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Development of Bacterial Cellulose Membrane from Sulfation SCOBY Kombucha for Polymer Electrolyte Membrane Fuel Cell (PEMFC) Applications

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Abstract

Electrolyte membranes are critical components in determining the performance of PEMFC systems. The use of commercial membranes remains constrained by high production costs, thereby necessitating the development of biomaterial-based alternatives. Cellulose derived from SCOBY Kombucha presents a promising candidate for electrolyte membranes, yet its intrinsic proton conductivity is insufficient and requires chemical enhancement. This study aimed to optimize the sulfation process to improve the electrochemical properties of SCOBY-based cellulose membranes. Variations in reaction time and sulfuric acid concentration were investigated, followed by characterization of the modified membranes through proton conductivity, ion-exchange capacity, swelling degree, mechanical properties, thermal stability, FTIR, and SEM-EDX analyses. The optimum condition was achieved at 2 hours and 2 M, yielding a proton conductivity of 5.44×10^{-2} S/cm^{-1}, an ion-exchange capacity of 3.58 meq/g, and a swelling degree of 111%, while exhibiting moderate mechanical strength and good thermal stability. FTIR and SEM-EDX confirmed successful incorporation of sulfonic acid groups. Overall, the modification produced a membrane with enhanced proton conductivity and ion-exchange capacity, although further mechanical reinforcement is required for practical PEMFC applications.

1. Introduction

A fuel cell is an electrochemical device that directly converts chemical energy into electrical energy. A fuel cell operates based on the principle of electrochemical reactions, for example, hydrogen reacting with an oxidizer such as oxygen. The reaction between the fuel and oxygen produces water [1]. Based on the type of electrolyte used, fuel cells are classified into liquid- and solid-type cells. Solid fuel cells are more widely developed due to their more practical packaging. A popular type of fuel cell on the market is the Polymer Electrolyte Membrane Fuel Cell (PEMFC), which uses a polymer membrane as the electrolyte to conduct protons from the anode to the cathode [2].

The most commonly used electrolyte membrane for PEMFCs is Nafion. The advantages of Nafion membranes include high proton conductivity and good thermal and chemical stability, enabling them to operate effectively at high temperatures and in acidic environments commonly found in fuel cells [3]. Nevertheless, Nafion membranes have several limitations, including high production costs and limited environmental friendliness. Therefore, new polymer materials are needed that can be used as electrolyte membranes with relatively lower production costs and better environmental compatibility.

One material with potential as a polymer electrolyte membrane for fuel cells is bacterial cellulose, which is derived from renewable biological sources and can form uniform, thin films with relatively good mechanical strength [4]. In addition, its hydrophilic nature facilitates water retention, which is necessary for proton conduction. However, high hydrophilicity can also lead to excessive swelling. Although increased water uptake can accelerate proton transport via the Grotthuss and vehicle mechanisms, this condition may weaken hydrogen bonding between cellulose chains, thereby reducing the membrane's mechanical stability. On the other hand, bacterial cellulose naturally exhibits low proton conductivity due to the limited number of proton-carrying ionic groups in its structure. Therefore, chemical modification is required to improve its electrochemical performance. One effective method is sulfuric acid modification, which introduces strong acid groups onto the cellulose surface. In treatment with H_2SO_4, the dominant groups formed are generally sulfate ester groups ($-O-SO_3H$) attached to the cellulose chains. These fixed charge groups act as proton donor sites that can increase the ion exchange capacity and improve the membrane's proton conductivity. Cellulose modified with sulfuric acid contains sulfate ester groups ($-O-SO_3H$) that function as proton-conducting sites [5].

Cellulose membranes developed include nata de banana, nata de pina, and nata de cassava. Compared with laboratory-cultured bacterial cellulose, SCOBY kombucha is more readily obtained as a fermentation by-product and does not require a tightly controlled microbial cultivation process. The utilization of SCOBY also supports the development of sustainable materials by converting fermentation waste into value-added biomaterials. In addition, SCOBY has a dense and naturally interconnected nanofibril structure, making it a promising raw material for membrane fabrication. Therefore, in this study, the cellulose membrane used was derived from the symbiotic culture of bacteria and yeast (SCOBY) as a sustainable and environmentally friendly electrolyte membrane. SCOBY appears as a thick, rubbery disc, white to brownish in color [6].

