

Hydrophobic PVDF Nanofibers-covered Cathode and Corn cob-based anode for Enhanced Energy Sewage Wastewater-driven Microbial Fuel Cells

Nasser A. M. Barakat^{1,*}, Shima gamal¹, Mamdouh M .Nassar¹, Olfat A.Fadali¹, Hager M. Moustafa¹ and Omnia H. Abdelraheem²

¹ Chemical Engineering Department, Faculty of Engineering, Minia University, El-Minia 61516, Egypt.

²Sciences Engineering Department, Faculty of Engineering, Beni-Suef University Beni-Suef 62511, Egypt

Corresponding author: Nasser A. M. Barakat, Tel: +20862348005, E-mail: nasbarakat@mu.edu.eg

Abstract

In this study, we present a novel approach to enhance power generation in MFCs by incorporating several innovative modifications. The MFC design eliminated the need for a membrane by depositing a hydrophobic PVDF nanofibers layer to facilitate oxygen penetration while preventing water **passing**. Additionally, a new anode material was developed by sintering corn cob under a nitrogen atmosphere, providing improved performance and cost-effectiveness. The MFC operated as an air-cathode system, eliminating the need for expensive electron acceptors. Furthermore, the MFC was driven by sewage wastewater, demonstrating the potential for waste-to-energy conversion. The power generation performance of the MFC was evaluated and compared to traditional carbon-based anode materials. The results showed a significant increase in power density with the corn cob anode, reaching 515 W/m². In **comparison**, the power densities obtained with carbon felt, carbon cloth, and carbon paper anodes were 94, 95, and 140 W/m², respectively. These findings highlight the superiority of the corn cob anode in terms of power generation efficiency. The demonstrated increase in power density with the corn cob anode highlights the potential for scaling up MFCs for practical applications, including wastewater treatment and remote power sources.

Keywords: Microbial Fuel Cell; Renewable Energy; Electrospinning; Corn cob anode; Power density

1. Introduction

Microbial fuel cells (MFCs) have emerged as a promising technology for sustainable and renewable power generation. These bioelectrochemical systems harness the metabolic activity of microorganisms to convert organic matter directly into electrical energy[1-3]. MFCs offer several advantages over traditional fuel cells, such as their ability to utilize abundant and diverse organic waste streams as fuel sources. This characteristic makes MFCs particularly attractive for applications in wastewater treatment plants, where they can simultaneously treat wastewater and generate electricity[4, 5].

One significant challenge facing the commercialization of MFCs is their relatively low power output. While MFCs have demonstrated the ability to generate electricity, the current density achieved is often insufficient for practical applications. The low power output is attributed to various factors, including high internal resistance within the system, limited mass transport of reactants, and the inefficient transfer of electrons from microorganisms to the electrodes. Moreover, utilizing a membrane **causes appreciable energy loss through the MFC**. The presence of a membrane can lead to internal resistance and limitations in mass transport, thus affecting the overall performance of the system[6, 7].

Scalability is another major challenge in the commercialization of MFC technology. Many early-stage MFC designs have been limited to laboratory-scale experiments, making it challenging to translate their performance to larger, real-world applications. Scaling up MFCs requires addressing issues such as maintaining stable microbial communities, managing system dynamics, and optimizing electrode materials and configurations. Additionally, there is a need for robust and reliable MFC designs that can withstand the operational demands and variability of different application scenarios[8, 9].

Cost-effectiveness is a critical factor that influences the commercial viability of MFCs. The current high costs associated with MFC materials, electrode manufacturing, and system maintenance pose significant barriers to widespread adoption. Expensive components, such as platinum catalysts used at the cathode, contribute to the overall cost of MFCs. Additionally, the production and treatment of wastewater or other fuel sources used in MFCs can also impact the economic feasibility of the technology. Finding cost-effective alternatives and optimizing MFC designs to reduce overall system costs are essential for advancing commercial applications[10-12].

Addressing these challenges is crucial to realizing the full potential of MFCs for practical applications, such as wastewater treatment, renewable energy generation, and remote power sources. This manuscript aims to contribute to overcoming these challenges by presenting novel approaches to enhance power generation in MFCs. By eliminating the need for a membrane, using cost-effective materials, and optimizing system design, we strive to improve the performance, scalability, and cost-effectiveness of MFC technology, paving the way for its successful commercialization[13-15].

