**Optimization of Acid-Activated Zeolite as a PES Membrane Filler for Microbial Fuel Cell Application**

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**Abstract*.***Membranes are a crucial component of Microbial Fuel Cell (MFC) systems, responsible for proton transport and maintaining a viable bacterial environment. Mixed Matrix Membrane (MMM) combining polymer and zeolite offers a promising, more effective, and cost-friendly alternative MFC process compared to conventional MFC membranes. This study investigates the effect of acid concentration used to activate Zeolite as a filler for polyethersulfone (PES) membranes. Zeolite-Y that is used was activated using varying concentration of hydrochloric acid (HCl) as the concentration of acid that is used to activate zeolite can affect its proton transfer performance and overall structural stability. The effect of acid activation studied using X-Ray Diffraction (XRD) and confirm that acid-activated zeolite alters the initial zeolite structure by dealumination process, shows in change of peaks intensity in the diffractogram. Furthermore, excessive acid concentration will result in collapse of zeolite structure. The fabricated MMMs were characterized by water contact angle (WCA) measurements, and evaluated for their MFC performance, in voltage, current, and overall power density. All membranes show higher water contact compared to neat PES membrane, although still classified as hydrophilic. The performance evaluation revealed that MMMs containing fillers activated using higher acid concentration exhibit lower current, voltage, and overall power generation. Overall, acid-activated zeolite shows suitability as a membrane filler for MFC applications. The MMM with 1 M Acid-Activated Zeolite (PES/AZ1) demonstrated the best overall MFC performance, meanwhile MMMs with inactivated zeolite-Y (PES/ZY) shows best long-term stability. This research explores the effects of zeolite activation process and its effects as a filler to optimize membrane for energy generation through MFC process.

**Keywords:** Acid-Activated Zeolite, Microbial Fuel Cell, Proton Exchange Membrane, PES Membrane, Proton Conductivity

# INTRODUCTION

In 2021, 71% of Indonesia's electricity was mainly produced by fossil fuels, which, in the process of generating electricity, emits excess CO2, which is a greenhouse gas that we need to reduce to achieve the Net Zero Emissions 2060 target. While 23% of the electricity is mainly produced by renewables, it is primarily sourced from hydropower coming from river streams [1,2]. At the same time, many rivers and water sources are polluted by untreated waste from homes and industries. The generation of energy from renewable sources with wastewater treatment can resolve the energy and water issues that we currently face. Microbial Fuel Cell (MFC) is a technology that generates electricity with the help of microbial activity that degrades organic waste, which makes this technology very attractive to solve these issues as it produces electricity with near-zero emissions, low temperature, and it's a simple design [3–6].

MFC consists of anode and cathode chambers that are separated by a selective barrier, mostly a membrane. Membrane setups are used to prevent oxygen transfer from cathode to anode, which often happens in a membrane-less setup. The membrane will maintain an electric current and enhance proton conduction. Meanwhile, the anode chamber is the part where microbial actions take place, which will generate electrons and protons through an oxidation reaction. The electrons that result from the reaction are transported to the cathode chamber through an external circuit, whereas the membrane will transport the protons. In the cathodes, both species combine oxygen and hydrogen ions, resulting in pure water production [7–10]. Membranes that are used in MFC can be categorized into two types, which are cation or anion exchange membranes. Both membranes are polymer-based, which is known for its mechanical strength. MFCs using proton exchange membranes are mainly used because of their capability to generate high conductivity, which anion exchange membrane lacks. Most commercially available membranes for MFC applications are called Nafion, which is a cation exchange membrane that transports protons through negative charge functional groups that are available in its polymer matrix. Unfortunately, Nafion’s capability is often hindered by biofouling, high cost, low proton conductivity at high temperature, excessive swelling in wet conditions, and poor ion-selectivity, which makes an alternative membrane needed for MFC applications [11].

