Performance evaluation of microbial fuel cells employing ceramic separator of different surface area modified with mineral cation exchanger

S. Bagchi¹ · M. Behera¹

Received: 27 August 2019 / Accepted: 22 January 2020 / Published online: 31 January 2020
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Abstract

Microbial fuel cell (MFC), one of the emerging topics of research, suffers from two chief limitations, low power generation and high cost of reactor materials. These problems limit its possibility of field scale implementation and commercialization. Hence, an attempt has been made in this study to highlight the utilization of lost cost ceramic material modified with mineral cation exchanger with various concentrations (10–50%) as separator along with studying the role of thickness and surface area in improving the overall performance of an MFC. The performance of the membranes with different thickness and kaolin concentrations was studied based on their proton exchange, ion transfer and oxygen diffusion capabilities. While, proton transfer coefficient was found to be highest in membrane of thickness 4 mm containing 30% kaolin, oxygen transfer coefficient was found to be lowest in the membrane of thickness 6 mm containing 50% kaolin. Thus, MFCs of optimised parameters with concentric anode chamber made with ceramic material were used to study the effect of membrane surface area on their performances. MFC with the highest membrane surface area showed highest power density, highest organics removal efficiency and least internal resistance. Thus, it was concluded from the results that high membrane surface area increases the performance efficiency of the MFCs by maintaining proper proton concentration on both side of the membranes. The results thus highlight the importance of a membrane in an MFC and how the presence of a cation exchanger can enhance the electricity generation.

Keywords Ceramic separator · Ion transport number · Oxygen mass transfer coefficient · Proton mass transfer coefficient · COD removal efficiency · Coulombic efficiency

1 Introduction

Increasing population and environmental pollution have made technologies like microbial fuel cells (MFCs) the next frontier in integrated waste management because of its capability to generate energy in the form of direct electricity from waste. A typical MFC consists of an anode and a cathode chamber. The organic waste is anaerobically oxidized in the anode chamber through the catalytic activity of microorganisms generating electrons, protons, and CO₂. The electrons and protons produced travel to the cathode chamber via the external wire and the membrane,

respectively, producing electricity. Protons in the cathode chamber react with oxygen and electrons to form water. The separator thus is an inevitable component of an MFC. Separators are semi-permeable membranes, which are used in MFCs to physically separate the chambers avoiding the mixing up of the electrolytes. Membrane-less MFCs showed better performance than MFCs with membrane because of less resistance to the flow of proton. However, without a membrane, diffusion of oxygen to anode and substrate to cathode increases, reducing the biocatalytic activity and subsequently the Coulombic efficiency (CE) of the cell greatly. The separators are preferably

✉ M. Behera, manaswini@iitbbs.ac.in; S. Bagchi, sb48@iitbbs.ac.in | ¹IIT Bhubaneswar, Bhubaneswar, Odisha, India.

cation exchange membranes (CEMs), as they have a crucial responsibility of transferring protons across them. The protons produced as a result of oxidation of the substrate in the anode chamber should necessarily be transported to the cathode chamber because otherwise, it would affect the microbial community in the anode compartment by developing acidic pH inside. Other than protons, these membranes transfer other cations such as Na^+ , K^+ , Ca^{2+} , and Mg^{2+} , which are present in much higher concentration than protons in the anode chamber. The passage of these ions across the membrane saturates the negatively charged ions of the membrane blocking the passage of protons. Besides proton and other cations exchange, oxygen and substrate diffusion also occur which are performance limitations of a membrane. Oxygen diffusion across the membrane can kill the obligate anaerobic exoelectrogens, while substrate diffusion can lead to biofouling of membranes and growth of bacteria in the cathode chamber. Biofouling of the membranes can increase the internal resistance, hence decreasing the power output. The fouling layer on a membrane was reported to increase the internal resistance by 20% decreasing the power output. Biofouling of membranes is unavoidable for long term operation of the reactors. It requires periodic cleaning or replacement which increases the capital cost of the operation, heightening the need for investigation of alternate membrane materials [1].