In this manuscript, we present a novel approach to enhance power generation in MFCs by eliminating the need for a membrane. Instead, we have implemented a unique design that allows for the penetration of oxygen while preventing water from passing through the cathode side. This was achieved by depositing hydrophobic PVDF (polyvinylidene fluoride) nanofibers, which provide a barrier to water while allowing for the diffusion of oxygen[3]. This design not only simplifies the MFC configuration but also improves the overall performance by reducing

internal resistance and facilitating the transport of reactants[16].

Additionally, we have developed a new anode material for our MFCs. Traditionally, carbon-based materials such as carbon cloth, carbon paper and carbon felt have been used as anodes in MFCs. However, these materials can be expensive and have limitations in terms of their surface area and catalytic activity. In our study, we prepared a novel anode by sintering corn cob under a nitrogen atmosphere at 1000°C. This process resulted in a highly porous and conductive material, providing an efficient substrate for microbial growth and electron transfer [17-19]. Furthermore, our MFCs utilize an air-cathode configuration, which eliminates the need for expensive compounds, such as platinum, as electron acceptors. The air-cathode design allows for the direct reduction of oxygen at the cathode, significantly reducing the cost of MFC operation[2, 20].

Lastly, our MFCs are driven by sewage wastewater, which serves as a readily available and abundant fuel source. Wastewater contains a high concentration of organic matter that can be utilized by microorganisms as a carbon source for energy production. By harnessing the power of microbial communities present in sewage, our MFCs offer a sustainable and cost-effective solution for simultaneous wastewater treatment and electricity generation. The results of our study demonstrate the potential of this innovative approach for practical applications in wastewater treatment and renewable energy generation.

2. Experimental section

2.1. Preparation of PVDF Nanofibers Layer

A PVDF solution was prepared by dissolving PVDF polymer (purity > 99%, molecular weight 200,000 g/mol) in a solvent mixture of N,N-dimethylformamide (DMF) and acetone. The PVDF concentration in the solution was optimized to achieve suitable viscosity for electrospinning. The PVDF-to-solvent ratio was 15 wt% to ensure proper spinnability. An electrospinning setup was assembled, consisting of a high-voltage power supply, a syringe pump, a metallic needle, and a grounded collector on which the cathode (3.5×3.5 cm² Pt-loaded carbon cloth) to form a hydrophobic surface. The prepared PVDF solution was loaded into

a syringe, and a needle with an appropriate gauge was attached. The needle was connected to the positive terminal of the high-voltage power supply, while the collector was grounded. The electrospinning process was performed at a controlled ambient temperature and humidity. The electrospinning parameters, such as the applied voltage, flow rate, and distance between the needle and collector, were optimized to obtain uniform and continuous PVDF nanofibers. The typical electrospinning conditions used in this study were as follows: applied voltage of 15 kV, flow rate of 1 mL/h, and needle-to-collector distance of 15 cm.

2.1. Anode preparation

Corn cob-derived anodes were prepared by sintering corn cob particles under a nitrogen atmosphere. The corn cob was thoroughly cleaned, dried, and ground into fine particles. The ground corn cob was then placed in a crucible and heated in a tube furnace at 1000°C under a flowing nitrogen gas atmosphere for 2 hours. After cooling, the sintered corn cob particles were collected and used as the anode material.

2.3. MFC configuration

The MFC was designed as a cost-effective and portable device by utilizing an air cathode configuration. The cathode consisted of a carbon cloth electrode covered with PVDF nanofibers, with one side facing the air to control oxygen diffusion and prevent water leakage. The other side of the cathode was loaded with Pt/C particles (0.5 mg/cm²) and faced the water side. A high corrosion resistance stainless steel sheet served as the current collector at the cathode side, while the graphitized corn cob anode was directly connected to the data logger. For comparison, carbon cloth and carbon papers (3.5×3.5 cm) were used as anodes under similar conditions. To initiate the MFC operation, the prepared anodes were immersed in an 80 ml solution of sewage wastewater after purging with nitrogen for 10 minutes. The microorganisms were allowed to immobilize on the anode by injecting the media into an anaerobic chamber until the open circuit voltage (OCV) stabilized and the equilibrium of the two half-reactions was achieved. The MFC was then operated by connecting the anode and cathode collectors using a potentiostat. The anode and cathode potentials were

measured using an Ag/AgCl reference electrode. The OCV was measured using a potentiostat connected to a GL220 midi-logger for data recording. To describe the current as a function of voltage (LSV), the MFC circuit was closed by adjusting the external resistance (load) once the OCV reached stabilization. Linear sweep voltammetry (LSV) was performed at a scan rate of 1 mVs⁻¹, starting from the observed highest OCV down to zero voltage.

