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Optimizing Proton Conductivity in Sulfonated Polystyrene Membranes via Phosphorous Pentoxide Doping

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Abstract: Recently, Proton conducting membrane fuel cells (PCMFCs) have been recognized as a potential future and excellent medium for efficient power sources. In this present work, Polystyrene has been used to make a proton conductive membrane, mixing with phosphorus pentoxide (P_2O_5), targeting to achieve low cost and high proton conductivity under low humidity conditions. Several membranes are studied with varying amounts (three compositions: 5 wt.%, 10 wt.%, and 20 wt.%) of P_2O_5 concerning polystyrene weight and doped in 20% diluted sulfuric acid. It is expected that a new proton transport pathway is provided between the phosphoric acid and sulfuric acid in dry conditions. Results of conductivity, obtained by Electrochemical Impedance Spectroscopy (EIS), have shown excellent proton conductivity at room temperature. The 10 wt.% modified P_2O_5 membrane exhibited a higher order of proton conductivity, approximately two orders of magnitude compared to pure PS membrane at dry conditions (approximately 10^{-2} S/cm), which is the highest value among the fabricated membranes. The Fourier transform infrared spectrometer (FTIR) analysis confirmed the sulfonation of the modified membranes. These membranes are also characterized by scanning electron microscopy (SEM) and tensile test. The tensile test showed the highest strength of 1.8 MPa; while the SEM images proved the porous structure of the membranes, which is helpful to improve the proton conducting membrane (PCM) structure. So, the 10 wt.% P_2O_5 modified membrane Is a promising candidate as a novel PCM and have potential applications for use in fuel cells.

1. Introduction

In recent times, Proton-exchange membrane fuel cells (PEMFCs) have been taken into account as an excellent medium for acceptable and efficient power sources. Portable electronic device batteries, fuel cells, and desalination use proton exchange membranes (PEM) in a wider range because of their significant effectiveness (Bi et al., 2008; Mirfarsi et al., 2020). The most popular proton exchange membrane is Nafion, which has high proton conductivity and chemical and electrochemical stability. Nevertheless, despite its excellent acceptability in PEMFCs, it has some inconveniences in terms of high cost, loss of water uptake at high temperatures, and low conductivity at low humidity (Mirfarsi et al., 2020; Nimir et al., 2023; Rowshanzamir et al., 2015; Zhang et al., 2008). Among the polymers used in PEMs, many studies have been done with sulfonated poly (ether) ketone (SPEEK) polymer with fillers, nanoparticles, and other polymers/acids because of its better performance. The proton conductivity of a blended hybrid membrane with SPEEK, sulfonated polystyrene (SPS), and PWA (soluble hydrophilic additives) was improved by 10^{-1} S/cm. However, all observations were carried out in

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conditions of 100% relative humidity and higher temperatures (60 ^{0}C) (Moorthy et al., 2023a; Munavalli et al., 2018a, 2018b). A novel Cross-linked Sulfonated poly-arylene ether ketone (SPAEK) hybrid membrane with a metal-organic framework showed an enhanced proton conductivity of 0.184 S/cm at 80 ^{0}C (Gao et al., 2018; Guo et al., 2021; Moorthy et al., 2023b; Nayak et al., 2023). In another study, phosphoric acid doped blend hybrid membrane (sulfonated polyimide (SPI) and polybenzimidazole (PBI) showed proton conductivity of around 0.5 S/cm at 120 ^{0}C and 45% relative humidity (RH) (Ali et al., 2024; Hooshyari et al., 2020; Maiti et al., 2022; Mohamad Nor et al., 2022). However, the measurement of conductivity was done at higher temperatures, and in a humid environment, having complex fabrication procedures. At higher temperatures, the protons' movement increased, and ultimately, it showed enhanced proton conductivity. It is found that till now, the fabricated and reported membranes in the literature showed 10^{-1} S/cm conductivity either at higher temperatures or in humid environments (Bose et al., 2011).

