



Membrane Electrode Assembly Based on Sulfonated Polysulfone-Activated Zeolite Composite Membrane for Fuel Cell Applications

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Abstract

The depletion of fuel cell reserves shows the need for alternative energy sources. In this context, fuel cells provide a promising solution, generating power without emissions. The membrane electrode assembly (MEA) in fuel cells is a critical component to maximize combustion efficiency. Therefore, this study aims to develop MEA using a composite membrane based on sulfonated polysulfone-activated zeolite (PSfs-Z). The membrane was obtained by first sulfonating polysulfone (PSf) and then adding activated zeolite. Optimum fuel performance was achieved with the activated zeolite using 3 N HCl. Sulfonation was completed, as demonstrated by a sulfonation degree of 32.63%. The absorption bands of the SO₂ group vibration from SO₃H of PSfs were identified at a wave number of 1111.00 and 1259.33 cm⁻¹. The successful formation of MEA was confirmed by its morphology, as observed using a scanning electron microscope, with the formation of a catalyst layer and a membrane in the cross-section. The highest proton conductivity and cell potential obtained for PSfs-Z 3 N MEA were 0.01727 S.cm⁻¹ and 330 mV, respectively. This means the membrane electrode assembly based on a sulfonated polysulfone-activated zeolite composite membrane has a higher potential than the sulfonated zeolite ZSM-5 composite membrane for fuel cell applications.

1. Introduction

The dwindling reserves of fossil fuels are necessitating a future energy crisis as humanity's needs grow and become increasingly diverse. This situation has led to extensive study into developing various alternative energy sources. Fuel cells are renewable electrochemical cells that convert chemical energy directly into electrical energy [1] without generating harmful emissions, such as carbon dioxide (CO₂) and carbon monoxide (CO).

The membrane widely used in fuel cells is a fluoropolymer membrane with branched chains containing sulfonate groups, known by the trade name Nafion. This membrane has a high proton conductivity of 0.082 S.cm⁻¹ at 80°C and 99.9% relative humidity. Nafion also has good chemical stability and a high methanol crossover (methanol permeability), but its proton conductivity decreases above 80°C [2]. Therefore, sulfonated polysulfone (PSfs) is believed to be an

alternative membrane to replace Nafion, which is prepared by sulfonation of polysulfone (PSf). According to a previous study, PSf is an aromatic polymer with decent oxidative and thermal stability, with a high glass transition temperature of up to 170°C [3]. The presence of sulfonate groups in PSf facilitates polymer charging, increasing membrane conductivity for fuel cell applications [4]. The charge amount depends on the number of sulfonate groups in the PSf framework. However, as more sulfonate groups are introduced into PSfs, mechanical strength decreases sharply, and at certain levels of sulfonation, the polymer becomes water-soluble. This drastic decrease in mechanical strength limits the sulfonation level of PSf [5]. Based on this explanation, improving the performance of aromatic polymer membranes with sulfonation is often followed by the addition of supporting materials to form a composite [4].

In this research, the formation of a composite membrane was carried out by adding zeolite to PSfs because the structure of zeolite contains large free spaces as nanoporous materials and shows the ability to desorb and absorb water in large amounts [6]. These properties are essential in fuel cell applications. However, there is an aspect of zeolite composition, namely extra-framework cations such as K^+ , Na^+ , Ca^{2+} , and Mg^{2+} [7], which may interfere with fuel cell applications, requiring zeolite activation to remove these contaminants. Activated zeolite (Z) can be obtained by heating or by adding acid or base. Zeolite activation using HCl could remove impurities that clog pores and increase the surface area [8].