3. Results and discussion

Fig. 1 displays a photo image for the original and sintered corncob anode. As shown in the figure, the morphology of the corn cob did not destroy during the graphitization process. Moreover, the mechanical properties were adequate to handle the graphitized corn cob as an anode in the MFC. The SEM image of the sintered corn cob (Figure 2) reveals the presence of numerous pores in the sample, indicating a porous structure within the proposed anode. The observation of these pores is of great significance as it provides insights into the application in MFC. The presence of pores in the MFC can have several positive implications. Firstly, the porous structure increases the effective surface area available for microbial colonization. This allows for a greater population of electroactive bacteria to adhere to the anode, leading to enhanced microbial activity and improved electron transfer. Additionally, the increased surface area facilitates the contact between the bacteria and the substrate, improving the efficiency of organic matter degradation and subsequent electron generation[21]. Moreover, the porous structure allows for efficient diffusion of the substrate and products within the MFC. The pores act as conduits for the transport of wastewater and oxygen, ensuring a continuous supply of nutrients and electron acceptors to the microorganisms. This facilitates the maintenance of favorable conditions for microbial growth and activity, leading to sustained and improved power generation[22].

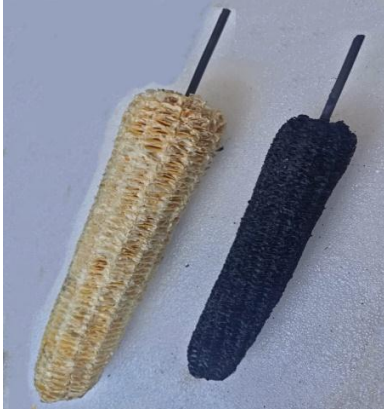


Fig. 1 Photo image for the original and sintered corn cob.

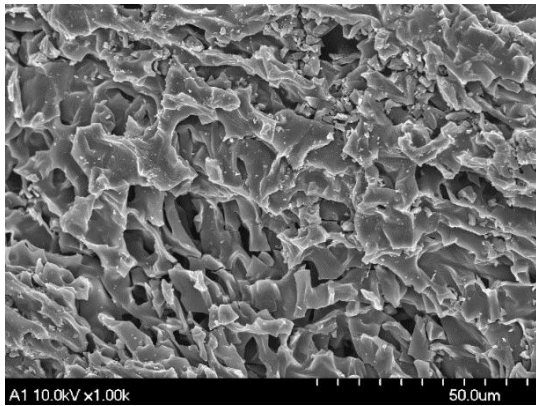


Fig. 2 SEM image for the sintered corn cob.

The use of X-ray diffraction analysis (XRD) is a reliable method for studying the composition of materials that are crystalline or semi-crystalline. Figure 3 illustrates the XRD pattern of the proposed corncob anode, which was prepared at a temperature of 1000 °C. According to the International Centre for Diffraction Data (ICDD) card number 46-1045, which is maintained by the Joint Committee on Powder Diffraction Standards (JCPDS), the anode consists of silicon oxide (quartz). The presence of strong diffraction peaks at two theta values of 20.8°, 26.6°, 36.5°, 50.1°, 59.9°, and 68.2°, which correspond to crystal planes (100), (101), (110), (112), (211), and (203), respectively, indicates the presence of quartz (SiO₂). Moreover, a broad diffraction peak at a 2θ value of approximately 24° suggests the presence of amorphous graphite, which was obtained from lignocellulosic raw materials. The XRD patterns for all three formulations were similar,

and they all contained similar compositions, namely SiO₂-incorporated graphite.

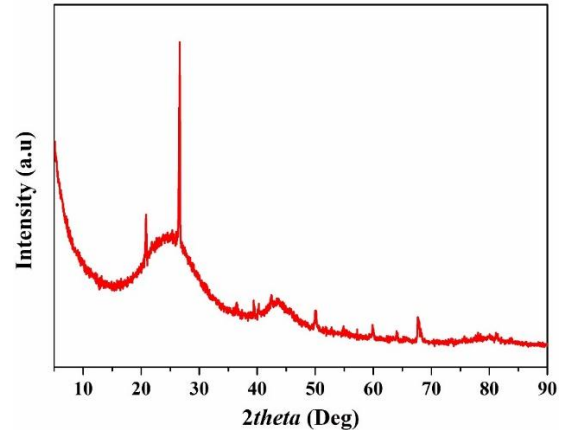


Fig. 3 XRD pattern for the sintered corn cob.