Although the base material originates from renewable biological sources, the chemical modification process through sulfation involves the use of strong acids, which may pose environmental impacts if not properly managed. Nevertheless, compared with the production of fluoropolymer-based membranes such as Nafion, which requires complex petrochemical-based synthesis processes, the use of bacterial cellulose remains a more sustainable approach. In the future, the development of more environmentally friendly sulfation methods may become a direction for further research to enhance the overall sustainability of this material. Thus, in this study, the sulfonated bacterial cellulose membrane derived from SCOBY kombucha is expected to be utilized as an electrolyte membrane in PEMFC applications. The success of this research is anticipated to provide a promising alternative membrane material for PEMFC, contributing to the development of sustainable energy technologies. Membrane technology offers significant advantages compared with conventional methods, including low cost, energy efficiency, and ease of operation.

To date, research on the use of bacterial cellulose from SCOBY kombucha as an electrolyte membrane in PEMFC remains limited, particularly in studies that optimize sulfation conditions while balancing proton conductivity, degree of swelling, and mechanical strength. Recent studies report that properly functionalized cellulose-based proton exchange membranes can achieve high proton conductivity under fully hydrated conditions. However, such systems generally require more complex modification routes or the addition of conductive fillers, and they still face trade-offs among conductivity, degree of swelling, and mechanical stability. Therefore, the development of simpler and more scalable modification strategies for bacterial cellulose, particularly that derived from SCOBY kombucha, remains an important research focus. Accordingly, this study aims to optimize the sulfation time and concentration of SCOBY kombucha–based bacterial cellulose membranes to achieve a balance between proton conductivity, degree of swelling, and mechanical strength. This study is expected to provide a simple and potentially scalable modification approach for the development of more sustainable biomaterial-based electrolyte membranes [7].

2. Experimental

The methods used in this study include the preparation of cellulose membranes from SCOBY obtained through kombucha fermentation, the sulfation of the membranes with sulfuric acid at varying sulfation times and acid concentrations to determine the optimal conditions, and the characterization of the sulfonated membranes. Membrane characterization was performed by evaluating proton conductivity via Electrochemical Impedance Spectroscopy (EIS), ion-exchange capacity, swelling degree, mechanical properties, thermal stability, functional group analysis by FTIR spectroscopy, and surface morphology and elemental composition by SEM–EDX.

2.1. Materials

Bacterial cellulose membranes were obtained from SCOBY produced through kombucha fermentation. The materials used included black tea, sucrose, starter kombucha, distilled water, NaOH, CH_3COOH, H_2SO_4, and HCl. All chemicals were of analytical grade and used without further purification.

2.2. Preparation for Cellulose Membrane

SCOBY was cultivated in a sterile sweetened tea medium consisting of 2 g black tea and 40 g sucrose dissolved in 500 mL of distilled water. After cooling, 200 mL starter kombucha and one SCOBY layer were added, followed by fermentation at room temperature for 6–7 days. The resulting bacterial cellulose was washed with boiling water for 15 minutes, treated with 1% (w/v) NaOH for 24 hours to remove impurities, then with 1% (w/w) CH_3COOH for 24 hours, and rinsed with distilled water until neutral pH. The purified cellulose was first dried at room temperature to remove residual moisture. The dried material was then pressed at 100 bar for 5 minutes using a hydraulic press to form dense membranes. After pressing, the membranes were further dried at room temperature to ensure complete removal of residual moisture before subsequent characterization. The resulting membranes had an average thickness of approximately 0.01 cm (≈100 μm), measured using a digital micrometer [8].