Modifying Nafion’s membranes to make composite or mixed matrix membranes is a common practice to increase MFC performance. Unfortunately, due to its initial cost, much attention has been paid to polymer-based mixed matrix membranes to reduce the cost. One of the polymers that can be used is polyether sulfone (PES). This polymer has excellent mechanical stability, cost-efficient, great film-forming capacity, and is resistant towards many hydrocarbons’ compounds [12]. Unfortunately, its ion conductivity is quite low, due to SO2 groups that are in the chains, which do not form free ions, although it’s an electron-withdrawing group. To enhance it, sulfonating its sulfone chain is the most effective route to take. Unfortunately, reagents used for this process, such as fuming sulfuric acid and sulfuric trioxide, are highly toxic. Furthermore, this process will compromise the mechanical and chemical strength of polyether sulfone [13–16]. Introducing inorganic materials that have specific properties into the polymer to make mixed matrix membrane is a safer and viable option to fabricate membrane for MFC applications.

Inorganic materials can provide continuous phase that can elevate proton conductivity and water sorption in the MMMs. Materials such as metal oxide (SiO2, TiO2), zeolite, and zirconium phosphate have been introduced in MFC applications. Among various inorganic fillers, zeolites, as crystalline aluminosilicates with well-defined pore structures and high surface areas, stand out as particularly well-suited for PES-based MMMs. Their high ion-exchange capacity, thermal stability, and selective molecular sieving can significantly improve membrane characteristics crucial for MFCs, including proton conductivity, water retention, and anti-fouling capabilities. Specifically, Zeolite-Y, known for its FAU structure, large pore size (≈7.4 Å), and high surface area, offers enhanced water sorption and retention due to its inherent hydrophilicity and high cation exchange capacity [17–20]. These properties are critical for maintaining high proton conductivity and preventing membrane dehydration in MFCs.

However, the full potential of Zeolite-Y can be further unlocked through acid activation. This process, typically using strong acids like HCl, primarily aims to dealuminate the zeolite framework, which increases the silicon-to-aluminum (Si/Al) ratio. This controlled dealumination can fine-tune hydrophilicity for better polymer compatibility, generate Lewis acid sites, and most importantly, enlarge micropores or create mesopores, significantly improving mass transport within the zeolite [21,22]. Additionally, acid treatment removes impurities and facilitates the conversion of the zeolite into its protonated (H-form), forming Brønsted acid sites essential for proton conduction [17]. While aggressive activation can compromise structural integrity, a controlled approach enhances pore accessibility, surface acidity, and ultimately, the zeolite's contribution to membrane performance.

Therefore, this research will focus on activating Zeolite-Y using varying concentrations of HCl solution (1 M, 2 M, and 3 M) to optimize its properties. The activated Zeolite-Y will then be incorporated as fillers into polyethersulfone (PES) to fabricate novel mixed matrix membranes. Prior research focused on sulfonated polymer and the addition of sulfonate-based or sulfonate modified material into the membranes, the specific impact of simple acid activation process for MFC and its impact between zeolite and PES polymer interaction has not been thoroughly investigated. This study hypothesized that zeolite activation will alter zeolite-polymer interaction, which will influence the performance in terms of proton transport, power generation, and stability of the MMMs. These PES/activated Zeolite-Y MMMs will be thoroughly characterized and evaluated for their performance in Microbial Fuel Cell applications, aiming to develop a cost-effective, high-performance, and sustainable membrane solution for combined energy generation and wastewater treatment.

# EXPERIMENTAL

## Materials

Polyether sulfone (PES), n-methyl pyrrolidone (NMP), chloric acid (HCl, 37%), Deionized Water, Commercial zeolite-Y, ammonium chloride (NH4Cl), monosodium phosphate (NaH2PO4.2H2O), disodium phosphate (NaHPO4.H2O), potassium chloride (KCl), and Nutrient Broth (NB).