The SEM image of the deposited PVDF layer on the cathode reveals the presence of smooth and **bead-free** nanofibers as shown in Fig. 4. This observation is significant as it indicates a successful electrospinning process and the formation of a desirable nanofiber morphology for the intended application in the MFC. The smooth and beads-free nanofibers suggest a uniform and continuous coating of the PVDF layer on the cathode surface. **The average diameter of the produced nanofibers is 320±20 nm.** This uniformity is crucial for ensuring efficient oxygen diffusion and preventing water leakage, as aforementioned. The absence of beads or irregularities in the nanofibers indicates a consistent electrospinning process, where the polymer solution was appropriately controlled to maintain a stable jet during the fiber formation[23, 24]. The super hydrophobic nature of the PVDF layer is evident from the water contact angle measurement, as indicated by the inset in the figure. The water contact angle of approximately 140 degrees suggests a highly hydrophobic surface, which further supports the intended purpose of the PVDF layer in preventing water pass through the cathode. The hydrophobicity of the PVDF layer helps repel water, preventing its accumulation on the cathode surface and avoiding flooding, which can impede the oxygen diffusion necessary for efficient MFC operation. The super hydrophobic properties of the PVDF layer contribute to the overall success of the MFC design, ensuring the separation of the air and water sides and

maintaining a conducive environment for microbial activity [25-27].

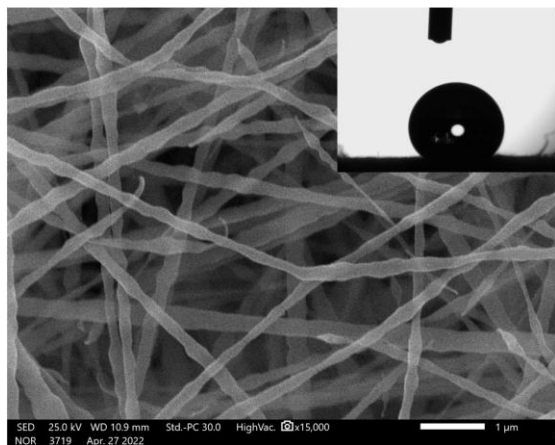


Fig. 4 SEM image of the deposited PVDF layer on the surface of the cathode, the inset displays the water contact angle on the deposited layer.

The open circuit potential (OCV) is an important parameter to assess the performance and stability of MFCs. In Figure 5, the OCV versus time graph demonstrates the behavior of different anode materials, including graphitized corn cob, carbon paper, carbon felt, and carbon cloth, in terms of OCV stabilization and maximum OCV values. The observed similarity in the OCV behavior between the graphitized corn cob and carbon paper anodes suggests that these materials exhibit comparable electrochemical characteristics. Both anodes reached a maximum OCV of approximately 0.78 V, indicating similar electron transfer capabilities and overall performance. Furthermore, the stabilization time of around 47 working hours for both anodes indicates a relatively quick establishment of the equilibrium between the anode and cathode reactions. This rapid stabilization is favorable for MFC operation, as it allows for efficient electron generation and transfer, leading to improved power generation.

In contrast, the carbon felt anode displayed a longer stabilization time compared to the other investigated anodes. The required stabilization time of approximately 137 hours suggests slower establishment of the anode-cathode equilibrium and electron transfer kinetics. This delay in stabilization can be attributed to the intrinsic properties of the

carbon felt material, such as its surface morphology and conductivity. The lower maximum OCV value of 0.68 V for the carbon felt anode further supports the notion of compromised electron transfer and reduced power generation potential. However, it is important to note that even though the carbon felt anode exhibited the longest stabilization time and the lowest maximum OCV, it still contributed to power generation, albeit at a relatively lower efficiency compared to the other anodes.

The carbon cloth anode demonstrated intermediate performance in terms of stabilization time and maximum OCV. The required stabilization time of approximately 82 hours indicates a moderate establishment of the anode-cathode equilibrium, while the stable OCV value of 0.74 V suggests efficient electron transfer and power generation capabilities. The performance of the carbon cloth anode falls between that of the graphitized corn cob/carbon paper anodes and the carbon felt anode, highlighting its potential as a viable option for MFC applications[28].

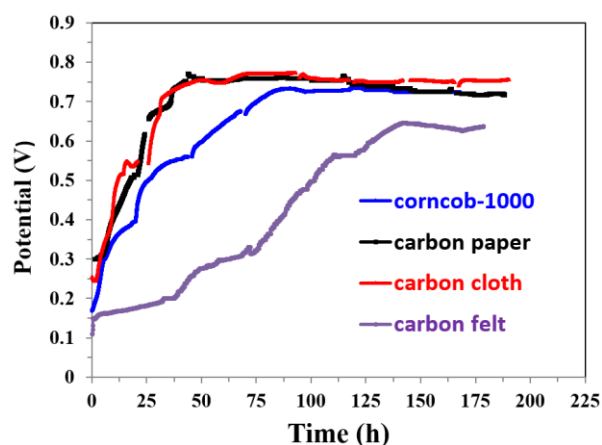


Fig. 5 OCV of the assembled MFCs using commercial anodes and the proposed corn cob-based anode.