In this present work, it is challenging to synthesize a proton conductive membrane having a low cost, an easier fabrication process, and high proton conductivity under dry conditions. Polystyrene, one of the low-cost materials, can be used as a proton conductive membrane, which is mixed with Phosphorus Pentoxide (P_2O_5). In literature, it is demonstrated that the phosphoric acid-infused membrane exhibited a significantly enhanced proton conductivity, achieving values nearly two orders of magnitude higher than those of undoped membranes. This considerable increase highlights the efficacy of phosphoric acid as a dopant in facilitating proton transport, suggesting its potential for applications requiring high proton conductivity (Yue et al., 2016a, 2016c, 2016b). Therefore, In the current work, a modified sulfonated polystyrene membrane is made using P_2O_5, and it is anticipated that, under dry or low humidity circumstances, it will create a new proton transport channel between the phosphoric acid and sulfuric acid As a result, these Proton-conductive membranes offer improved fuel cell efficiency and durability by enabling effective proton transport even in low-temperature, dry conditions, reducing the need for complex humidification systems. This makes them ideal for compact and portable applications, as well as environmentally friendly by minimizing water consumption and system complexity (Shalaby et al., 2014).

2. Experimental procedure

2.1 Materials

Polystyrene (PS) (molar mass: one repeating unit of styrene (C_8H_8) 104.1 g/mol; density: 1.04 g/cm³), N-Methyl-2-Pyrrolidone (NMP; 99% purity), concentrated sulfuric acid (H_2SO_4; 96% purity), and phosphorus pentoxide (P_2O_5; molar mass: 283.889 g/mol; density: 2.39 g/cm³) were procured from Jonaki Logical Co. Ltd., an authorized importer of these chemicals from Germany.

2.2 Preparation of Homogeneous Solution and Casting

Pure PS and P_2O_5 were mixed for fabricating the proton conductive membrane by solution-casting method. In the first step, a mechanical stirrer is used to prepare the homogenous PS-P_2O_5 solution. PS and P_2O_5 were dissolved separately in NMP at approximately 95 ^{0}C and 60 ^{0}C temperatures, respectively, where PS and P_2O_5 were taken in a ratio of 1:10 by weight percentage for fabricating 10 wt.% membranes. Then these two solutions were stirred together in one mixture. P_2O_5 solution in NMP took almost 20 minutes to be soluble, while the PS solution needed around 1 hour to get a clear homogenous solution. When both of the solutions were stirred together, the temperature was kept fixed at around 100 ^{0}C and the mixing rpm was maintained at 400-500 rpm. In the second step, after preparing the solution, the pure and modified PS membranes were cast on a glass plate at room temperature. Approximately 15 ml of solution was cast for one membrane to get the desired thickness. Then the casted solution was left at room temperature overnight to solidify completely. Similarly, another two different modified membranes with P_2O_5 5 wt.%, and 20 wt.% were fabricated in this work.

2.3 Characterization

A Fourier transform infrared spectrometer (FTIR) is used to characterize different functional groups. Here infrared specification was obtained considering transmittance and frequencies in the range of 500 cm^{-1}.-4000 cm^{-1}.

The proton conductivity of membranes was measured by the electrochemical impedance spectroscopy technique throughout a frequency range of 1.0 Hz–1.0 MHz with a wavering voltage of 110 mV –210 mV within the particular conductivity cell. In that cell, the membrane was placed between two stainless-steel cathodes. It is an established method to measure the proton conductivity of the PEM membrane. All impedance calculations were performed at room temperature. The membrane resistance (R) was calculated by capturing the impedance bend with the real axis at the high-frequency endpoint, which is obtained from a Nyquist plot. The proton conductivity was calculated according to the Eqn. (1)

$$\sigma = t / RA \quad (1)$$

Where σ is proton conductivity, R is membrane resistance, t is the thickness of the membranes and A is the cross-sectional area (Ahmad et al., 2006; Sherafat et al., 2015).

The scanning electron microscope (SEM) is used to analyze the morphological microstructure of the membrane. The samples were not coated with conducting material.

Mechanical characteristics of the membranes were determined using an Electro-Mechanical tensile testing machine. Gauge lengths of the sample were taken at 25 mm, while speed had been maintained at 5 mm/min.

X-ray diffraction (XRD) has been used to identify the phases present in the material and determine its crystallographic structure, which provides valuable information about crystal lattice parameters and crystallinity of the polymer.

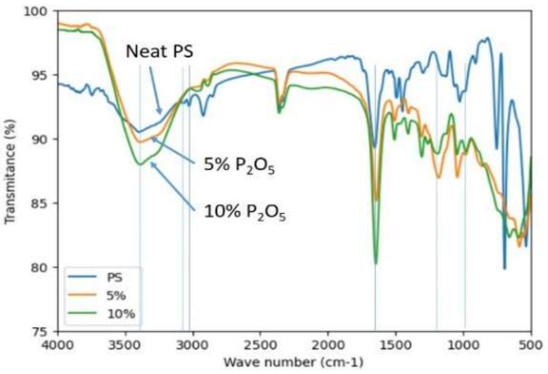

Figure 1: FTIR spectra for (a) Pure PS, (b) 5 wt.% P_2O_5 and (c) 10 wt.% P_2O_5.

