**Performance Comparison of PES/Activated Zeolite Mixed Matrix Membrane for Microbial Fuel Cell Applications**

Atiqah Atiqah, M Haikhal Fazad, Cicilia Puspa Rini, Avintha Devany Hanwisha, Hendrayanto Sulistyo Putro, Triyanda Gunawan a)

Author Affiliations

*Department of Chemistry, Faculty of Science and Data Analytics, Institut Teknologi Sepuluh Nopember*

*Surabaya, Indonesia*

a) *Corresponding author: triyanda@its.ac.id*

**Abstract*.***Membranes are widely used and play significant role in Microbial Fuel Cell (MFC) process. MFC uses bacteria as a catalyst to convert chemicals to electrical energy, offering a potential solution to our current energy crisis. In this research, we fabricated Mixed Matrix Membrane (MMMs) based on poly-ether sulfone (PES) polymer and adding Acid-Activated Zeolite (AZ) as a filler to make PES/AZ MMMs to enhance MFC’s performance. This study focuses on optimizing the amount of acid-activated zeolite fillers, with various concentrations of 0.1, 0.3 and 0.5%wt of polymer, which can differ in its result and the proton conductivity performance. X-Ray Diffraction (XRD) shows thatoverall Zeolite-Y structure is still predominantly preserved after activation using 2 M hydrochloric acid, although structural changes have begun to be induced. Meanwhile, MMMs are fabricated through phase inversion method and characterized using water contact angle (WCA) measurements and evaluated for their MFC performance. Overall, all MMMs show increase in water contact angle compared to PES Membrane, which means that adding filler will increase membrane hydrophobicity, although all MMMs are still classified as hydrophilic. The performance evaluation shows that 0.1%wt of Acid-Activated Zeolite MMM (0.1AZ) shows the best performance among all membrane, reaching peak voltage, current, and power density up to 71 mV, 32 mA/m2, and 2272 mW/m2, respectively. Compared to inactivated zeolite, acid-activated zeolite MMMs shows superior performance in all aspects, reaching 514% increase in power density, highlighting its superior performance. Overall, low loading of acid-activated zeolite shows the optimal performance as a membrane filler for MFC performance. This research explores the optimization of novel filler-polymer composition of MMMs to achieve high performance of energy generation through MFC process.

**Keywords:** Acid-Activated Zeolite, Mixed Matrix Membrane, Microbial Fuel Cell, PES Membrane.

# INTRODUCTION

To achieve the Net Zero Emissions target and limit the increase of Earth's temperature to under 1.5°C. Unfortunately, our current usage of fossil fuels is still excessive, especially for generating electricity and powering our industry, which results in CO2 emissions. One of the ways to keep the pledge is to transition our current energy system to renewables to produce electricity [1]. Hydropower system that relies on streams and river flows in many places are the main renewable energy source that we have today. Unfortunately, large volumes of industrial and household waste are discharged into these water flows without any treatment, leading to increasing environmental concerns and hindering the use of these waters to generate electricity[2].

One promising solution that addresses both energy generation and wastewater treatment is the Microbial Fuel Cell (MFC). MFCs utilize the metabolic processes of electroactive microorganisms to degrade organic matter while generating electricity [3–5]. These systems operate under mild conditions, produce minimal emissions, and are relatively simple in construction—making them suitable for decentralized, sustainable applications.

A typical MFC comprises an anode and cathode chamber separated by an ion-selective membrane. The membrane serves to limit oxygen crossover from cathode to anode and facilitates proton transport, which is essential for maintaining electrical current. Microbial oxidation at the anode produces electrons and protons; the electrons are directed through an external circuit, while the protons migrate through the membrane to the cathode, where they combine with oxygen to form water[6]. Ion exchange membranes in MFCs are generally classified as cation or anion exchange types, both of which are polymer-based due to their structural durability. Cation exchange membranes, particularly proton exchange membranes (PEMs), are preferred for their superior ionic conductivity [7,8]. Nafion, a commercially available PEM, has been extensively used in MFCs owing to its effective proton transport via sulfonic acid groups. However, Nafion’s widespread application is hindered by drawbacks including high cost, biofouling susceptibility, thermal dehydration, and limited mechanical strength, which necessitate the need to develop alternative membrane materials [9].