The various kinds of membranes used till date apart from CEMs are anion exchange membrane [2], ultrafiltration membranes [3], bipolar membrane [4], microfiltration membranes [5], etc. Nafion, the most extensively studied membrane has attached hydrophilic sulfonate groups to hydrophobic fluorocarbon backbone, which enhances its conductivity [6]. Other membranes like Nafion namely Hyflon [7] and Zirfon [8] have shown higher internal resistance and higher oxygen permeability than Nafion [8, 9]. While AEMs facilitate proton transfer by using phosphate and carbonate as the carrier, BPMs, offers an added advantage of transport of hydroxyl ions produced due to water splitting [10, 11]. J-cloths containing porous fabrics have also shown successful results, because of the formation of a biofilm layer formed on the surface, which reduces the oxygen diffusion, decreasing the resistance. Similarly, salt bridges containing 2% KCl and 10% agar also showed lesser oxygen permeability, but high internal resistance, making them unsuitable for practical application [12]. Separator electrode assemblies (SEAs) categorized into two types, viz. separator cathode assemblies and sandwich-types SEAs can reduce the internal resistance by reducing the distance between the electrodes [13, 14].

Development of ceramic separators brought a breakthrough in separator research because of its advantages of low cost, chemical, thermal and mechanical stability,

easy synthesis, maintenance, and availability and non-ion selectivity [15–18]. Clayware ceramic membranes have also shown high cation exchange capacity and low oxygen and substrate diffusivity. Modifying the separators using mineral cation exchangers like kaolinite or montmorillonite improved their performance further, because of an increase in the concentration of silica and alumina which after reacting with oxygen provides negative centers to bind the cations [19]. Tamakloe et al. [20] reported higher electricity generation in air cathode MFCs employing ceramic separators than aqueous cathode MFCs. Jana et al. [21] fabricated a concentric MFC using ceramic material as a separator between the anode and cathode chamber, where effluent from the anode chamber was used as catholyte. Jadhav et al. [22] reported further improvement of the effluent by aerobic treatment and the addition of sodium hypochlorite as a disinfectant in the cathode chamber. Other configurations like internal cathode chamber and liter scale MFCs containing more than one membrane in series or parallel were also designed and they showed good results in terms of voltage generation and wastewater treatment [23]. The other low-cost separator materials used till date are coconut shell [24], sulfonated biochar [25], regenerated silk fibroin [26], polyvinyl alcohol [27], polypropylene fabric [28], natural rubber [29], lignocellulosic materials [30], etc., where most of them showed better performance than MFCs employing Nafion as separator.

Thus, this experimental work focuses on optimizing proportions of kaolinite in the membrane, membrane thickness, and membrane surface area. Kaolin proportion and membrane thickness were optimized based on proton, oxygen and ion diffusion across the membrane. The one with higher proton and ion exchange capacity along with lower oxygen diffusion was chosen. Ceramic membranes in MFCs with optimized parameters were then fabricated with different surface areas and their performances were then studied in terms of power generation and COD removal in MFCs.

2 Materials and methods

2.1 Fabrication and characterization of ceramic separators

Ceramic separators of three different thicknesses and five different compositions were fabricated using natural soil and kaolin. Locally available soil was mixed with different percentages of kaolin (10%, 20%, 30%, 40%, and 50%) and the mixtures were filled in molds of different thicknesses (4 mm, 5 mm, and 6 mm). They were placed on a plastic cover while molding and drying to avoid the moisture to hinder the

formation by escaping. They were dried in the open air for 5 days and then in a hot air oven at 150 °C for 2 h and finally in a muffle furnace at 650 °C for 30 min.

Energy-dispersive X-ray Spectroscopy (EDS) of all the soil samples was done using Oxford Instruments, 51-XXM 1004, Germany. The soil samples were first oven-dried at 105 °C for 2 h and then coated with gold before conducting EDS.