In previous research, sulfonated polysulfone-zeolite ZSM-5 composite membranes at room temperature were indicated to have a proton conductivity of $0.00965 S \cdot cm^{-1}$ [9]. Fuel cell applications require an electrolyte membrane with high hydrophilicity to enable efficient proton flow. Furthermore, Fuel cell performance in terms of proton conductivity could be improved by combining a polymer electrolyte and catalyst composite membrane into a single unit, known as a membrane electrode assembly (MEA). The MEA is a core part of the fuel cell in increasing proton conductivity and accelerating electrochemical reactions [10]. Meanwhile, the hydrophilicity/hydrophobicity of zeolite depends on the SiO_2/Al_2O_3 ratio. The higher the SiO_2/Al_2O_3 or Si/Al ratio in zeolite, the lower the hydrophilicity of the zeolite. The activated zeolite has SiO_2/Al_2O_3 ratio of 7.66 [11], whereas zeolite ZSM-5 has a Si/Al ratio of 23 [9]. Therefore, MEA based on sulfonated polysulfone-activated zeolite composite membrane has a higher proton conductivity potential than sulfonated zeolite ZSM-5 composite membrane for fuel cell applications.

2. Experimental

2.1. Tools and Materials

The materials used in this study included PSf (Sigma-Aldrich), 50-mesh zeolite from Sukabumi, sulfuric acid, fuming sulfuric acid containing 65% SO_3 (oleum), nitrogen gas, technical chloroform, dichloromethane, methanol, NaOH, HCl, $K_3[Fe(CN)_6]$ solution, Na_2HPO_4 solution, phenolphthalein, Ag/C catalyst, and deionized water. The tools used were glassware, an oven, a three-necked flask, compartments, a Cu electrode, a carbon electrode, an analytical balance, a multimeter, a JEOL JSM 836 OLA SEM (Geological Survey Center), a Bruker Tensor 27 FTIR (National Nuclear Technology Agency (BATAN)), a DTG-60 Shimadzu (BATAN), and an LCR-meter impedance analyzer (Membrane Biophysics Laboratory, Department of Physics, Faculty of Mathematics and Natural Sciences, Bogor Agricultural University).

2.2. Synthesis of Sulfonated Polysulfone (PSfs)

A total of 10 g PSf was dissolved in chloroform to obtain 10% (w/v) PSf solution. This was followed by the gradual addition of 20 mL of oleum to a separating funnel connected to a three-necked flask, through which nitrogen gas was flowing. SO_3 gas from oleum was pushed

into a PSf solution under nitrogen, which was heated to $40^\circ C$ and stirred with a mechanical stirrer. The synthesis of PSfs was carried out for 60 minutes in a fume hood.

2.3. Determination of Degree of Sulfonation (DS)

The 0.1 g of PSfs membrane was weighed and soaked in 10 mL of 1 N NaOH for 3 days. The remaining NaOH was then titrated with 1 N HCl standardized to $Na_2B_4O_7$ using 3 drops of phenolphthalein indicator to determine the endpoint. The titration was carried out until the color changed from pink to colorless, and the DS value was obtained using Equation 1.

$$DS = \frac{(V_{initial} - V_{final}) \times N_{HCl} \times BE_{SO_3}}{\text{Sample weight}} \times 100\% \quad (1)$$

Where, $V_{initial}$ is the volume of HCl used for the blank (mL), V_{final} is the volume of HCl used for the sample (mL), N_{HCl} is the normality of HCl (N), and BE_{SO_3} is the equivalent weight ($g \cdot eq^{-1}$).

2.4. Activation of Natural Zeolite with Acid Treatment

A total of 5 g zeolite was dissolved in 20 mL of 1 N, 3 N, and 5 N HCl, and heated for 24 hours at $90^\circ C$ while stirring using a magnetic stirrer. It was then filtered and washed with deionized water until chloride ions were removed. The zeolite was dried at $110^\circ C$ for 3 hours and heated at $450^\circ C$ for 3 hours to remove residual solvent.

2.5. Sulfonated Polysulfone-Activated Zeolite Membrane Preparation

PSfs membrane was dissolved in dichloromethane using a stirrer. Subsequently, 5% of activated zeolite relative PSfs weight was added to the solution, stirred until homogeneous, and sonicated for 30 minutes. The obtained PSfs-Z solution was printed on a glass plate coated with adhesive tape on the edge (1 layer).

2.6. Membrane Activation

The membranes were soaked in H_2O_2 for 1 hour, then in H_2SO_4 for 1 hour. This process was followed by rinsing with deionized water.