Recent research has focused on developing composite or mixed matrix membranes (MMMs) to improve MFC performance and reduce material costs. Polyether sulfone (PES) has emerged as a promising polymer candidate due to its hydrophilic nature, excellent thermal and chemical stability, and compatibility with biological environments. Nevertheless, PES exhibits low intrinsic proton conductivity, as its sulfone groups do not readily generate mobile ions. Although sulfonation can enhance its conductivity, the process involves hazardous reagents and can compromise the membrane’s mechanical properties [3,10,11].

A safer and more effective strategy involves embedding inorganic fillers into PES to form MMMs with enhanced functionality. Inorganic materials can facilitate proton transport and improve water retention, key factors in membrane performance. Among the various fillers investigated—such as titanium dioxide, silica, and zirconium phosphate—zeolites have gained considerable attention. These crystalline aluminosilicates possess high surface area, defined pore structures, and notable ion-exchange capacity [12–14] Zeolite-Y features a large pore size (~7.4 Å) and strong hydrophilicity, which contribute to improved water uptake and proton conduction, making it a suitable filler for PES-based MMMs in MFC applications.

The functional performance of Zeolite-Y can be significantly enhanced through acid activation. This treatment—commonly performed with strong acids such as citric and hydrochloric acid selectively removes aluminum atoms from the framework, effectively increasing the silicon-to-aluminum (Si/Al) ratio. Controlled dealumination not only optimizes the zeolite’s hydrophilicity for better compatibility with polymer matrices but also introduces Lewis acid sites and facilitates the development of enlarged micropores or mesopores, thereby improving mass transport properties [15]. Furthermore, acid treatment eliminates residual impurities and converts the zeolite to be more acidic, introducing Brønsted acid sites that are beneficial for proton conduction. When carefully regulated, this process enhances pore connectivity, surface acidity, and structural accessibility without compromising the zeolite’s crystallinity—ultimately improving its effectiveness as a filler in mixed matrix membranes [16].

In the development of mixed matrix membranes (MMMs), the amount of filler incorporated plays a critical role in determining overall membrane performance [17,18]. Excessive filler loading can lead to particle agglomeration, interfacial defects, and reduced mechanical integrity, while insufficient loading may result in negligible improvements [9]. Previous studies mainly focus on membrane modifications, such as polymer and surface modification, or adding different types of materials. Besides, simple modification such as fabrications of MMMs composed of PES polymer and acid-activated zeolite filler has not been thoroughly studied before. Therefore, this study aims to determine the optimal loading of acid-activated Zeolite-Y into a polyether sulfone (PES) matrix for MMM fabrication. Zeolite-Y was incorporated at varying concentrations of 0.1, 0.3, and 0.5wt% relative to PES. The resulting PES/activated Zeolite-Y membranes will be characterized and evaluated for their performance in Microbial Fuel Cell (MFC) applications, to develop a cost-effective, high-performance, and sustainable membrane suitable for integrated 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 a 2 M 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 obtained zeolite was subsequently designated as AZ, which stands for Activated Zeolite.

## Mixed Matrix Membrane Fabrication

Mixed Matrix Membranes (MMMs) are fabricated using the dry-wet phase inversion method. To prepare the dope solution, 50 grams were initially prepared by dispersing 17%wt of polyethersulfone (PES) in 40 mL of NMP. Subsequently, 0.1, 0.3, and 0.5%wt of Zeolite-Y (ZY) and acid-activated Zeolite (AZ) fillers were slowly dispersed into the NMP and mixed for 30 minutes using a magnetic stirrer. This was followed by sonication in a water bath sonicator for an additional 30 minutes. After sonication, the PES powder was slowly added to the filler-NMP solution, and the mixture was stirred for 24 hours at 80°C with 300 rpm. Following the stirring period, the solution was left overnight to allow for bubble removal. The prepared dope solution was then cast onto a glass plate to fabricate the MMMs. The glass plate with the cast solution was subsequently immersed in water to facilitate membrane detachment. The obtained MMMs were then soaked, first in water and then in ethanol solution, overnight for each respective solvent. After soaking, the MMMs were dried at room temperature for 2-3 days until all visible moisture had evaporated from their surfaces. Each fabricated MMM is named 'xAZ' or 'xZY', where 'x' represents the weight percentage of filler added into the polymer matrix.