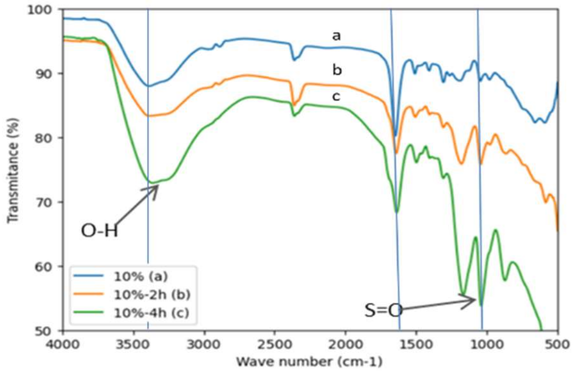

Figure 2: FTIR spectra of (a) 10 wt.% P_2O_5 membrane without sulfonation and (b) 10 wt.% P_2O_5 membrane with 2 hr. sulfonation, (c) 10 wt.% P_2O_5 membrane with 4 hours sulfonation.

3. RESULT AND DISCUSSION

3.1 Spectral characterization

FTIR investigation was conducted to assure the appearance of phosphorus pentoxide in the polymer matrix and to confirm the sulfonation. Underlying spectra of Polystyrene and 5 wt. %, 10 wt. % loaded Phosphorus pentoxide is shown in Figure 1. In pure PS, there are absorption peaks at the wave numbers of 3070 cm^{-1} and 3040 cm^{-1} due to CH_2 and aromatic C-H stretching vibration absorption (Naumi et al., 2018). Compared to pure PS, these two peaks become weak and disappear with increasing P_2O_5 content in the modified membrane. It indicated that the interaction between P_2O_5 and PS changed the structure of the PS polymer matrix. A new peak in the spectra of modified membranes at 1000 cm^{-1} and 1200 cm^{-1} can be attributed to the stretching that is symmetric of P-O groups on P_2O_5 (Lee et al., 2006). A higher pick is found at 1705 cm^{-1}, which can be described as P_2O_5 stretching vibration of the (C=O) carbonyl group, perhaps, because of the association among P_2O_5 and polymer matrix, in addition to, construction of compound membrane (De Bonis et al., 2014; Mecheri et al., 2008). The presence of -OH groups is demonstrated by the large peak at 3349 cm^{-1} – 3400 cm^{-1}. However, a larger proportion of phosphoric acid, which changed the amounts of intermolecular interaction of hydrogen bonds, caused the peak of -OH groups to widen from a pristine PS membrane to a membrane that had been modified with P_2O_5 (Li et al., 2024; Misenan et al., 2025; Mohanta et al., 2016; Naumi et al., 2018; Nguyen & Park, 2023).

Figure 2 shows the FTIR spectra for 10 wt.% P_2O_5 without sulfonation and 10 wt.% P_2O_5 for 2, 4 hours sulfonated modified membranes. It is already shown in Fig. 1 that a bigger absorption band was found around 3427 cm^{-1}. The presence of this broadband observed at 3427 cm^{-1} (merged with phosphoric acid intermolecular interaction) is assigned to the O–H stretching vibration of sulfonic acid groups. SPS was successfully completed, as evidenced by the stretching vibrational bands associated to the sulfonate groups that appeared at 1120 cm^{-1} (asymmetric O=S=O), 1082 cm^{-1} (symmetric O=S=O), and 850 cm^{-1} (S-O) stretching (Kowsari et al., 2015).

3.2 Proton Conductivity

Figure 3 presents the conductance of protons in the membrane at room temperature as a result of P_2O_5

concentration, which was measured in an anhydrous state. The proton conductivity of PS membranes increases up to 10 wt.% P_2O_5 indicates that there is a tendency for increasing proton conductivity with a corresponding P_2O_5 percentage increase. However, after that, further loading of P_2O_5 causes a decrease in proton conductivity, perhaps due to the dense concentration of protons in the membrane structure, which inhibits the movement of protons easily (Han et al., 2024). The maximum proton conductivity of 10^{-2} S/cm was found for the 10 wt.% P_2O_5 modified membranes, measures in fully anhydrous condition (Bose et al., 2011).