Proton mass transfer coefficients for separators were determined using a double-chambered reactor, where the chambers were separated using ceramic membranes of different surface areas and kaolin concentrations as described by Ghadge et al. [19]. One chamber (anode) was filled using deionized water of pH 6.8 and NaOH solution of concentration 0.1 M with a pH of 10.5 was filled in the other chamber (cathode). The change in pH was measured using a pH probe (HACH multi HQ40d). The proton mass transfer coefficient (k_H , cm/s) was then calculated using a formula given in Eq. 1 [31].

$$k_H = -\frac{v}{2At} \ln \left(\frac{C_a + C_c - 2C_p}{C_a} \right) \quad (1)$$

where A is the surface area of the ceramic separator, v is the volume of anolyte, C_a and C_c are the initial proton concentration in anode and cathode chamber, respectively, C_p is proton concentration in the cathode chamber at time t .

The oxygen mass transfer coefficient was measured in the same reactor by sparging nitrogen gas in the anode to make the chamber anaerobic. High dissolved oxygen (DO) concentration was maintained in the catholyte by continuously aerating the chamber. DO probe (HACH multi HQ40d) was used to measure dissolved oxygen concentration at an interval of 15 min. Oxygen mass transfer coefficient (k_o , cm/s) was calculated using the formula in Eq. 2 [32]

$$k_o = -\frac{v}{At} \ln \left(\frac{C_{oc} - C_{oa}}{C_{oc}} \right) \quad (2)$$

where C_{oc} is the DO concentration in the cathode chamber, and C_{oa} is the DO in the anode chamber at time t .

The ion transport number (t_+) was determined by adding different doses of Na^+ ions in both chambers of the reactor described before. The change in potential difference was determined by placing two reference electrodes (Ag/AgCl, +197 mV vs. Standard Hydrogen Electrode, SHE) in both the chambers. The potential difference was then recorded using a digital multimeter (Keysight 34972A) at 3 min interval. The t_+ was calculated using Eq. 3 [33]

$$E_v = -\frac{RT}{F} (2t_+ - 1) \ln \frac{C_1}{C_2} \quad (3)$$

where E_v is the cell voltage (mV) across the separator; T is the temperature (°K); F is the Faraday constant (C mol^{-1}),

R is the molar gas constant, t_+ is the transport number of cations, and C_1 and C_2 are the concentrations of sodium ions in the respective chambers.

2.2 Fabrication and operation of MFC

Three MFCs were fabricated of concentric chamber configuration with anode as inner and cathode as the outer chamber. The anode chambers were made up of clayware ceramic material with optimized soil and kaolin concentration where the walls of the anode chambers acted as proton exchange membrane (Fig. 1). The volumes of anode chambers were different (50 mL-MFC1, 190 mL-MFC2, 240 mL-MFC3) thus ensuring the different surface areas (273.4 cm², 427.3 cm², and 619.7 cm², respectively). Plastic beakers of similar volumes were used as cathode chambers in the case of all the three MFCs. Anode and cathode materials used were stainless steel (110 cm²) mesh and graphite plates (96 cm²) respectively. The anodes and cathodes were connected externally through copper wires with an external load of 100 Ω. Sludge from the sediment of a nearby pond was taken and was used as inoculum for the anode chambers. It was incubated at 35 °C for 1 day before the start-up of MFCs. The sludge was first sieved and then heated for 10 min at 105 °C to suppress the growth of methanogens. Approximately 20% of anode volume was inoculated with sludge and rest of the volume was filled with synthetic wastewater of chemical oxygen demand (COD) of 622 mg/L, 786 mg/L and 3000 mg/L respectively in the three MFCs to have equal organic loading of 0.239 g/d. The composition given by Bagchi and Behera [34] was used to prepare the synthetic feed. The MFCs were operated in batch mode for the first 2 weeks to acclimatize the microbes to the synthetic feed with a feed interval of 3 days. Then continuous mode of operation was started maintaining a hydraulic retention time (HRT) of 15 h.