## Characterization

The crystallinity of neat Zeolite-Y and its acid-activated counterpart is identified using X-Ray Diffraction (XRD). Meanwhile, the hydrophobicity of the membrane is determined by measuring its Water Contact Angle (WCA) using the sessile drop method, which involves placing a water droplet onto the membrane surface. 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 membrane 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 a UV-Vis Spectrophotometer in 600nm wavelength. 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 a 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 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 (XRD) Result

The crystallinity and structural integrity of Zeolite-Y and its acid-activated counterpart are revealed by the XRD diffractogram in Figure 2. Activation with 2 M HCl demonstrates that the crystallite faujasite structure remains largely preserved, particularly evident in the main peaks at 2θ < 30°. However, for many medium and small-sized peaks beyond this range, a decrease in intensity and peak broadening are observed, which are indicative of dealumination caused by the acid solution [7]. This suggests that although the overall Zeolite-Y structure is still predominantly preserved, structural changes have begun to be induced.

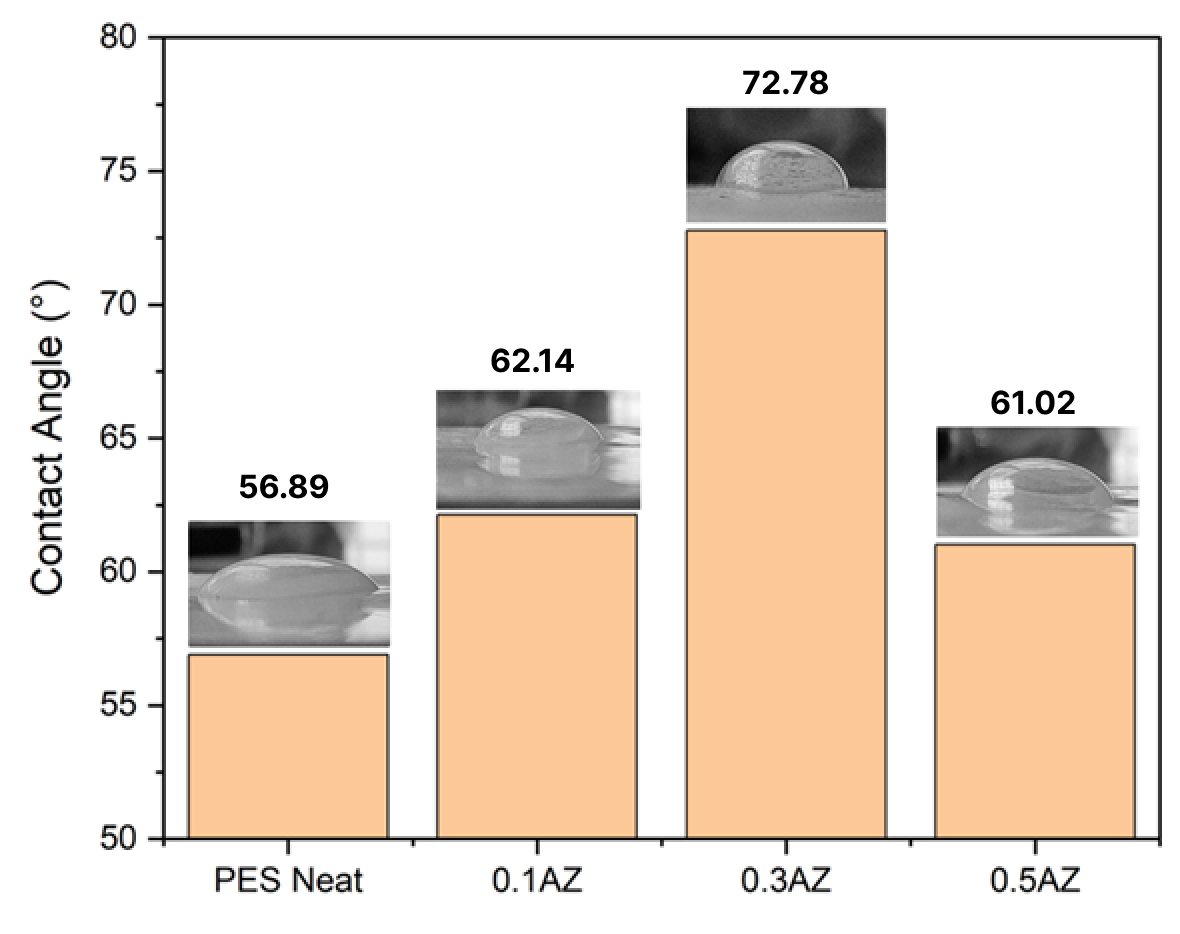
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**FIGURE 2.** XRD Diffractogram of Commercial Zeolite-Y and Activated Zeolite-Y