2.7. Water Uptake Test

Activated and unactivated PSf and PSfs-Z membranes were each cut into $1 \times 1 \text{ cm}^2$ pieces, oven-dried at $100^\circ C$ for 2 hours, and weighed to determine the dry weight. The membranes were then immersed in deionized water at room temperature for 24 hours. The samples were cleaned with a tissue and weighed to determine the wet weight. The difference between the wet and dry weights was used to determine the water uptake. Each measurement was performed in duplicate. Water uptake was calculated using Equation (2).

$$\text{Water uptake} = \frac{\text{wet weight} - \text{dry weight}}{\text{dry weight}} \times 100\% \quad (2)$$

2.8. MEA Synthesis

The catalyst was synthesized by mixing 20% Ag/C catalyst ink with 5% membrane solution and chloroform, stirring until a paste formed. The membrane was then pressed with the catalyst onto carbon on both sides and molded using a hot press.

2.9. Membrane Proton Conductivity Measurement

The membrane was cut to the required electrode size, consisting of Cu metal. The electrodes were activated by immersion in 1 N HCl for 1 day, followed by immersion in 1 N NaOH for another day. After activation, the active electrodes were washed with deionized water and soaked until ready for use.

The areas of both activated and unactivated membranes were measured using a digital micrometer, with the electrode size (A) and thickness recorded. The membrane thickness was proportional to the distance between the two carbon electrodes (l). Furthermore, the conductance was measured by sandwiching the membrane between the two electrodes. These electrodes were connected to the LCR-meter's positive and negative terminals, which provided the membrane conductance (G). The value obtained was converted to conductivity per unit distance, called proton conductivity, using Equation 3.

$$\sigma = G \frac{l}{A} \quad (3)$$

Where, σ is the proton conductivity (S.cm^{-1}), A is the surface area (cm^2), l is the distance between the two electrodes (cm), and G is the conductance value (S).

2.10. Fuel Cell Application Test

The potential difference in the fuel cell system was measured across the anode and cathode compartments. Compartment A, the anode system, was filled with 160 mL of 0.3 M methanol solution, while compartment B, the cathode system, was filled with 80 mL of 1 mM $\text{K}_3\text{Fe}(\text{CN})_6$ solution and 80 mL of Na_2HPO_4 solution. Membranes were glued to the center of the two vessels, and electrodes were immersed in both solutions, which were then connected to the positive and negative poles. The potential difference was measured with a voltmeter.

3. Results and Discussion

3.1. Sulfonated Polysulfone (PSfs)

PSfs membranes were obtained by adding oleum (SO_3) as a sulfonating agent into polysulfone (PSf). Sulfonating agents are chemical compounds that play a role in replacing C-H bonds with SO_3H groups through a chemical modification of the membrane. The presence of sulfonic groups in PSf increased hydrophilicity and improved proton conductivity of its polymer membrane for a fuel cell. There are many compounds used as sulfonating agents, such as H_2SO_4 [12], chlorosulfonic acid [13], chlorosulfonic acid, and trimethylchlorosilane [14].

The use of chloroform as PSf solvent was based on the principle of "like dissolves like." PSfs membranes were synthesized with the aid of nitrogen gas, which acted as a propellant for SO_3 to enter the PSf solution. Nitrogen gas was known to be inert and did not react with PSf or oleum. Heating at 40°C for 60 minutes during sulfonation was used to make the mixture more homogeneous and accelerate the reaction, as shown by a color change from colorless (Figure 1a) to brownish-yellow (Figure 1b) after sulfonation.

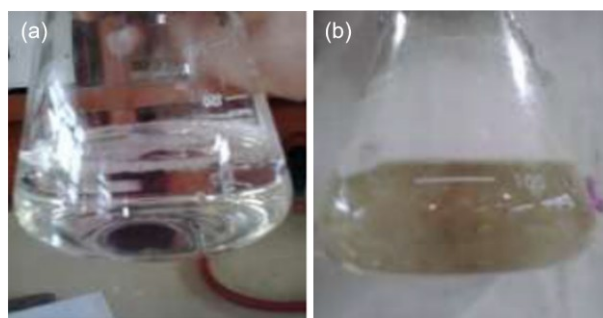


Figure 1. Color changes (a) before, and (b) after sulfonation of the PSf solution