The power density generated by MFCs is a crucial parameter that reflects the overall performance and efficiency of the system. In Figure 6, the power density is plotted against the corresponding current density for different anode materials, including graphitized corn cob, carbon felt, and carbon paper. Notably, the corn cob anode demonstrates significantly higher power density compared to the other anodes. The graphitized corn cob anode

exhibits a remarkable power density of 535 mW/m^2 , corresponding to a current density of 3250 mA/m^2 . This high power density can be attributed to several factors. Firstly, the unique properties of the graphitized corn cob, such as its high surface area, porosity, and conductivity, contribute to enhanced microbial activity and efficient electron transfer. These factors create favorable conditions for the growth and attachment of electroactive bacteria, leading to increased power generation. Additionally, the graphitization process further enhances the electrical conductivity of the corn cob, facilitating the flow of electrons from the microbial community to the anode.

In contrast, carbon felt, carbon cloth and carbon paper anodes exhibit lower power densities. The calculated power densities for carbon felt, carbon paper, and graphitized corn cob are 147 , 103 and 96 mW/m^2 , respectively, at corresponding current densities of 830 , 120 and 250 mA/m^2 . The relatively lower power densities of these anodes can be attributed to factors such as lower surface area, reduced conductivity, or differences in the microbial community composition and activity.

The significant difference in power density among the anodes underscores the importance of anode material selection in MFC performance. The graphitized corn cob anode exhibits superior power generation capabilities, highlighting its potential as a highly efficient anode material for MFCs. The higher power density achieved with the graphitized corn cob anode can have substantial implications for the commercial-scale implementation of MFC technology, as it allows for more efficient electricity production and improved energy conversion. Further research can focus on understanding the underlying mechanisms behind the superior performance of the graphitized corn cob anode, including the specific microbial community dynamics, biofilm formation, and electron transfer pathways. Optimization of the anode materials and design based on these insights can lead to further improvements in power density and overall MFC performance. Additionally, studies exploring the long-term stability and durability of the graphitized corn cob anode, as well as its scalability and cost-effectiveness, can provide valuable information for potential industrial applications.

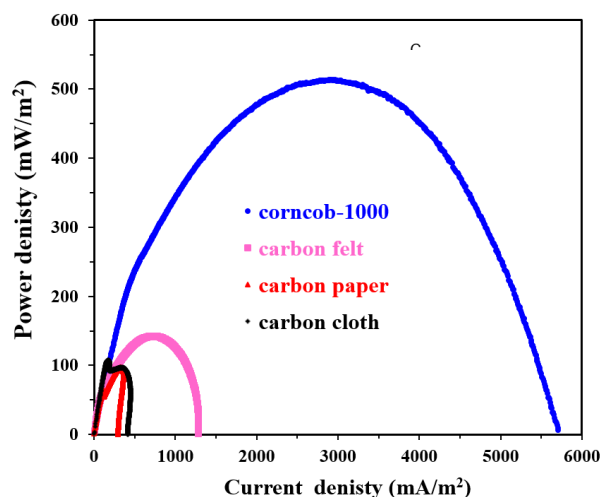


Fig. 6 Generated power density from the assembled MFCs versus the observed power density using commercial anodes and the proposed corn cob-based anode.

Figure 7 demonstrates the impact of utilizing sodium acetate as an additive in the anolyte solution on the power density generated by the corn cob-based MFC. The results reveal a significant increase in power density from 210 to 515 mW/m^2 upon the addition of sodium acetate. The observed increase in power density can be attributed to the role of sodium acetate as a supplementary carbon source for the microbial community in the anode chamber. Sodium acetate serves as an additional substrate that can be readily metabolized by the microorganisms, leading to enhanced microbial activity and increased electron generation. By providing a supplementary carbon source, sodium acetate promotes the growth and metabolic activity of electroactive bacteria in the anode biofilm. This results in higher rates of organic matter degradation and improved electron transfer efficiency. The additional carbon source can replenish any potential carbon limitation in the anode chamber, allowing the microorganisms to sustain their metabolic activity and produce more electrons, thereby increasing the power density.