## Water Contact Angle (WCA) Measurement

The result of the water contact angle measurement is shown in **Figure 3**. Surface interaction with water can be classified into 2: hydrophobic groups (if θ >90°) and hydrophilic (if θ <90°). From the results, all fabricated membranes are classified as hydrophilic membranes, which makes them suitable for MFC applications. The Neat PES membrane shows the lowest contact angle with 56.89°, which is expected since PES is mostly grouped as a hydrophilic polymer. Adding activated zeolite-Y filler initially shows an increase in the contact angle as filler loading increases, reaching a maximum at 0.3% wt. Specifically, adding 0.1%wt leads to a slight increase to 62.14° in its contact angle. The dealumination of zeolite-Y that occurs during the activation process does make zeolite-Y more hydrophobic [8]. Therefore, a higher loading of hydrophobic filler should theoretically increase its contact angle, as fillers spread throughout the matrix, thereby decreasing the polymer's effect on the surface. This theoretical expectation is proven at 0.3% wt, which increased the contact angle up to 72.78°. Unfortunately, this phenomenon is not observed at 0.5%wt, where the contact angle is decreased to 61.02°, closer to that of 0.1AZ and Neat PES; this likely happens because of the agglomeration of filler particles. The filler loadings investigated in this research (0.1, 0.3, and 0.5%wt) are relatively low. However, even within such low loading ranges, the tendency of zeolite particles to agglomerate within the polymer matrix can lead to phenomena like that observed at 0.5%wt [10].