The success of sulfonation was shown by the DS value. The greater the DS value, the more sulfonate groups were substituted into PSf, as shown in Figure 2. High DS of the composite polymer membrane improves the performance of the fuel cell by enhancing proton conductivity [15]. The DS was determined by acid-base titration, in which the membrane was immersed in NaOH solution, which caused sulfonate groups to bind Na^+ ions. The remaining Na^+ that was not bound to the membrane was titrated with hydrochloric acid. Furthermore, the difference in the volume of hydrochloric acid used for NaOH titration on the blank and the sample was used to determine DS. PSf membrane, which had a DS of 9.21% increased to 32.63% after sulfonation to form PSfs.

The sulfonate group on the aromatic ring of PSfs indicated successful sulfonation. Based on the FTIR results, an increase in the absorption bands was observed at 1111.00 and 1259.53 cm^{-1} (Figure 3), indicating vibration of the SO_2 group from SO_3H . According to Padmavathi *et al.* [16], PSfs exhibit absorption bands of the SO_2 group vibration in SO_3H at 1110 and 1350 cm^{-1} . Additionally, the SO_2 symmetric stretching band appears at 1101 and 1167 cm^{-1} [14]. The presence of absorption bands at wavenumbers 1774.51 , 1903.74 , and 2040.69 cm^{-1} indicates the presence of substituted groups on the PSf aromatic ring at positions 1, 2, 4- [17].

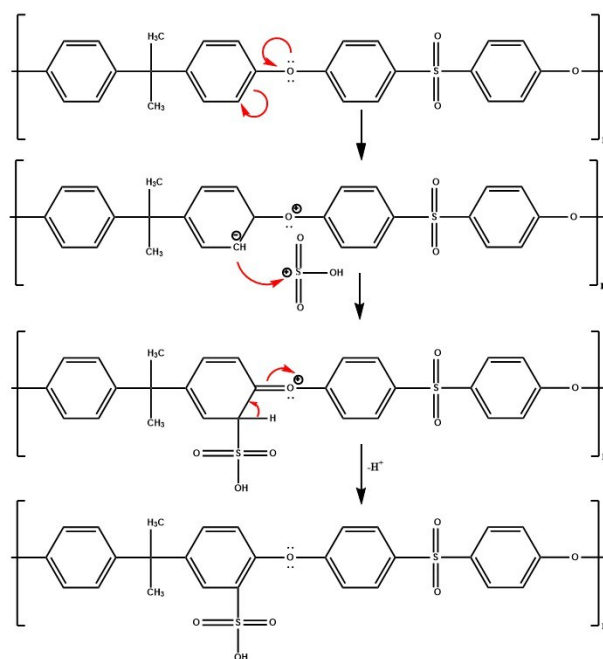


Figure 2. Sulfonation reaction in PSf

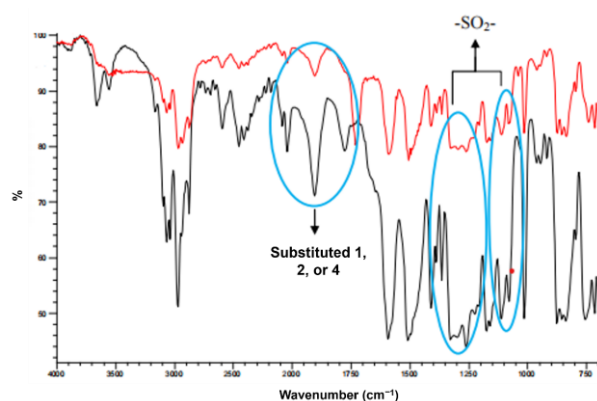


Figure 3. Spectra of PSf and PSfs

3.2. Water Uptake

Water uptake measures the amount of water absorbed by the membrane. The water molecules function in acid dissociation and facilitate proton transport [18]. The higher the water uptake in the membrane, the better the proton flow. The analysis results indicate that the PSfs-Z 3 N composite membrane had the highest water uptake among the membranes (Figure 4). Furthermore, activation of the PSfs-Z 3 N composite membrane with H_2O_2 and H_2SO_4 could increase water uptake to 14%.