A Nyquist plot is shown in Figure 4 for 10 wt.% membranes. Typically, A standard Nyquist plot features a complete semicircle in the high-frequency region, indicating charge transfer resistance and double-layer capacitance, while the low-frequency region often displays an inclined line associated with diffusion processes or Warburg impedance. In this study, however, an incomplete semicircle is observed, likely due to structural variations in the polymer films, such as differences in surface roughness and thickness. These inconsistencies contribute to non-ideal impedance behavior and the formation of an inclined line, reflecting the complexities of ion transport dynamics within the membranes (Mohanta et al., 2016). If the incomplete semicircle is extrapolated, the real axis value represents the membrane resistance, R; while in this case it was found at least below 5Ω. So the proton conductivity was calculated by taking 5Ω resistance for 10 wt.% modified membranes.

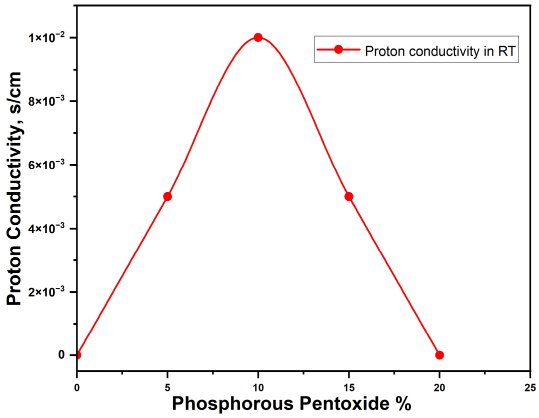

Figure 3: Proton conductivity curve concerning P_2O_5 loading.

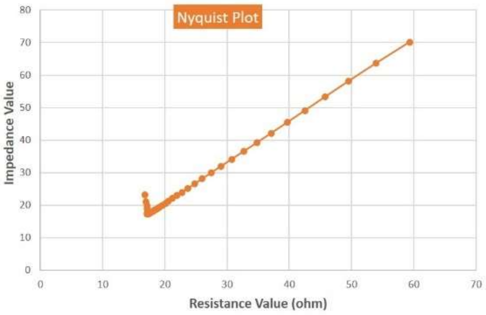

Figure 4: Nyquist plot for 10 wt.% P_2O_5 membrane for proton conductivity.

3.3 Mechanical characteristics

Figure 5 displays the stress-strain curve of the modified membranes. Membrane with 10 wt.% P_2O_5 has an excellent strength of approximately 1.5 MPa with 4.3% elongation at breaking point, where two consecutive curves of similar samples give the same strength. On top of that sulfonated one has a prominent strength of just about 1.8 MPa along with an elongation of 3.3%.

3.4 SEM Investigation

SEM was carried out to analyze the surface morphology of the membrane. Figure 6 depicts the images of the pure PS and 10 wt.% P_2O_5 membrane. The possession of space for the inorganic compound P_2O_5 led to small pores on the polymer matrix; these porous structures enhance the proton conducting path. Figure 7 indicates that sulfonated 10 wt.% P_2O_5 membranes have smaller pores. However, FTIR and proton conductivity analysis show a greater proton conductivity of the sulphonated membranes. So, from SEM images, it can be concluded that

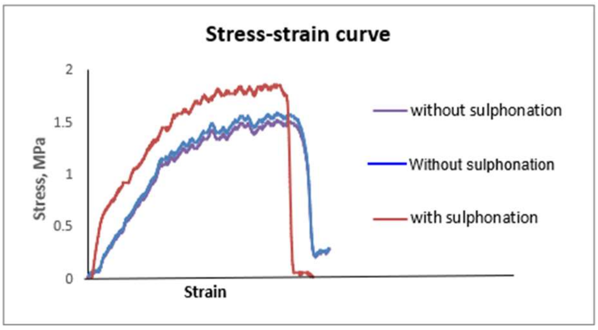

Figure 5: Stress-strain curve with and without sulfonation of 10 wt.% P_2O_5 membrane.

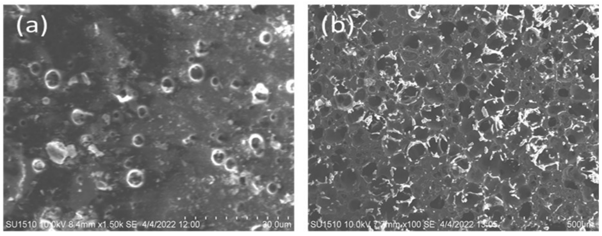

Figure 6: SEM images of (a) pure PS, (b) 10 wt.% P_2O_5 modified membranes.