Furthermore, the presence of sodium acetate can also influence the microbial community composition in the anode chamber. Certain species of bacteria are known to utilize acetate as their primary carbon source. The introduction of sodium acetate into the system may selectively favor the growth of acetate-utilizing bacteria, which can exhibit higher

electroactivity and contribute to increased power generation [29, 30]. It is important to note that the optimal concentration of sodium acetate needs to be carefully determined, as excessively high concentrations may lead to negative effects such as substrate inhibition or pH disturbances. The concentration of 0.4 g of sodium acetate used in this study appears to have a positive impact on power density without causing detrimental effects.

Future research can focus on elucidating the specific metabolic pathways and microbial community dynamics associated with the utilization of sodium acetate in MFCs. Understanding the interactions between the added carbon source, microbial community composition, and electron transfer mechanisms will provide valuable insights for the development of strategies to enhance power generation in MFCs. Additionally, investigations into the long-term stability and sustainability of sodium acetate supplementation in MFC operation can contribute to its practical implementation in real-world applications [31, 32].

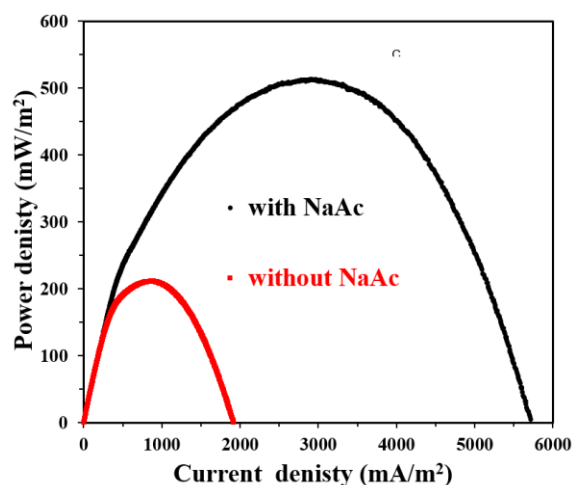


Fig. 7 Generated power density from the assembled MFCs versus the observed power density using corn cob-based anode with and without nickel acetate salt.

Figure 8 depicts the impact of adding sodium acetate to the MFC system on the open circuit voltage (OCV) and stabilization time. The results demonstrate that the addition of sodium acetate did not significantly affect the maximum OCV, but it led to a reduction in the required stabilization time. The observed stability in the maximum OCV suggests that the addition of

sodium acetate did not alter the overall electrochemical behavior of the MFC system. The maximum OCV represents the equilibrium state between the anode and cathode reactions, indicating the efficiency of electron transfer and the overall performance of the MFC. The fact that the maximum OCV remained unchanged indicates that the addition of sodium acetate did not negatively impact the electron transfer processes or hinder the microbial activity responsible for power generation. However, the notable reduction in the required stabilization time from approximately 75 hours to around 32 hours indicates an accelerated establishment of the biofilm formation upon the addition of sodium acetate. Stabilization time refers to the period needed for the MFC to reach a steady-state condition where the electrochemical reactions and microbial processes are balanced. A shorter stabilization time is desirable in MFC operation as it allows for quicker initiation of power generation and more efficient utilization of the system. The reduced stabilization time can be attributed to the beneficial effects of sodium acetate on the microbial community in the anode chamber.

The addition of sodium acetate as a supplementary carbon source in MFCs offers a practical approach to enhance the operational efficiency of the system. By reducing the required stabilization time, MFCs can achieve faster startup times and improved power generation capabilities. This can have significant implications for various applications, especially those requiring rapid and reliable power generation from MFCs.

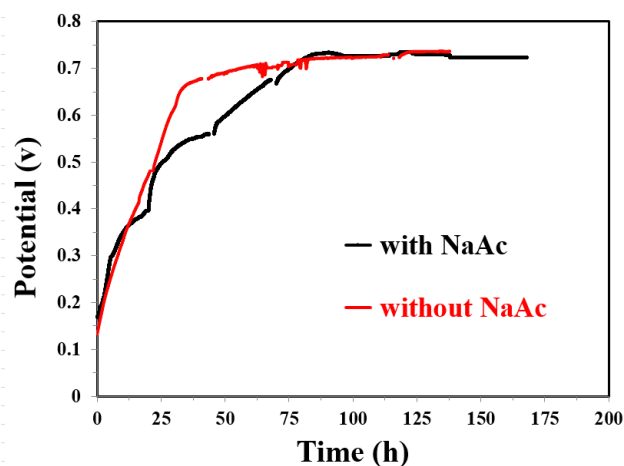


Fig. 8 OCV of the assembled MFCs using the proposed corn cob-based anode with and without sodium acetate addition.