Increasing the concentration of HCl, the zeolite activating agent, to 3 N increased the water uptake of the PSfs-Z composite membrane. This improvement is attributed to acid treatment removing pore-blocking impurities and increasing the zeolite surface area, thereby enhancing its ability to absorb water and increasing the hydrophilicity of the composite membrane [19]. The hydrophilicity/hydrophobicity of zeolites depends on the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio. The zeolite framework consists of SiO_4 and AlO_4 tetrahedral linked by shared oxygen atoms. In acidic solutions, Al–O bonds are less stable; thus, aluminum is readily removed from the framework (dealumination), forming extra-framework Al species and H^+ . This process increases the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio, meaning more Si–O–Si bonds relative to Si–O–Al bonds. Because Si–O–Al bonds are more polar than Si–O–Si bonds, a lower proportion of Si–O–Al bonds corresponds to higher hydrophobicity of the zeolite [11]. In this study, excessive dealumination during activation with HCl concentrations above 3 N is indicated by the decreased water uptake of the PSfs-Z 5 N composite membrane.

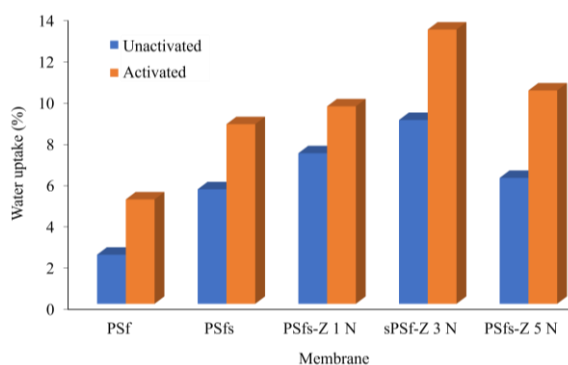


Figure 4. Water uptake of unactivated and activated membranes

3.3. Thermal Stability

Thermal stability analysis of PSfs and PSfs-Z 3 N membranes was carried out with 30°C as the starting point (Figure 5). The thermogram similarity PSfs and PSfs-Z 3 N membranes indicate three weight-loss steps: between 30 and 180°C due to the loss of water and residual solvent; between 180 and 400°C attributed to splitting off sulfonic groups; and thermal degradation above 500°C due to decomposition of the PSf backbone. The data presented for PSf are consistent with previous work [9]. Furthermore, the presence of zeolite in PSf slightly reduces decomposition of the PSf backbone, as shown by lower weight loss in N PSfs-Z 3 N compared to PSfs.

3.4. Proton Conductivity and Morphology

The proton conductivity of a membrane reflected its ability to conduct protons. High proton conductivity indicates a good fuel cell. PSfs-Z 3 N composite membrane in the form of MEA exhibited the highest proton conductivity $0.01727 \text{ S}\cdot\text{cm}^{-1}$ compared to membranes (unactivated and activated) $0.00247 \text{ S}\cdot\text{cm}^{-1}$ and $0.00265 \text{ S}\cdot\text{cm}^{-1}$, respectively (Figure 6). This is due to interfaces in the membrane electrode assembly between neighboring layers, and on both the anodic and cathodic side, namely proton exchange membrane/catalyst layer (PEM/CL), catalyst layer/microporous layer (CL/MPL), and microporous layer/carbon fiber paper (MPL/CFP). These interfaces in MEA play an important role in fuel cell performance and durability, relating their function to mechanical adhesion, mass transport, charge transfer, and heat conduction [20].