## Filler Preparation

5 grams of commercial Zeolite-Y were mixed into 100 mL of an HCl solution. This solution was stirred for 30 minutes at room temperature. Following stirring, the Zeolite-Y was separated from the HCl solution and thoroughly washed with deionized water until a stable pH was achieved. The resulting Acid-Activated Zeolite-Y was then dried in an oven at 60-80°C overnight. The HCl solutions used for activation were varied at 1 M, 2 M, and 3 M. Each obtained zeolite was designated as AZx, where 'x' represents the concentration (in M) of the HCl solution used for activation.

## Mixed Matrix Membrane Fabrication

Mixed Matrix Membranes (MMMs) are fabricated using the dry-wet phase inversion method. A 50-gram dope solution was prepared, primarily by dispersing 17% wt of polyether sulfone (PES) in 40 mL of NMP. Subsequently, 0.3% wt of AZ (acid-activated zeolite) was slowly dispersed into the NMP and mixed for 30 minutes using a magnetic stirrer. The mixture was then sonicated in a water bath sonicator for another 30 minutes. After sonication, the PES powder was slowly added to the filler-NMP solution and stirred for 24 hours at 80°C with 300 rpm. Following the 24-hour stirring period, the solution was left overnight to allow for bubble removal. The dope solution was then cast onto a glass plate to fabricate the MMM. The glass plate was subsequently soaked in water to detach the membrane. The obtained MMM was then soaked, first in water and then in an ethanol solution, overnight for each respective solvent. After soaking, the MMM was dried at room temperature for 2-3 days until all visible moisture had evaporated from its surface.

## Characterization

## The crystallinity of each acid-activated Zeolite-Y structure is identified using X-Ray Diffraction (XRD). The result is a diffractogram, which represents the intensity of X-rays scattered from the material at various diffraction angles. Meanwhile, the hydrophobicity of the membrane is determined by measuring its Water Contact Angle (WCA). This is typically performed using the sessile drop method, where a water droplet is carefully placed onto the membrane surface and the contact angle is measured. A membrane is classified as hydrophilic when its contact angle is less than 90 degrees.

## Fuel Cell Performance Test

Fuel Cell Performance is tested using a double-chamber microbial fuel cell (MFC) reactor with bacteria in the anode part, electrolyte solution in the cathode part, and carbon used as the electrode, which is connected by a multimeter. The bacteria are cultured by inoculating *Bacterium* *strain* BS2049 into 10 mL of sterile Nutrient Broth (NB). Incubation happens for 24 hours. Optical density (OD) was measured using UV-Vis Spectrophotometer in 600nm. The electrolyte solution is made by mixing 0,31 g of NH4Cl, 0,13 g of KCl, 4,79 g of NaH2PO4, and 2,75 g of Na2HPO4 with demineralized water in 1 L volumetric flask. The MMM is placed between the reactor and acts as a separator of both solutions in the anode and cathode parts of the chamber. Current and voltage are measured using a multimeter for 3 hours. The data obtained are used to calculate power density, with the equation below:

A close-up of a tester

Description automatically generated

**FIGURE 1.** Fuel Cell Performance Test using MFC Double Chamber Reactor

# RESULTS

## X-Ray Diffraction Result

The crystallinity and structural integrity of Zeolite-Y and its acid-activated counterparts are confirmed by the XRD diffractogram shown in Figure 2. Both Zeolite-Y and AZ1 exhibit well-defined and sharp peaks, indicating a highly crystalline structure in both materials, and proving that 1 M HCl activation does not significantly alter the Zeolite-Y framework. It is possible that low-concentration activation can increase due to the removal of out-of-framework and other amorphous substances, which could increase material crystallinity [23]. Although the intensity of its characteristic peak around 6° decreases, other peaks show more refined peaks than their non-activated counterpart. AZ2 still displays characteristic peaks, but its intensity decreases again, and peaks become broader. This suggests that 2 M HCl activation begins to induce structural changes in Zeolite-Y, possibly through dealumination, amorphization, and reduction in crystallite size. AZ3 shows significantly diminished peaks compared to the others, indicating that 3 M HCl activation causes severe alterations to the Zeolite-Y structure. These results collectively confirm that increasing the acid concentration during zeolite activation leads to a change in Zeolite-Y stable structure, possibly due to the removal of the framework aluminum [24,25]. Such structural changes in Zeolite-Y will consequently alter its pore network, surface area, and ion-exchange capabilities, all of which are crucial for the performance of MFC.