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

## Fuel Cell Performance

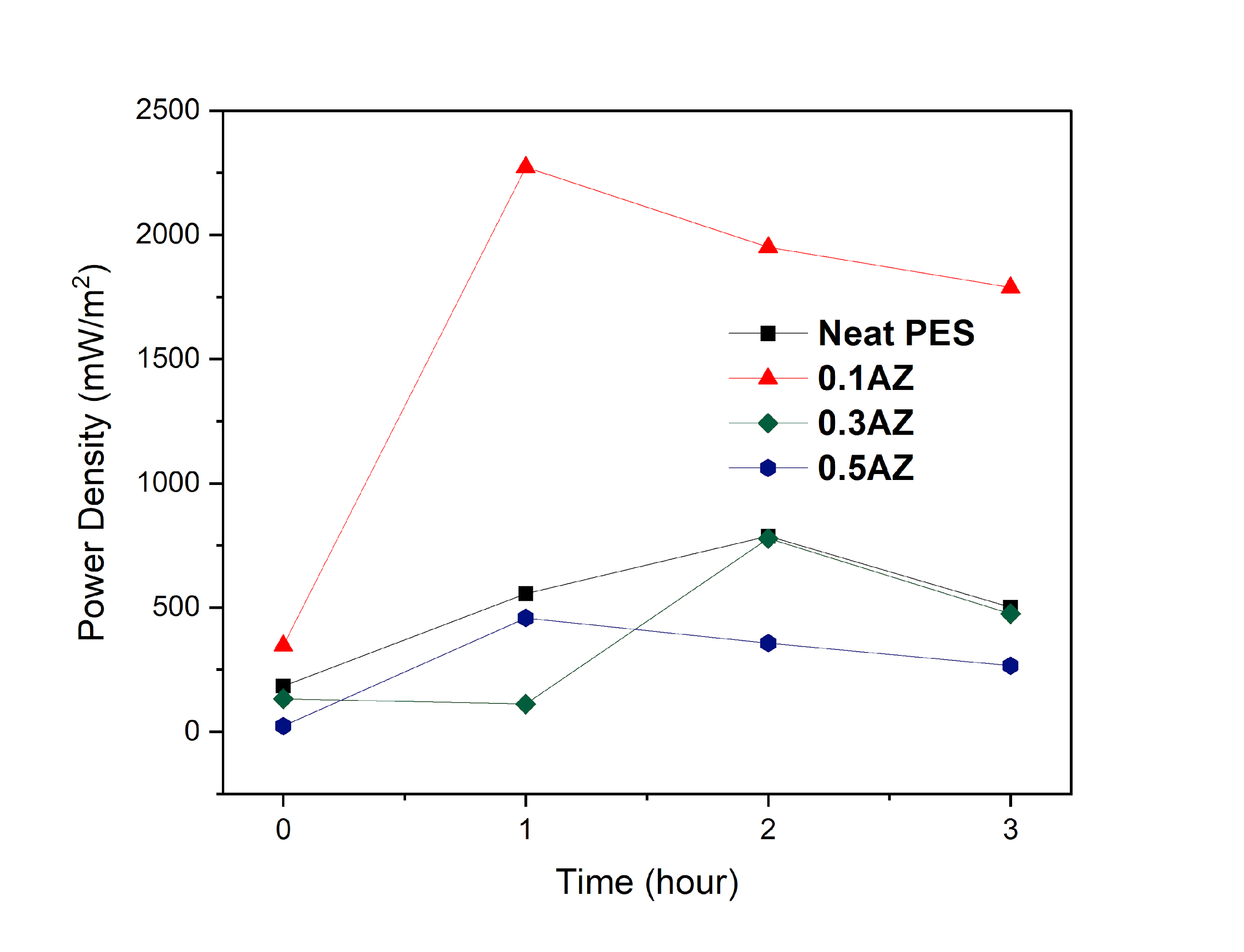
To evaluate Microbial Fuel Cell (MFC) performance, it is crucial to assess the system's capability to generate both voltage and current over time. The voltage generated by the membranes is presented in Figure 4(a). The Neat PES membrane initially generated a very low voltage around 5.67 mV, which increased to 39.38 mV, before a slight decrease to 33.46 mV in the final hour. The 0.1AZ membrane started at 27.8 mV, a moderate voltage level, then showed a significant increase over time, particularly sharp in the first hour up, reaching 71 mV, the highest overall voltage. In contrast, the 0.3AZ membrane exhibited a very high initial voltage around 53.16 mV, but this performance unfortunately decreased consistently, down to 31.64 mV over the 3-hour duration, indicating a lack of stability. The 0.5AZ membrane displayed a relatively similar voltage profile to the Neat PES membrane, which ranged around 9-30 mV, suggesting minimal enhancement at this higher filler concentration.

Concurrently, the current generation capabilities of the membranes are shown in Figure 4(b). The Neat PES membrane started with a very high current around 32.5 mA/m2, but this rapidly decreased over the experimental period, down to only 15 mA/m2. The 0.1AZ membrane mirrored its strong voltage trend, showing a significant increase in current generation, especially reaching 32 mA/m2 in the first hour, and maintaining a high level thereafter. The 0.3AZ membrane exhibited a delayed increase in current generation, peaking around 20 mA/m2 in the second hour before decreasing to 15 mA/m2 in the final hour. Similar to its voltage performance, the 0.5AZ membrane showed generally low current generation, which ranged from 2.5-12.5 mA/m2, and did not significantly outperform the Neat PES in a sustained manner. Based on these results, the 0.1AZ membrane consistently exhibits the best overall performance among all tested membranes, demonstrating strong and relatively stable generation of both voltage and current throughout the 3-hour evaluation period.