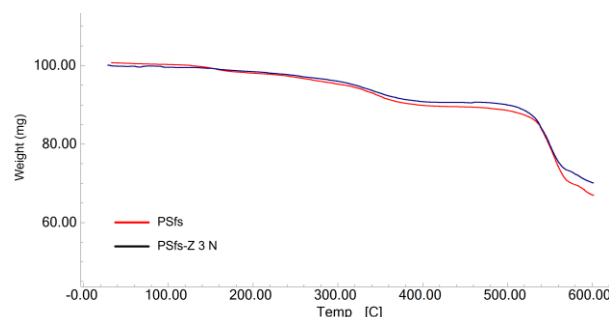


Figure 5. Thermogram of PSfs and PSfs-Z 3 N membranes

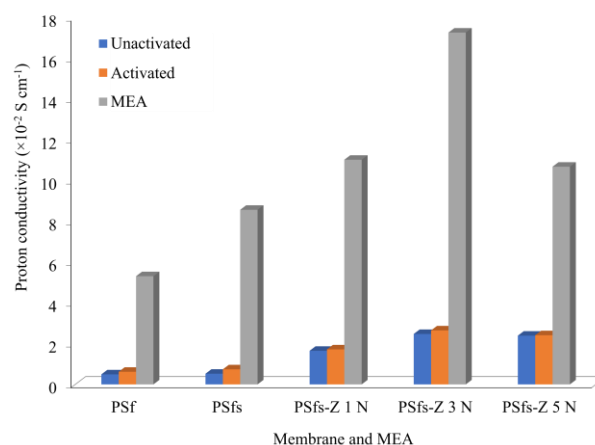
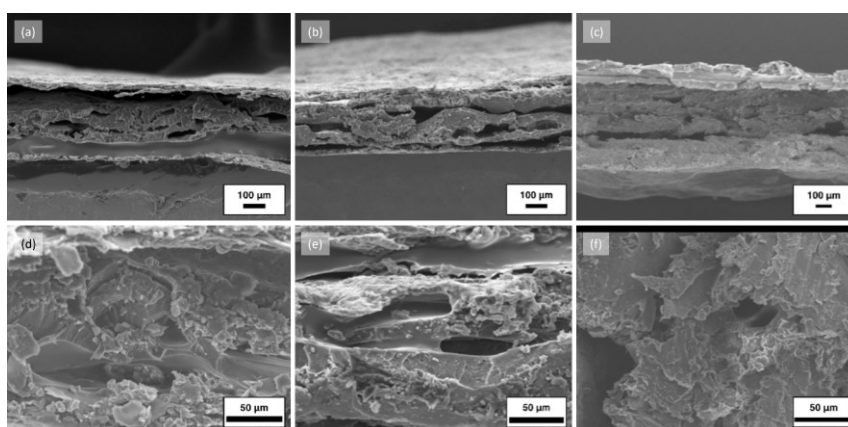


Figure 6. Proton conductivity of the unactivated, activated membrane, and MEA

Table 1. Sulfonated polysulfone (SPSU or PSfs) and Nafion composite membranes for fuel cell (DMFC)

MEA	Membrane characteristic			
	Water uptake (%)	Proton conductivity (S.cm ⁻¹)	Catalyst	Reference
SPSU/NIMs-GO composite	34.1	0.23 @ 75°C	Pt/C	[21]
SPSU-LDH composite	31	0.0137 @ 120°C	Pt/C	[22]
SPSU-LDH nanocomposite	29	0.102 @ 120°C	Pt-Ru/C (anode) and Pt/C (cathode)	[23]
SPSU/ZrP	38	0.158 @ 80°C	Pt-Ru/C (anode) and Pt/C (cathode)	[24]
SPSU/Sulfonated ZSM-5	45.41	0.00965 @ RT	-	[9]
PSfs-activated zeolite	14	0.01727 @ 25°C	Ag/C	This Research
Nafion/SiO ₂	29.23	0.172 @ 110°C	-	[25]

**Figure 7.** SEM images ($\times 100$) of the cross-section of (a) PSf, (b) PSfs, and (C) PSfs-Z 3 N (c); SEM images ($\times 500$) of the surface of (d) PSf, (e) PSfs, and (f) PSfs-Z 3 N

The success of MEA fabrication was proven by the formation of the catalyst and membrane layers in a single unit. The catalyst used in this research was Ag/C, a substitute for Pt, which is relatively cheaper. In addition, the Ag catalyst has a standard cell potential (+ 0.80 V) that is 1 level below that of the Pt catalyst (+1.50 V). Thus, the use of Ag catalysts in MEA is expected to sufficiently increase proton conductivity for fuel cell applications.