Fig. 1 Schematic diagram of the reactor

2.3 Analysis and calculation

The performances of MFCs were evaluated in terms of COD removal efficiency and power generation. Daily voltage generation was measured using a digital multimeter (Keysight 34972A) by measuring the voltage across a 100 Ω resistance. The current was calculated by the relation $I = (V/R)$ and power was calculated by the relation $P = (I \times V)$. The power density was calculated using the relation $P.D. = P/S_{A_{an}}$ where $S_{A_{an}}$ is the surface area of the anode electrode. Electrode potentials were measured by connecting the electrodes to a reference electrode (Ag/AgCl, 197 mV vs. SHE). Open circuit potentials were measured after keeping the circuit in open condition for 10 min. The polarisation study was performed under variable external resistance in which resistance was decreased from 5000 to 5 Ω and the corresponding voltages were recorded. For every resistance, current, power, current density (current normalized to electrode surface area) and power density (power normalized to anode chamber volume) were calculated. The internal resistance was considered equal to the slope of the linear part of the voltage versus current curve. Coulombic efficiency (CE) was calculated using Eq. 4 [35]

$$CE = \frac{8I}{Fq\Delta COD} \quad (4)$$

where I is the current generated, q is the flowrate of influent, F is the faraday's constant and ΔCOD is the difference between influent and effluent COD. The COD concentration in influent and effluent was measured using the closed reflux protocol suggested by APHA [28].

3 Results and discussion

3.1 EDS analysis

The EDS analysis of earthenware separators showed an appreciable amount of silica and aluminum as the key inorganic element in a typical soil sample. Highest concentration of SiO_2 (72.1%) was seen in membrane containing 50% kaolin followed by 64.7%, 54.2%, 31.4%, 28% in membrane containing 40%, 30%, 20% and 10% kaolin, respectively. The concentrations of the other major compounds present are given in Table 1. The clay particles contain alumina (Al^{3+}), which upon reaction with oxygen form an octahedral structure with a negative center that attracts and hold positively charged ions. Silica, on the other hand, if present, reacts with oxygen to form a tetrahedral structure. Kaolin contains alternate sheets of octahedron and tetrahedron in every layer making its cation exchange

Table 1 Concentration of the major compounds present in the soil samples

Elements	10%	20%	30%	40%	50%
SiO_2 (%)	28.0	31.4	54.2	64.7	72.1
Al_2O_3 (%)	21.23	19.1	34.63	34.2	37.91
K (feldspar) (%)	0.672	1.03	2.19	1.78	1.21
Fe (%)	8.6	7.14	11.4	13.4	14.8
Ti (%)	0.65	0.73	0.76	0.78	0.81
MgO (%)	0.33	0.48	0.56	0.69	0.77

capacity (CEC) to be 15–20 meq/100 g [19]. Thus, the higher the concentration of silica, the higher will be the cation holding and exchanging capacity of the membrane.

3.2 Proton and oxygen mass transfer coefficient

The highest proton mass transfer coefficient was seen in the separator of thickness 4 mm and kaolin composition of 30% by weight (6.63×10^{-4} cm/s). This higher proton mass transfer coefficient can be attributed to less thickness of the separator and the presence of the cation exchanger kaolin. Because of a high concentration of silica, kaolin provides many negative centers, enhancing the transfer of positively charged protons. However, if present in high concentration, it was seen to affect the porosity of the membrane. For example, there was a very sharp decrease seen in the mass transfer coefficient for membranes containing 40% and 50% kaolin for all the thicknesses, which happened because of the reduction in porosity of membranes with an increase in the concentration of kaolin. The highest proton mass transfer coefficient reported in the current work is much higher than the highest proton transfer coefficient obtained by Ghadge et al. [19]. They recorded the highest proton mass transfer coefficient of 8.18×10^{-6} cm/s in earthen separator containing 20% montmorillonite. The separator containing 20% kaolin showed a proton mass transfer coefficient of 5.43×10^{-6} cm/s, which was lower value reported in the presented manuscript for separators of the same thickness and kaolin concentration (Fig. 2). The proton diffusion coefficient noted in the case of membranes containing 0% of any cation exchanger was 3.4×10^{-6} cm/s and for Nafion, it was 2.63×10^{-4} cm/s, which was lower than any of the values obtained in the current work.