Overall, MFC is a promising renewable energy device when its growth factors could be optimized. MFC growth is influenced by various factors that can impact its performance and efficiency. These factors can be summarized as follow [33]:

1. The type and concentration of organic substrates available for the microorganisms in the MFC greatly influence their growth. MFCs can utilize a wide range of organic compounds, such as glucose, acetate, and wastewater, as fuel sources. The availability and composition of the substrate affect the metabolic activity of the microorganisms and their overall growth.
2. The pH level and temperature of the MFC environment significantly impact microbial growth. Different microorganisms have specific pH and temperature ranges in which they thrive. Maintaining optimal pH and temperature conditions promotes microbial activity and enhances MFC performance.
3. Oxygen availability affects the microbial community composition and growth in MFCs. Aerobic microorganisms require oxygen for respiration, while anaerobic microorganisms can thrive in low or no oxygen conditions. The design of the MFC and the oxygen supply to the cathode can influence the growth of specific microbial species.
4. The choice of electrode material can influence the growth and activity of microorganisms in MFCs. The electrode surface characteristics, such as surface area, conductivity, and chemical composition, can affect microbial attachment, biofilm formation, and electron transfer processes. Different materials can favor the growth of specific microbial species and impact overall MFC performance.
5. Factors external to the MFC, such as light exposure, nutrient availability, and presence of contaminants, can influence microbial growth. Light can affect the photosynthetic activity of certain microorganisms, while the presence of pollutants or toxic compounds can inhibit microbial growth and reduce MFC performance.
6. The composition and diversity of the microbial community within the MFC play a crucial role in its

growth. The competition for resources, interspecies interactions, and synergistic relationships between different microorganisms impact their growth rates and overall MFC performance.

Optimizing these factors and understanding their interactions is essential for enhancing microbial fuel cell growth and efficiency. Researchers continue to explore strategies to manipulate these factors to improve MFC performance and make microbial fuel cells a viable and sustainable technology for energy generation and wastewater treatment.

4. Conclusions

By utilizing sewage wastewater as the fuel source, we demonstrated the feasibility of waste-to-energy conversion, showcasing the environmental sustainability of the proposed MFC design. Significantly, the power density achieved with the corn cob anode reached an impressive 515 W/m², surpassing the power densities obtained with carbon felt, carbon cloth, and carbon paper anodes (94, 95, and 140 W/m², respectively). Moreover, sodium acetate addition distinctly enhances the cell performance. The proposed modifications not only enhance power generation but also address critical challenges faced in MFC commercialization. By utilizing low-cost and renewable resources, such as corn cob and sewage wastewater, MFCs can serve as sustainable and economically viable options for power generation and wastewater treatment. Future research endeavors may focus on optimizing the performance of the corn cob anode, further investigating the long-term stability and scalability of the proposed MFC design, and exploring potential applications in different environmental settings. Overall, the findings of this study contribute to the development of enhanced and cost-effective MFCs, fostering the transition towards a more sustainable and energy-efficient future.

Nomenclature:

MFC	Microbial Fuel Cell
PVDF	Polyvinylidene fluoride polymer
W/m ²	Watt per square meter
OCV	Open circuit voltage
LSV	Linear sweep voltammetry
XRD	X-ray diffraction analysis
SEM	Scanning electron microscope

