductive pathway, but it showed no significant influence on the thermal and electrochemical stability of the hybrid system. The electrolyte with MSQ at 10 wt% showed the highest ionic conductivity of 5×10^{-4} S cm^{-1}, and the electrochemical window was up to 3.6 V.

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