Oxygen diffusion from cathode to anode across the membrane was monitored over time using a DO probe. The highest oxygen mass transfer coefficient was seen in the membrane of thickness 4 mm containing 30% kaolin (6.17×10^{-6} cm/s). While the lowest mass transport coefficient was seen in the membrane of thickness 6 mm containing 50% kaolin (1.83×10^{-6} cm/s). Thus, oxygen

Fig. 2 Variation of proton mass transfer coefficient of separator with different thickness and kaolin concentration

transport across the membrane was also seen to be highly dependent on the thickness and porosity of the membranes. Chae et al. [36] reported the oxygen mass transfer coefficient of Nafion to be 2.8×10^{-4} cm/s, whereas, Ghadge et al. [19] measured it to be 0.49×10^{-3} cm/s and 31.6×10^{-5} cm/s, respectively. The oxygen mass transfer coefficients measured in the current study for the separators of all thicknesses and kaolin concentrations were lower than the values of Nafion reported earlier. While, Behera and Ghangrekar [15] measured the oxygen mass transfer coefficient of a clayware separator of 3 mm thickness to be 0.79×10^{-6} cm/s, Ghadge et al. [19] measured it to be 0.43×10^{-6} cm/s for a control separator of 4 mm thickness. The values of the oxygen mass transfer coefficient reported in the current study (Fig. 3) are higher which might have happened because of the difference in reactor configuration and soil composition.

3.3 Ion transport number

According to the results obtained from proton and oxygen diffusion tests, thickness and kaolin concentration were optimized to be 5 mm and 40%, respectively to have high proton mass transfer and less oxygen mass transfer. Thus, the ion transport number of a membrane of thickness 5 mm containing 40% kaolin concentration was measured and was compared to that of a control membrane. Membrane with 40% kaolin showed a higher ion transport number of 0.47, while the membrane without kaolin showed a maximum ion transport number of 0.38. However, the values are quite less compared to Nafion and membrane containing 20% montmorillonite which has a cation transport number of 0.97 and 0.96, respectively [19].

Fig. 3 Variation of oxygen mass transfer coefficient of separator with different thickness and kaolin concentration

The values reported in the current manuscript (Fig. 4) are lower than the values previously reported because of the difference in reactor configuration and soil composition.

3.4 Voltage and power generation in MFCs

At open circuit conditions, the maximum OCV of 800 mV, 809 mV and 857 mV in MFC1, MFC2 and MFC3, respectively were recorded. Average power densities of 93.2 mW/m^2 , 96.5 mW/m^2 and 100.1 mW/m^2 were recorded for MFC1, MFC2 and MFC3, respectively. After the 45th day, the electricity output in terms of voltage started becoming constant. The better performance in terms of power generation observed in the case of MFC3 can be attributed to higher membrane surface

Fig. 4 Variation in ion transport number with time of separator with and without kaolin