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

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

Power density serves as the direct overall performance indicator for the membrane, reflecting both the generated current and voltage. The power density of the membranes can be observed in Figure 5. The 0.1AZ membrane exhibits far superior performance compared to the other three membranes. These other three membranes show relatively similar endpoints and primarily differ in when their significant power density increase occurs. The 0.1AZ membrane's power density peaked at the first hour, reaching 2272 mW/m2, before gradually decreasing for the remaining hours until the experiment concluded. However, its final power density is reaching 1788 mW/m2, significantly higher than that exhibited by the other membranes. Meanwhile, Neat PES, 0.3AZ, and 0.5AZ membranes consistently demonstrated modest to low power densities. Specifically, the Neat PES membrane's power density increased during the first two hours up to 787 mW/m2, before subsequently decreasing to 502 mW/m2. The 0.3AZ membrane experienced a late increase to around 778 mW/m2, but in the end, its power density remained relatively similar to that of Neat PES. The 0.5AZ membrane's power density increased in the first hour to 458 mW/m2 and then decreased for the remaining time. Overall, the 0.1AZ membrane clearly shows the best performance, proving to be the optimal concentration of filler loading for the Mixed Matrix Membrane (MMM) in this study.



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

Generally, the incorporation of acid-activated zeolite-Y can significantly enhance the performance of Microbial Fuel Cells (MFCs). This is clearly illustrated in **Figure 6**, where the 0.1%wt loading of the acid-activated zeolite (0.1AZ) is directly compared to a Mixed Matrix Membrane containing 0.1%wt of inactivated zeolite-Y (0.1ZY). The 0.1AZ membrane consistently demonstrates significantly superior voltage and current generation compared to its inactivated counterpart. This enhancement is particularly pronounced in the first hour of operation, where the 0.1AZ membrane rapidly achieves its peak voltage, current, and power density, which are 71 mV, 32 mA/m2, and 2272 mW/m2, respectively. Meanwhile, the 0.1ZY membrane peaked at the end of the experiment, reaching 30.69mV, 15 mA/m2, and 441.75 mW/m2 in voltage, current, and power density, respectively. This superior electrochemical performance is unequivocally reflected in its much higher power density, where there is 514% power density increase between 0.1AZ and 0.1ZY, confirming that the acid activation process indeed improves MFC performance. The improved performance likely stems from the acid activation creating more highly active sites on the zeolite for efficient ion exchange mechanisms crucial to MFC operation, as well as potentially optimizing proton transport pathways [6].

Furthermore, the Water Contact Angle (WCA) measurements in **Figure 6(d)** reveal an interesting trend: the membrane with inactivated zeolite-Y (0.1ZY) exhibits a higher contact angle, indicating it is more hydrophobic than the membrane with acid-activated zeolite (0.1AZ). This observation is notable because, theoretically, acid activation (dealumination) of zeolite-Y is generally expected to increase hydrophobicity by removing hydrophilic aluminum sites. The reverse happening here—where the activated zeolite membrane shows increased hydrophilicity—suggests alternative mechanisms at play. One plausible explanation for this unexpected hydrophilicity in the 0.1AZ membrane is that the acid activation process may lead to better dispersion of the zeolite particles within the polymer matrix. Improved dispersion could result in a greater exposure of hydrophilic PES segments on the membrane surface or a more favorable arrangement of the activated zeolite that enhances its interaction with water at this specific low loading [11].

|  |  |
| --- | --- |
| **(a)** | **(b)** |
|  |  |
| **(c)** | **(d)** |

**FIGURE 6.** Voltage (a), Current (b), Power Density (c) and Water Contact (d) Comparison between PES/ZY and PES/AZ with 0.1%wt Loading

# CONCLUSION

From both the characterization and performance tests, it is evident that activation of Zeolite-Y using a 2 M HCl solution largely preserves the zeolite-Y crystalline structure, although initial structural changes can be observed, as confirmed by XRD analysis. Incorporating 0.1%wt of this activated zeolite (AZ) into the polymer demonstrates the best performance for the MFC, where there is 514% increase in power density compared to inactivated zeolite-Y. The activated zeolite overall shows better performance by enhancing its ion exchange capabilities, particularly at its initial stage. Future work could explore further optimization conditions for MMMs, for example, by investigating different activation concentrations or methods, to search for MMMs that provide stable performance over time.

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