2.3. Sulfation Process and Optimization

Sulfation was carried out by immersing the cellulose membranes in H_2SO_4 solution. Optimization was performed by varying (i) sulfation time, 2, 4, 6, and 8 hours using 0.18 M H_2SO_4, and (ii) H_2SO_4 concentration, 1.0, 1.5, 2.0, and 2.5 M at the optimum sulfation time. The optimization of the sulfation process was conducted using a one-variable-at-a-time (OVAT) approach, following the method reported by Rahmawati *et al.* [7]. In this approach, sulfation time was first optimized at a relatively low sulfuric acid concentration (0.18 M) to minimize rapid acid-induced degradation of the cellulose structure and to observe gradual changes in proton conductivity. After identifying the optimum sulfation time (2 hours), this duration was fixed for subsequent evaluation of sulfuric acid concentration effects. The investigated concentration range (1.0–2.5 M) falls within commonly reported sulfation conditions for cellulose-based membranes, allowing independent assessment of the influence of concentration on membrane properties. After treatment, the membranes were heated in an oven at 105°C for 150 seconds, washed thoroughly with distilled water to remove residual acid, and dried at room temperature. The optimum condition was determined based on the highest proton conductivity [7].

2.4. Characterization of Sulfonated Membranes

2.4.1. Proton conductivity

Proton conductivity was evaluated using Electrochemical Impedance Spectroscopy (EIS) to determine the ionic transport behavior of the membranes. Measurements were carried out at room temperature over a frequency range of 20 Hz–2 MHz. The bulk membrane resistance (R) was obtained from the high-frequency intercept of the Nyquist plot. Proton conductivity (σ) was calculated using the measured membrane thickness and the effective electrode area [7].

2.4.2. Ion Exchange Capacity (IEC)

Ion exchange capacity was measured to quantify the amount of sulfonic acid groups introduced into the cellulose matrix. The membranes were first converted to the protonated form by immersion in 0.1 M HCl, then neutralized and exchanged with 0.01 M NaOH. After ion exchange, a measured aliquot of the NaOH solution was back-titrated with standardized H_2SO_4 to determine the remaining NaOH concentration, and the IEC value was calculated based on the total solution volume [7].

2.4.3. Swelling Degree

The swelling behavior of the membranes was examined to assess their water-uptake capacity and hydrophilicity. Dry membranes were immersed in distilled water for 24 hours until equilibrium was reached. The swelling degree was calculated from the mass difference before and after immersion, providing insight into membrane hydration characteristics [7].

2.4.4. Mechanical Properties

Mechanical performance was analyzed using a Universal Testing Machine (UTM) to determine tensile strength and elongation at break. Measurements were conducted at room temperature with a crosshead speed of 10 mm/minute. The obtained stress–strain curves were used to evaluate the mechanical stability of the sulfonated membranes.

2.4.5. Thermal Stability

Thermal behavior was investigated by Thermogravimetric Analysis (TGA) to evaluate mass loss and degradation stages of the membranes. The analysis was performed under a nitrogen atmosphere from 25 to 600°C at a heating rate of 10°C/minute, allowing assessment of thermal resistance and decomposition characteristics [9].

2.4.6. Functional group analysis

Chemical structural changes before and after sulfation were analyzed using Fourier Transform Infrared (FTIR) spectroscopy. Spectra were recorded in the range of 4000–500 cm^{-1} to identify characteristic absorption bands associated with cellulose and sulfonic acid groups [10].

2.4.7. Surface Morphology and Elemental Composition

Surface morphology and microstructural features of the membranes were observed using Scanning Electron Microscopy (SEM). Elemental composition and sulfur distribution were analyzed by Energy Dispersive X-ray spectroscopy (EDX) to confirm the successful incorporation of sulfonic acid groups within the membrane structure [10].

3. Results and Discussion

This section presents the results obtained from the sulfation of SCOBY-based cellulose membranes and discusses the effects of sulfation time and sulfuric acid concentration on the properties of the resulting membranes. The discussion focuses on the relationship between structural and chemical modifications and membrane performance, as evaluated through proton conductivity, ion exchange capacity, swelling behavior, mechanical properties, thermal stability, functional group analysis, and surface morphology.

3.1. Effect of Sulfation Time on Proton Conductivity

The effect of sulfation time on the electrochemical properties of the cellulose membranes was evaluated using EIS. Nyquist plots obtained for membranes sulfonated for 2, 4, 6, and 8 hours show a clear variation in membrane resistance, indicating changes in proton transport behavior. The membrane sulfonated for 2 hours exhibited the lowest resistance (1.56 Ω) and the highest proton conductivity (1.54×10^{-2} S/cm^{-1}). Due to limitations in manual membrane casting, achieving identical thickness was challenging; therefore, the membranes prepared in this study had thicknesses ranging from 0.04 to 0.07 cm. Each membrane thickness was individually measured and used in the conductivity calculation to ensure accurate comparison among samples.