A graph of different colored lines

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**FIGURE 2.** XRD Diffractogram of Commercial Zeolite-Y and Activated Zeolite-Y

## Water Contact Angle Measurement

The water contact angles (WCAs) of MMMs are shown in Figure 3. A lower contact angle indicates higher hydrophilicity, meaning the membrane will exhibit better water permeation and wettability. The Neat PES membrane exhibits the lowest contact angle, signifying that among all fabricated membranes, neat PES is the most hydrophilic. This result is expected, as polyether sulfone (PES) is generally classified as a relatively hydrophilic polymer. The PES/ZY membrane shows higher contact angle than Neat PES, from 56.89°to 65.87°, indicating a slight decrease in hydrophilicity (or an increase in hydrophobicity) compared to the pure PES membrane. Acid activation of the zeolite generally makes the membrane more hydrophobic, up to 67.29° and 72.78° for PES/AZ1 and PES/AZ2, which exhibit higher contact angles than both Neat PES and PES/ZY. This increased hydrophobicity can be attributed to dealumination during acid treatment, which removes hydrophilic aluminum sites and induces pore structural changes, thereby affecting the interactions of zeolite with water [24]. Interestingly, the PES/AZ3 membrane shows a decreased contact angle compared to PES/AZ1 and PES/AZ2 which were down to 62.98°. Although its contact angle is still higher than that of Neat PES, it is lower than the other MMMs. This change in trend can be attributed to the severe structural changes of zeolite resulting from 3M HCl activation, as corroborated by the XRD results. The drastic alteration of the zeolite structure at this high acid concentration may expose new surface sites or lead to a different surface morphology that allows for better water interaction, despite the loss of crystallinity.

A graph of different sizes of pes and pes

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**FIGURE 3.** Water Contact Angle Measurement for Each Membrane

## Fuel Cell Performance

The voltage generated by the membranes exhibits varied trends, as depicted in Figure 4(a). Neat PES shows a significant increase for the first two hours, which jumps from 5.67 mV to 39.38 mV, then down to 33.46 mV for the final hour. PES/ZY demonstrates a consistent increase in voltage generation throughout the period, from 26 to 45 mV. Conversely, PES/AZ1's voltage initially increased to 56.5 mV, but then decreased for the last 2 hours, although its endpoint remains relatively similar to PES/ZY membrane. Meanwhile, PES/AZ2 shows a steady decrease in voltage, which started from 53.16 mV down to 31.64 mV. PES/AZ3 consistently generates low voltage for the entire duration, around 5 mV. In terms of voltage generation, PES/ZY exhibits a more consistent trend, but PES/AZ1 maintains a higher overall voltage generation throughout the experiment.

Figure 4(b), meanwhile, illustrates the current generated by the membranes over a 3-hour period. The Neat PES membrane shows a decreasing current over time, despite starting with the highest initial current around 32.5 mA/m2. In contrast, the Mixed Matrix Membranes (MMMs) generally exhibit an increasing current trend. Both PES/ZY and PES/AZ1 show relatively similar current levels around 20-23 mV. However, PES/ZY demonstrates a continuously increasing current from 13.25 to 22 mA/m2, whereas PES/AZ1's current increases for the first hour up to 25 mA/m2, then decreases slightly to 22.5 mA/m2 for the second hour and declines significantly to 2.5 mA/m2 in the final hour. PES/AZ2 displays a drastic increase in the second hour, from 2.5 to 20 mA/m2. Meanwhile, PES/AZ3 maintains a low and stable current flow around 1-2 mA/m2 throughout the entire duration. Among all tested membranes, PES/ZY exhibits the most stable current generation over the 3-hour period.

