



Ionic polymer–metal composite actuators obtained from sulfonated poly(ether ether sulfone) ion-exchange membranes



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ABSTRACT

The cost-effective and high-performance ionic polymer–metal composites (IPMC) were designed and prepared from ion-exchange membranes based on sulfonated poly(ether ether sulfone) (SPEES) with different degrees of sulfonation (DS). The precursor of SPEES, namely PEES, is commercially available and industrial grade. Moreover, the PEES can be transformed easily into ion-conductive SPEES through a simple sulfonation reaction. The ion exchange capacity (IEC) and water uptake (WU) of SPEES membranes increase with increasing their DS, and the proton conductivities of these hydrated SPEES membranes are subsequently enhanced. Compared with the commercial Nafion ion-exchange membrane, the SPEES membranes have higher IEC and WU. The IPMC actuators made of the SPEES membranes show the large bending strain and fast response under electric stimulation. The SPEES membrane with the highest DS (SPEES4) shows the best performance of IPMC actuators. The electromechanical behaviors of these IPMC actuators indicate that the SPEES is a candidate to substitute Nafion.

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1. Introduction

Ionic polymer–metal composites (IPMC) are one type of the most promising electroactive polymer (EAP), and have considerable potential as soft actuators and sensors owing to their large bending strain and large bending displacement under a low applied voltage [1–6]. IPMC are composed of an ion-exchange membrane (IEM) with two layers of noble metal electrodes plated on the both surfaces through electroless deposition, and they can show a fast bending deformation toward the anode due to differential water swelling between both sides of IPMC strip.

As one of the most crucial components in IPMC, the ion-exchange membrane reported in IPMC has been limited to a few commercially available perfluorinated ionic polymer products like Nafion and Flemion, owing to their excellent mechanical property, chemical stability, and high proton conductivity [7]. However, some drawbacks including back-relaxation under sustaining direct current (DC) voltage, low blocking force, high cost, and environmental unfriendliness hinder Nafion's practical applications. Therefore, researchers have been made many efforts to develop new ion-exchange membranes in the preparation of IPMC to

replace Nafion and improve the performance (strain or force) of IPMC actuators.

Learning from the chemical structure of Nafion, Jeong and Kim et al. prepared the first IPMC actuator using fluorinated acrylic copolymer membranes instead of Nafion [8,9]. No back relaxation was observed for all actuators during the DC test. However, the high voltage (3–8 V) was needed for the actuation. Moreover, fluorinated monomers were not cheap and not environmentally-friendly. Since then, nonfluorinated polymers composed of aromatic backbone, copolymers or polymer blend are considered to be one type of alternatives. Different kinds of sulfonated or carboxylated polymers have been used to prepare IPMC actuators [1,4,5,7–26]. As stated above, researchers have developed new, cost-effective and high-performance ion-exchange membranes as alternatives to the traditional Nafion membrane for IPMC actuators. Although some efforts of replacement were achieved, the preparation of high-performance ion-exchange membranes for IPMC is a matter of concern. Thus, it is still required to continue to study the ion-exchange membranes for use in high-performance IPMC actuators.

Poly(ether ether sulfone) (PEES), which is one of thermoplastic polymers, is well known for the excellent film-forming capacity, thermal and mechanical properties, and stability under acidic conditions (except concentrated sulfonic acid). The nonconductive PEES can be transformed easily into ion-conductive sulfonated

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PEES (SPEES) through a simple sulfonation reaction in concentrated sulfonic acid. And SPEES has been a potential material for proton exchange membranes instead of Nafion in fuel cells [27–29]. However, the sulfonated polymer membranes have not been developed for soft IPMC actuators until now. In this study, a series of SPEES with different degrees of sulfonation (DS) were synthesized via the sulfonation of the commercially available PEES. The effects of the sulfonation on IEC, water absorption, ionic conductivity and hydrated elastic modulus of SPEES membranes were evaluated. The electromechanical coupling performances (actuation behaviors) of this novel type of SPEES-based IPMC actuators were also investigated under low DC and sinusoidal voltages. These SPEES membranes could be further developed as cost-effective and high-performance IPMC actuators.

2. Experimental

2.1. Materials

Poly(ether ether sulfone) (PEES, Aldrich) was vacuum dried at 100 °C overnight prior to use. Tetraamineplatinum chloride hydrate ($[\text{Pt}(\text{NH}_3)_4\text{Cl}_2]$) was provided by Aladdin Industrial Inc., China. All other chemicals were supplied from Sinopharm Chemical Reagent Co., Ltd (China) and used as-received without further purification. Deionized water was used in all the experiments.