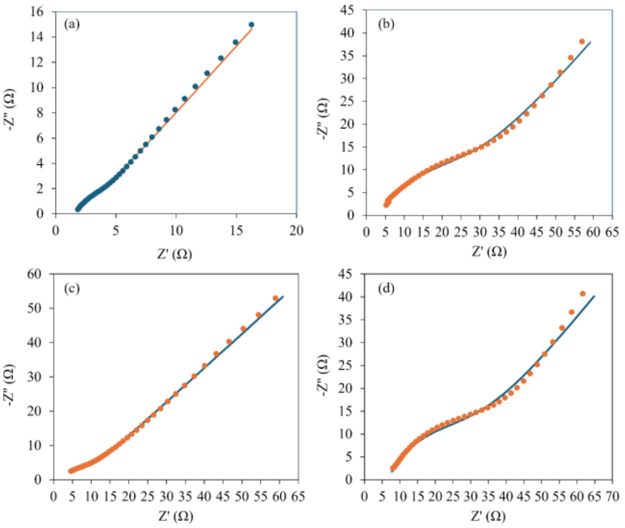

Figure 1. Nyquist curve based on EIS test with time 2 hours, (b) 4 hours, (c) 6 hours, and (d) 8 hours

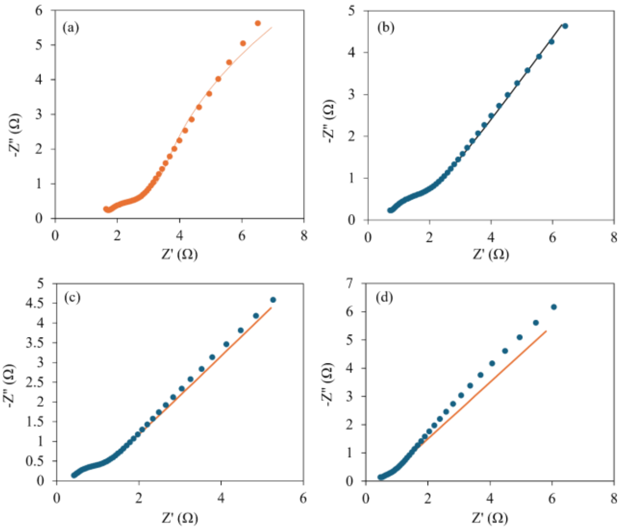

Figure 2. Nyquist curve based on EIS test with concentration (a) 1 M, (b) 1.5 M, (c) 2 M, and (d) 2.5 M

Increasing the sulfation time beyond 2 hours resulted in a gradual increase in membrane resistance and a corresponding decrease in proton conductivity. Prolonged exposure to sulfuric acid may induce structural weakening of the cellulose matrix and disrupt intermolecular hydrogen bonding, leading to discontinuity in proton transport pathways. These results indicate that a sulfation time of 2 hours provides sufficient incorporation of sulfonic acid groups while maintaining structural integrity of the membrane [11, 12].

3.2. Effect of Sulfuric Acid Concentration

The influence of sulfuric acid concentration on membrane performance was investigated by varying H_2SO_4 concentration from 1.0 to 2.5 M at the optimum sulfation time. As shown by the EIS results, membrane resistance decreased with increasing acid concentration up to 2.0 M, resulting in a maximum proton conductivity of 5.44×10^{-2} S/cm^{-1}.

The enhancement in conductivity at moderate acid concentration can be attributed to the increased density of sulfonic acid groups ($-SO_3H$), which serve as proton-donating sites and facilitate proton transport through both the vehicle and Grotthuss mechanisms. However, at 2.5 M, although the high-frequency intercept slightly decreased, the calculated proton conductivity declined due to increased membrane swelling and thickness variation, which influence the effective proton transport pathway. Excessive water uptake at higher acid concentration may increase structural heterogeneity and disturb continuous proton conduction channels. Therefore, 2.0 M H_2SO_4 was identified as the optimum concentration for membrane sulfation [13, 14].