Based on cross-section SEM images of PSf (Figure 7a), PSfs (Figure 7b), and PSfs-Z 3 N (Figure 7c), the presence of the top and bottom layers was identified as catalyst layers, while the middle layer was the membrane. However, there is delamination of the membrane/catalyst interfaces on PSf and PSfs membranes. The most crucial adhesion is between the membrane and the catalyst in MEA for proton transport. Adhesion improvement can be achieved by introducing interlocking or porous surfaces, such as zeolite on PSfs-Z 3 N composite membrane. Meanwhile, SEM images show that sulfonation of the PSf membrane (Figure 7d) does not affect the morphology of PSfs (Figure 7e), while the presence of zeolite in PSfs can reduce the formation of gaps/voids (Figure 7f).

A membrane electrode assembly based on sulfonated polysulfone-activated zeolite using 3 N HCl provides lower proton conductivity than other MEAs of sulfonated polysulfone or Nafion composite membranes, except for the LDH composite and sulfonated zeolite ZSM-5 (Table 1). The analysis results indicate that the proton conductivity of the composite membrane is affected by

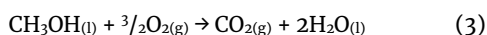
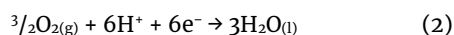
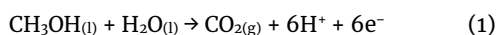
water uptake, catalyst, and operating temperature. In addition, there is a difference in the SiO₂/Al₂O₃ or Si/Al ratio in activated zeolite (SiO₂/Al₂O₃ ratio of 7.66) [11] compared to ZSM-5 zeolite (Si/Al ratio of 23) with a thickness of 0.013 and 0.023 cm [9], respectively. This indicates that activated zeolite has greater hydrophilicity and proton conductivity than ZSM-5, making it more advantageous as a composite material for membranes in fuel cell applications.

3.5. Fuel Cell (DMFC) Application

The fuel cell application test used a direct methanol fuel cell (DMFC), with the MEA enclosed in two compartments. The anode (compartment A) contained methanol as fuel, while the cathode (B) had a potassium ferricyanide solution as an indicator and a phosphate buffer. DMFC operates by converting methanol directly into electrical energy via a chemical reaction with a membrane serving as a selective barrier. Methanol oxidation at the anode produced CO₂ gas, protons, and electrons. CO₂ gas was released from the system, protons moved through the membrane, and reacted with oxygen to produce water. At the same time, electrons flowed to the cathode, causing a cell potential difference. At the cathode, Fe³⁺ from potassium ferricyanide was reduced to Fe²⁺ by the flow of electrons from the anode, evidenced by the appearance of a greenish-yellow color. The anodic oxidation of methanol is described in Reaction (1), while the cathodic process is shown in Reaction (2). The overall cell reaction is given in Reaction (3).

Table 2. Proton conductivity and cell potential of PSf, PSfs, and PSfs-Z in membrane electrode assemblies for fuel cell (DMFC) applications

MEA	Proton conductivity (S.cm ⁻¹)	Cell potential (mV)
PSf	0.00531	51
PSfs	0.00857	54
PSfs-Z 1 N	0.01102	202
PSfs-Z 3 N	0.01727	330
PSfs-Z 5 N	0.01068	230



The results of MEA application tests in fuel cells (DMFC) showed that the use of a composite membrane based on PSfs and activated zeolite using 3 N HCl provided an optimal cell potential of 330 mV, corresponding to maximum proton conductivity (Table 2). Zeolite, as a composite in PSfs membranes, significantly increased proton flow.

4. Conclusion

A membrane electrode assembly based on sulfonated polysulfone-activated zeolite (PSfs-Z) composite membrane shows higher potential than sulfonated zeolite ZSM-5 composite membrane for fuel cell applications, with proton conductivity and cell potential of 0.01727 S.cm⁻¹ and 330 mV, respectively. The performance of the fuel cell is influenced by water uptake of 14% and a sulfonation degree of 32.63% for sulfonated polysulfone, with sulfonate groups identified at 1111.00 and 1259.53 cm⁻¹. The presence of activated zeolite using 3 N HCl as a porous composite can increase water uptake, and it has good thermal stability.

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