2.2. Preparations of sulfonated poly(ether ether sulfone) (SPEES) and SPEES membranes

The PEES powders were obtained from the mechanical mixing of the PEES and chloroform [29]. Typically, the dried PEES powders (20 g) were added to 400 mL of H_2SO_4 for 10 min by strong mechanical agitation (600 rpm) at 10 °C, and the reaction continued for another period of time (*i.e.* 7, 9, 11 and 13 h, respectively) under vigorous stirring at 10 °C. Then, the mixture was added to cold deionized water. The precipitated was filtered and washed with deionized water repeatedly until a neutral pH level. The SPEES samples were dried to remove the water.

The SPEES membranes were prepared by the solution casting method. Firstly, the SPEES was dissolved in *N,N*-dimethylacetamide (DMAc). The solution was then cast onto a clean glass plate and spread thoroughly by using a doctor blade. The solvent was removed by multi-stage drying process in the range of temperature from 40 °C to 135 °C. In addition, in order to reduce water uptake and keep dimensional stability of the SPEES membrane with the highest degree of sulfonation (namely SPEES4 membrane), SPEES4 membranes were subjected to further heat treatment (*i.e.* drying at 155 °C for 3 h and at 175 °C for 1 h). The resulting membranes were peeled off from the glass plate. According to different times of sulfonation reactions, the degree of sulfonation (DS) of these four SPEES membranes was 53.86%, 60.27%, 64.77%, 81.83%, respectively. Following the increasing order of DS, SPEES membranes with different DS were named sequentially as SPEES1, SPEES2, SPEES3 and SPEES4, respectively.

2.3. Preparation of IPMC actuators

Electroactive IPMC actuators were fabricated using the SPEES membranes by an electroless plating method (namely, impregnation–reduction) as described in our previous reports [4,5]. The whole process of plating Platinum for SPEES-based IPMCs consisted of four steps: surface roughening, ion exchange, primary reduction and surface or secondary reduction. In order to obtain high conductive surfaces, the plating process except roughening was repeated with 3 times of primary plating and 1 time of sec-

ondary plating. The whole process of plating Platinum spent ca. 9 days. In addition, Nafion 117-based IPMC actuators were also prepared via the same chemical plating process.

2.4. Characterization

Fourier transform infrared spectroscopy (FTIR) spectra were recorded on an Equinox 55 spectrometer (Bruker) in the range of 4000–400 cm^{-1} . ^1H nuclear magnetic resonance (^1H NMR) spectra were performed by a Bruker AV400 NMR spectrometer with deuterated dimethyl sulfoxide (DMSO-d_6) as the solvent.

The thermogravimetric analysis (TGA, TGA-7, Perkin-Elmer, USA) and differential scanning calorimetry (DSC, Q2000, TA Instruments, USA) were run at a heating rate of 20 °C/min.

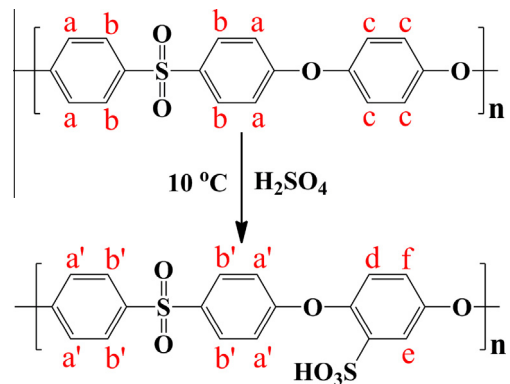


Fig. 1. Schematic representation of sulfonation of PEES. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

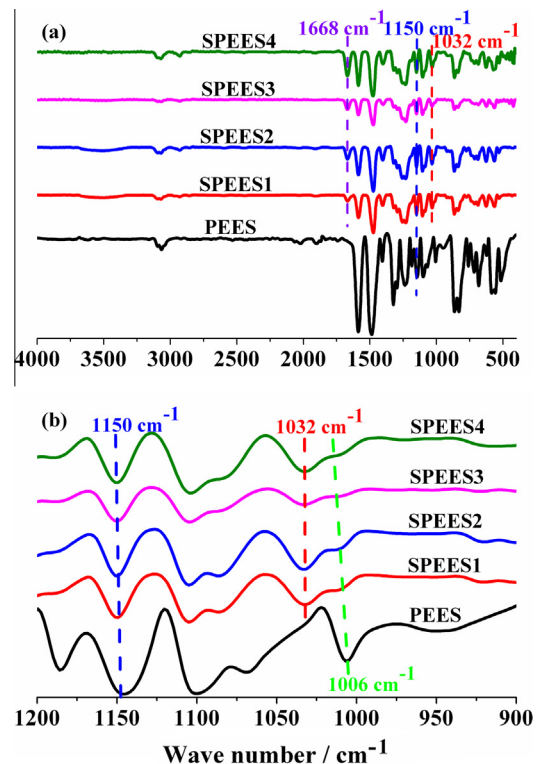


Fig. 2. FTIR spectra of PEES and SPEES: the full spectra (a), and magnified local detail (b). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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