3.3. Ion Exchange Capacity

IEC was measured to quantify the amount of sulfonic acid groups introduced into the cellulose membrane. The unsulfonated membrane exhibited a relatively low IEC value of 1.06 meq g^{-1}, indicating the minimal presence of acidic functional groups within the native cellulose structure. After sulfation under optimum conditions, the IEC significantly increased to 3.58 meq/g, confirming the successful incorporation of sulfonic acid groups into the cellulose matrix. The increase in IEC directly correlates with the observed enhancement in proton conductivity, as a higher density of acidic sites promotes efficient proton transfer within the membrane structure [15].

Table 1. Ion exchange capacity of cellulose membrane

Sample		Replicate 1	Replicate 2	Mean ± SD
Ion exchange capacity (meq/g)	Before sulfation	0.9532	1.1644	1.0588±0.1493
	After sulfation	3.5702	3.5969	3.5836±0.0189

Table 2. The swelling degree of the cellulose membrane

Sample		Replicate 1	Replicate 2	Mean ± SD
Swelling degree (%)	Before sulfation	71.43	63.16	67.29±5.85
	After sulfation	110.71	111.54	111.12±0.59

3.4. Swelling degree

The swelling degree of the membranes was evaluated to assess their hydrophilicity and water uptake behavior. The pristine cellulose membrane exhibited a swelling degree of approximately 67%, whereas the sulfonated membrane showed a substantial increase to 111%. This increase is attributed to the introduction of hydrophilic sulfonic acid groups, which enhance water absorption via hydrogen bonding. Increased water uptake facilitates proton transport by providing continuous hydration pathways, thereby enhancing proton mobility through both vehicle and Grotthuss mechanisms. However, excessive swelling may reduce intermolecular hydrogen bonding within the cellulose matrix, leading to decreased tensile strength and partial structural loosening. Moreover, excessive water uptake can dilute the fixed charge density of sulfonic acid groups, potentially limiting efficient proton conduction at higher levels of functionalization. Therefore, the swelling degree obtained under optimum sulfation conditions reflects a favorable balance between hydration-mediated conductivity enhancement and structural integrity of the membrane [16].

3.5. Mechanical Properties

Mechanical properties of the sulfonated membrane were evaluated by tensile testing. The membrane exhibited a tensile strength of 3.21 MPa with an elongation at break of 4.16%. For comparison, commercial Nafion membranes typically exhibit tensile strength values ranging from 20–35 MPa. The reduction in mechanical strength compared to pristine cellulose can be attributed to partial disruption of hydrogen bonding and increased amorphous regions caused by sulfation. However, the tensile strength of 3.21 MPa with an elongation at break of 4.16% remains relatively low compared to commercial Nafion membranes, indicating that further mechanical reinforcement or cross-linking strategies are required to enhance mechanical robustness for practical PEMFC applications [17].