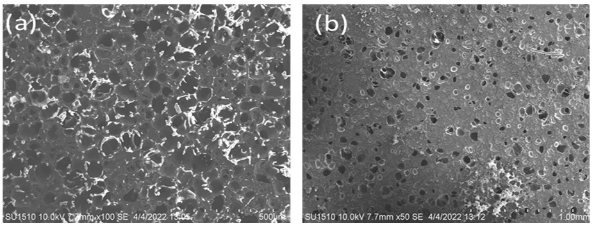

Figure 7: SEM images of (a) 10 wt.% P_2O_5 without sulfonation, (b) 10 wt.% P_2O_5 sulfonated membranes.

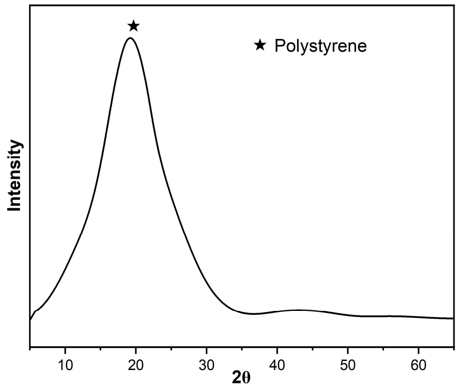

Figure 8: XRD pattern of 10 wt.% P_2O_5 doped polystyrene membrane.

doping with P_2O_5 and sulfonation makes the membranes porous. The sulfonation may increase the protons available to conduct, where the sulfonation is confirmed in FTIR analysis. The pore sizes are approximately 50 microns for both with sulfonated and without sulfonated membranes.

3.5 Phase composition of P_2O_5 doped polystyrene membrane

Figure 8 shows the X-ray diffraction (XRD) pattern of 10 wt.% P_2O_5-doped polystyrene, with intensity as a function of the diffraction angle 2θ. A broad peak around 20° 2θ, marked by a star, represents the primary diffraction characteristic of polystyrene, indicating its amorphous or semi-crystalline structure. The absence of distinct peaks from P_2O_5 suggests that the dopant is well-dispersed within the polymer matrix, without forming separate crystalline phases. This homogeneous distribution of P_2O_5 within polystyrene highlights the effective integration of the dopant, which enhances the material's structural uniformity.

4. CONCLUSION

This study described an effective and highly proton-conductive membrane based on PS and P_2O_5. Different membranes are fabricated with varying amounts of P_2O_5 concerning PS weight and are sulfonated in 20% diluted sulfuric acid to integrate with the sulfonated group in the PS network. The FTIR test confirmed the sulfonation of the -SO_3H group, which provides H^+ ions. The 10 wt.% P_2O_5 modified membrane showed the highest proton conductivity of about 10^{-2} s/cm. However, further increasing the amount of P_2O_5, for example, in the 20 wt.% membrane, more ions blocked the ions' movement path, which eventually reduced the conductivity. SEM images showed the porous structure, which confirmed the basis for higher proton conductivity. Under dry conditions, the measured proton conductivity of 10^{-2} S/cm for membranes containing 10 wt.% indicates their potential as effective solid electrolyte membranes for fuel cell applications. This level of conductivity is significant, suggesting that these membranes can facilitate ion transport efficiently, thereby enhancing the overall performance of fuel cells.

The outcome can be concluded as :

- FTIR spectra confirmed the incorporation of P_2O_5 in polystyrene, with new peaks indicating successful sulfonation and changes in hydroxyl interactions.
- Maximum proton conductivity of 10^{-2} S/cm was achieved at 10 wt.% P_2O_5 in anhydrous conditions, while decreased conductivity observed at higher concentrations due to ionic crowding.
- The Nyquist plot indicated incomplete semicircles, reflecting non-ideal impedance behavior and a membrane resistance below 5 ohms for the 10 wt.% P_2O_5 membranes.
- The 10 wt.% P_2O_5 membranes showed a tensile strength of about 1.5 MPa and an elongation at break of 4.3%, with enhanced strength but reduced flexibility in sulfonated membranes.
- SEM analysis revealed porous structures in 10 wt.% P_2O_5 membranes, beneficial for proton conduction, while sulfonation improved proton availability despite smaller pore sizes.
- The XRD pattern of P_2O_5-doped polystyrene shows a broad peak at 20° 2θ, indicating an amorphous structure with the dopant well-dispersed in the matrix.

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