Adding fillers into the polymer matrix results in varied outcomes for both voltage and current generation but overall can increase the MFC performance compared to neat PES membrane [3,11]. Zeolite-Y significantly improves the MFC's performance compared to Neat PES and demonstrates notable stability. The porous structure and ion-exchange properties of Zeolite-Y contribute to this improvement in MFC performance. AZ1 shows promising initial current and voltage generation, but experiences setbacks due to its decline, particularly in current generation. This suggests that the acid-activation process creates highly active sites that may degrade quickly, potentially from collapsing pore structure, as the aluminum framework is removed, which potentially leads to an imbalance in ion transport [26]. AZ2 exhibits a similar trend to AZ1, but its overall performance is less than that of AZ1. In contrast, AZ3 consistently displays poor performance, which aligns with its XRD and Water Contact Angle (WCA) measurements, demonstrating that acid activation at 3 M is detrimental to the Zeolite-Y structure and, consequently, its MFC performance [27].

|  |  |
| --- | --- |
| **(a)** | **(b)** |

**FIGURE 4.** Voltage (a) and Current (b) Measured for 3 Hours

Power density represents the direct overall performance of the membrane, as it is derived from the product of current and voltage, and its trends can be observed in Figure 5. The Neat PES membrane exhibits a low power density at the start around 184 mW/m2 followed by a slight increase up to 787 mW/m2 before declining again in the final hour to 502 mW/m2. PES/ZY consistently shows an increase in power density over the 3 hours, from 352 up to 1012 mW/m2 indicating stable and improved performance. PES/AZ1 demonstrates a sharp increase in power density at the first hour, from 1034 to 1413 mW/m2 before significantly decreasing in the subsequent hours, especially in the last hour which down to 112 mW/m2. PES/AZ2 shows an initial increase up to 778 mW/m2, but in terms of overall performance, it remains similar to the Neat PES membrane. Conversely, PES/AZ3 exhibits the lowest power density, which ranging from 2-10 mW/m2 indicating that the Zeolite-Y has been severely damaged by its 3M activation, which adversely affects its MFC performance. From these results, PES/AZ1 achieves the highest peak power density, suggesting that the activation of Zeolite-Y and its usage as a filler in Mixed Matrix Membranes (MMMs) significantly enhances MFC performance. Unfortunately, in terms of long-term stability, PES/ZY performs better overall, as evidenced by increasing trends in each measured parameter. 1 M Acid-Activated Zeolite (AZ1) shows enhanced performance due to controlled dealumination process. This process increased Si/Al ratio that affects number of active sites and improves proton transport. However, it compromises long term stability of zeolite. Without the aluminum framework that builds the initial uniform structure, the structure can collapse over time.

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**FIGURE 5.** Power Density of Each Membrane for 3 Hours

# CONCLUSION

From both the characterization and performance tests, it is evident that increasing acid concentrations to activate Zeolite-Y can severely damage its crystalline structure, as confirmed by XRD analysis, ultimately leading to inferior MFC performance. However, 1 M acid activation appears to be ideal for Zeolite-Y, as it largely preserves the crystalline structure and significantly enhances the immediate performance of the MFC. Unfortunately, this acid activation of Zeolite-Y also results in a loss of long-term stability for the Mixed Matrix Membrane (MMM), with its performance declining after a significant initial increase. Meanwhile, the inactivated Zeolite-Y, which retains its original crystalline structure, demonstrates substantial improvement and superior long-term stability in MFC performance. Therefore, future work could explore further optimization conditions for MMMs that can effectively balance initial performance enhancement with sustained long-term stability. This could involve, for instance, investigating lower concentrations of acid for zeolite activation, exploring different activation methods, or optimizing membrane filler loading.

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