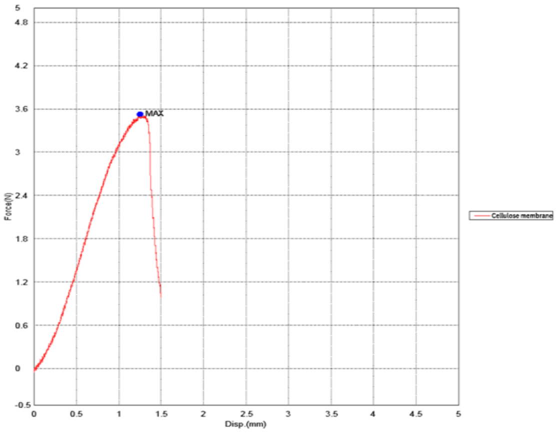

Figure 3. Sulfonated cellulose membrane tensile test curve

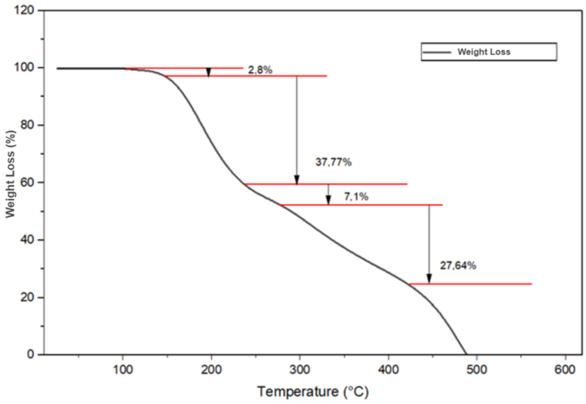

Figure 4. TGA curve sulphonose membrane

3.6. Thermal Stability

The thermal stability of the sulfonated cellulose membrane was investigated using TGA. The TGA curve revealed an initial weight loss below 150°C, corresponding to the evaporation of physically bound and absorbed water. A second stage around 240°C is attributed to decomposition of sulfonic acid groups, followed by degradation of the cellulose backbone at higher temperatures. The membrane exhibited thermal stability up to approximately 240°C, exceeding the typical operating range of proton exchange membrane fuel cells (60–120°C), indicating suitability for electrochemical applications. However, mechanical softening may occur at lower temperatures due to plasticization by water and sulfonic acid groups [18, 19].

3.7. FTIR Analysis

FTIR spectroscopy was employed to identify chemical structural changes resulting from sulfation. The spectra of sulfonated membranes showed characteristic cellulose absorption bands, including O–H stretching and C–O–C vibrations. In addition, increased absorption intensity in the region of 1000–1300 cm^{-1} was observed, corresponding to the overlapping vibrations of the S=O and C–O groups. The enhancement of these bands confirms the successful introduction of sulfonic acid groups into the cellulose structure without significant alteration of the cellulose backbone [20, 21].

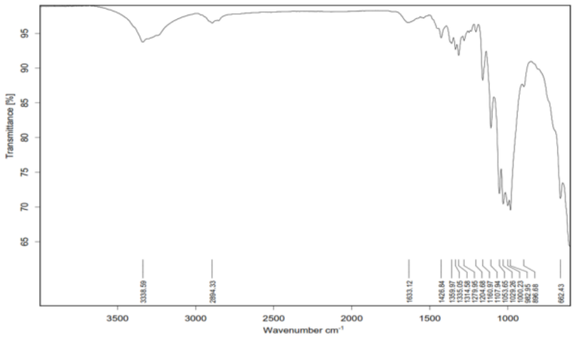

Figure 5. Infrared spectrum of cellulose membrane before sulfation

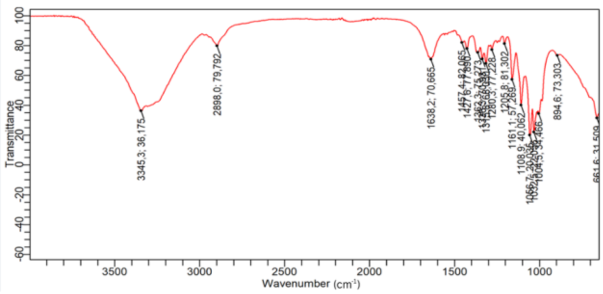

Figure 6. FTIR spectrum of the sulfurized cellulose membrane

Table 3. FTIR-ATR of the cellulose membrane

Sample	Wavenumber (cm⁻¹)	Functional group
Cellulose membrane (before sulfation)	3338.59	O–H stretching
	1160.97	C–O–C stretching
	1029.26	C–O stretching (primary alcohol)
Cellulose membrane (after sulfation)	3345.28	O–H stretching
	1161.06	C–O–C stretching
	1427.57	C–O–S vibration
	1362.34	S–O stretching
	1336.25	S=O stretching (sulfonate)
	1056.70	C–O stretching (primary alcohol)

3.8. SEM–EDX Analysis

Surface morphology analysis by SEM at magnifications of 5,000× and 15,000× revealed that the sulfonated membrane possessed a porous and rough surface with irregular pore distribution. Such morphological features are favorable for water retention and proton transport [22].