Fig. 5 Change in voltage generation with time

area which helped in preventing pH splitting thereby providing the exoelectrogens with a more suitable environment to grow and generate more electrons. Figure 5 shows the change in voltage generation observed with respect to time during the operation of MFCs in continuous mode. Better performance of MFC3 than MFC2 and MFC1 was further ensured by both high anode and cathode potentials measured in MFC3 followed by MFC2 and MFC1 sequentially. Higher anode and cathode potentials show better oxidizing and reducing conditions maintained because of proper maintenance of pH in anode and cathode chambers. The average pH measured for anolyte throughout the operations of MFC1, MFC2, and MFC3 was 6, 6.2 and 6.6, respectively. Higher pH in the anode chamber of MFC3 shows better proton transfer across the membrane because of the higher membrane surface area. However, after approximately 1 month of the continuous mode of operation, the voltages were found to reduce in all the three MFCs. This might have happened because of methanogenesis, which is often seen in the case of long term operation of MFCs [37]. The parameters for measuring the electrical performance of the microbial fuel cell showed higher values than reported in previous studies because of optimized kaolin concentration and thickness of the separator. For example, the maximum power density reported by Harnisch et al. [11] was 24.32 mW/m^2 , which was significantly lower than the maximum power densities noted in the current study. The highest power generation reported by Ghadge et al. [19] from the reactor with separator containing 20% montmorillonite showed a maximum power density of 142 mW/m^2 , which was comparable to the highest power generated in the current study (100 mW/m^2).

3.5 COD removal efficiency and Coulombic efficiency

Average COD removal efficiencies of 58.54%, 60%, and 74.31% were seen in MFC1, MFC2, and MFC3, respectively. The steady-state condition in terms of COD removal efficiency was seen on 50th, 55th and 58th day for MFC1, MFC2, and MFC3, respectively. The highest COD removal efficiency in MFC3 can be again attributed to the highest membrane surface area enhancing the microbial growth by improved proton transport thereby preventing the lowering down of pH in the anode chamber. Average coulombic efficiencies of MFC1, MFC2, and MFC3 obtained were 45.4%, 20.7%, and 4.3% respectively. MFC1 was observed to have the highest coulombic efficiency due to the lowest flowrate owing to the least volume and least COD removal efficiency. Figure 6 shows the variation in COD removal efficiencies observed with time during the operation. However, the values were found to be lower than reported by Behera and Ghangrekar [15], who reported higher COD removal efficiency by MFCs of earthen separator thickness ranging from 3 to 8.5 mm [11]. This might have happened because of longer HRT, which might have given the bacteria longer time to oxidize the substrate.

3.6 Polarization

Polarization studies were performed by varying the external load, once the cell reached stable state condition. A maximum power density of 61.23 mW/m^2 was obtained in MFC3. While the maximum power densities in MFC2 and MFC1 were 21.85 mW/m^2 and 10.27 mW/m^2 respectively. The internal resistances of MFC1, MFC2 and MFC3 were measured to be 77.31Ω , 42.85Ω , and 36.08Ω , respectively.

Fig. 6 Change in COD removal efficiency with time

which was much lower than the values reported by Harnisch et al. [11]. Higher power density in the MFC with the highest membrane surface area (MFC3) shows better performance because of improved microbial activity due to suitable growth conditions. The lowest internal resistance obtained in MFC3 denotes lower Ohmic losses because of better transport of ions across membranes.

4 Conclusion

A microbial fuel cell is an integrated waste management technology whose performance can be improved and the manufacturing cost can be reduced by using clayware separators. Earthen separators having different thicknesses and different concentrations of mineral cation exchangers were thus tested in terms of proton diffusion, oxygen diffusion and ion transport across the membranes. All these parameters were found to be highly dependent on the separator thickness and kaolin concentration. The separator of 4 mm thickness containing 30% kaolin showed the highest proton mass transfer coefficient, whereas, the membrane of thickness 6 mm containing 50% of kaolin showed the least oxygen diffusion coefficient. Thus, the thickness of 5 mm and kaolin concentration of 40% were optimized and were used to fabricate membranes of different surface area to see the effect of surface area on the performance of MFC. The MFC with the highest surface area showed the highest OCV of 857 mV, the highest power density of 100.1 mW/m² and the lowest internal resistance of 36.08 Ω. This shows better performance of the MFC because of improved biocatalytic activity and lesser Ohmic losses owing to better microbial growth conditions and enhanced ion transport.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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