NaAC Sodium acetate

References

- [1] B.E. Logan, B. Hamelers, R. Rozendal, U. Schröder, J. Keller, S. Freguia, P. Aelterman, W. Verstraete, K. Rabaey, *Environmental science & technology*. 40 (2006) 5181-5192.
- [2] N.A. Barakat, M.T. Amen, R.H. Ali, M.M. Nassar, O.A. Fadali, M.A. Ali, H.Y. Kim, *Polymers*. 14 (2022) 1542.
- [3] N.A. Barakat, R.H. Ali, H.Y. Kim, M.M. Nassar, O.A. Fadali, G.M. Tolba, H.M. Moustafa, M.A. Ali, *Nanomaterials*. 12 (2022) 3961.
- [4] C. Santoro, C. Arbizzani, B. Erable, I. Ieropoulos, *J. Power Sources*. 356 (2017) 225-244.
- [5] A.E. Franks, K.P. Nevin, *Energies*. 3 (2010) 899-919.
- [6] A.A. Yaqoob, M.N.M. Ibrahim, S. Rodríguez-Couto, *Biochem. Eng. J.* 164 (2020) 107779.
- [7] J.V. Boas, V.B. Oliveira, M. Simões, A.M. Pinto, *Journal of Environmental Management*. 307 (2022) 114525.
- [8] A.T. Hoang, S. Nižetić, K.H. Ng, A.M. Papadopoulos, A.T. Le, S. Kumar, H. Hadiyanto, *Chemosphere*. 287 (2022) 132285.
- [9] F.T. Kabutey, Q. Zhao, L. Wei, J. Ding, P. Antwi, F.K. Quashie, W. Wang, *Renewable and Sustainable Energy Reviews*. 110 (2019) 402-414.
- [10] T. Cai, L. Meng, G. Chen, Y. Xi, N. Jiang, J. Song, S. Zheng, Y. Liu, G. Zhen, M. Huang, *Chemosphere*. 248 (2020) 125985.
- [11] R. Selvasembian, J. Mal, R. Rani, R. Sinha, R. Agraphari, I. Joshua, A. Santhiagu, N. Pradhan, *Bioresource technology*. 346 (2022) 126462.
- [12] M.T. Amen, A.S. Yasin, M.I. Hegazy, M.A.H.M. Jamal, S.-T. Hong, N.A. Barakat, *Royal Society Open Science*. 8 (2021) 210996.
- [13] C.-C. Chang, S.-L. Li, A. Hu, C.-P. Yu, *Chemosphere*. 266 (2021) 129059.
- [14] M. Masoudi, M. Rahimnejad, M. Mashkour, *Int. J. Hydrogen Energy*. 46 (2021) 8226-8238.
- [15] Y. Yang, E. Lin, S. Sun, H. Chen, A.T. Chow, *Industrial Crops and Products*. 128 (2019) 70-79.
- [16] H.-Y. Jung, S.-H. Roh, *Journal of nanoscience and nanotechnology*. 20 (2020) 5711-5715.
- [17] A.A. Yaqoob, M.N.M. Ibrahim, K. Umar, J. *Environ. Chem. Eng.* 9 (2021) 106111.
- [18] J.M. Moradian, S. Wang, A. Ali, J. Liu, J. Mi, H. Wang, *Catalysts*. 12 (2022) 894.
- [19] J.M. Khudzari, Y. Gariépy, J. Kurian, B. Tartakovsky, G.V. Raghavan, *Biochem. Eng. J.* 141 (2019) 190-199.
- [20] A. Anjum, S.A. Mazari, Z. Hashmi, A.S. Jatoi, R. Abro, *J. Electroanal. Chem.* 899 (2021) 115673.
- [21] G. Harshitha, A. Sahoo, R. Sethy, *Biocatalysis and Agricultural Biotechnology*. 20 (2019) 101191.
- [22] X. Fan, Y. Zhou, X. Jin, R.B. Song, Z. Li, Q. Zhang, *Carbon Energy*. 3 (2021) 449-472.
- [23] N.A. Erfan, N.A. Barakat, B.J. Muller-Borer, *Colloids Surf., A*. 576 (2019) 63-72.
- [24] M.R. Ali, N.A. Barakat, R. Bassiouny, I.M. El Moghazy, M.M. Ragab, (2020).
- [25] H. Li, Y. Sun, J. Wang, Y. Liu, C. Li, *Applied Catalysis B: Environmental*. 307 (2022) 121136.
- [26] N.A. Barakat, MDPI, 2022, p. 607.
- [27] I.M. Mohamed, V.-D. Dao, C. Liu, N.A. Barakat, H.-S. Choi, *J. Sol-Gel Sci. Technol.* 91 (2019) 342-352.
- [28] D. Liu, Q. Chang, Y. Gao, W. Huang, Z. Sun, M. Yan, C. Guo, *Electrochim. Acta*. 330 (2020) 135243.
- [29] Y. Jin, Y. Wu, B. Li, H. Zhu, Y. Li, M. Zhuang, H. Fu, *IOP conference series: earth and environmental science*, IOP Publishing, 2020, p. 012036.
- [30] W. Liu, Y. Wu, *J. Environ. Chem. Eng.* 9 (2021) 106761.
- [31] S. Naik, S.E. Jujjavarapu, *J. Environ. Chem. Eng.* 9 (2021) 105318.
- [32] T. Foudhaili, T.V. Rakotonimaro, C.M. Neculita, L. Coudert, O. Lefebvre, *J. Environ. Chem. Eng.* 7 (2019) 103149.
- [33] H.O. Mohamed, M. Obaid, A.S. Yasin, J.H. Kim, N.A. Barakat, *RSC advances*. 6 (2016) 111657-111665.