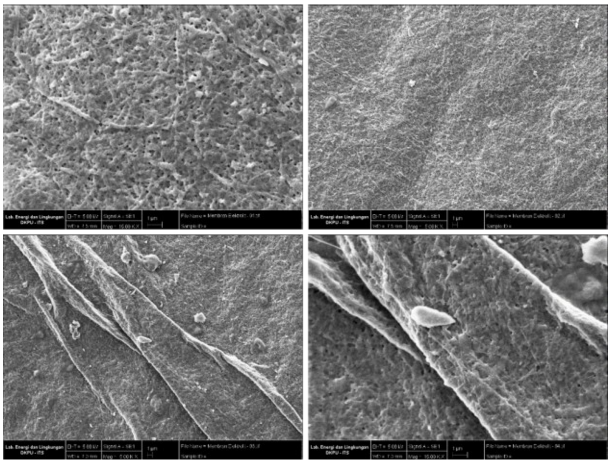

Figure 7. Sulfodated cellulose membrane surface SEM test results

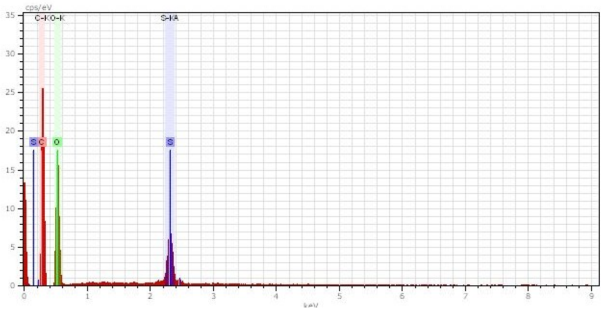

Figure 8. Sulfur content of sulfolate cellulose membrane EDX test results

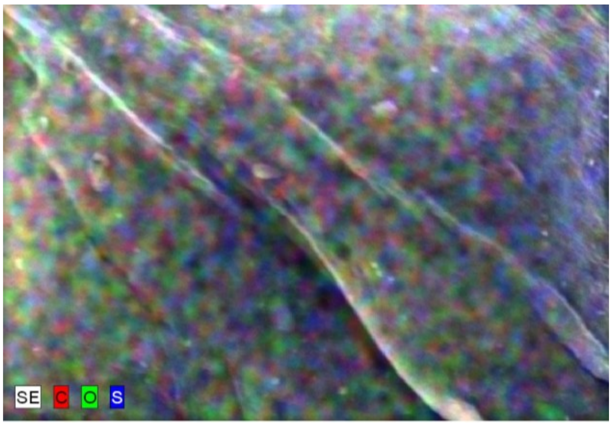

Figure 9. Distribution of carbon, oxygen, and sulfur on the membrane

EDX analysis confirmed the presence of sulfur uniformly distributed on the membrane surface, with a sulfur content of approximately 8.04 wt%, providing direct evidence of successful sulfation. The sulfur content detected by EDX represents surface composition, whereas IEC reflects bulk ionizable group density. Therefore, slight discrepancies may arise due to surface sensitivity limitations of EDX analysis. The combined SEM–EDX results demonstrate that sulfation modifies both the chemical composition and microstructure of the cellulose membrane, contributing to improved proton conduction performance [23].

4. Conclusion

Sulfonated cellulose membranes derived from SCOBY kombucha were successfully prepared and evaluated as proton-conducting membranes, with the sulfation process significantly influencing their physicochemical, mechanical, and electrochemical properties. The optimum sulfation condition was achieved at a sulfation time of 2 hours and a sulfuric acid concentration of 2.0 M, resulting in the highest proton conductivity of 5.44×10^{-2} S/cm^{-1}, which was attributed to the successful incorporation of sulfonic acid ($-SO_3H$) groups into the cellulose structure as confirmed by ion exchange capacity measurements, FTIR analysis, and SEM–EDX results; However, excessive sulfation time or higher acid concentration led to structural alteration and excessive swelling, which negatively affected membrane performance. The sulfonated membranes exhibited improved ion-exchange capacity and water uptake; however, their mechanical strength remains lower than that of commercial PEM membranes and requires further improvement. Despite this limitation, the membranes demonstrated adequate thermal stability within the typical operating temperature range of proton exchange membrane systems, indicating their potential as sustainable proton exchange membranes for electrochemical applications. The most critical next step is evaluating single-cell PEMFC performance and long-term durability under wet–dry cycling conditions.

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