Design and Evaluation of a Scaled-up Microbial Fuel Cell Operating with Blackwater

by

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Master of Science

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ABSTRACT

Energy can be harvested from wastewater using microbial fuel cells (MFC). In order to increase power generation, MFCs can be scaled-up. The MFCs are designed with two air cathodes and two anode electrodes. The limiting electrode for power generation is the cathode and in order to maximize power, the cathodes were made out of a C-N-Fe catalyst and a polytetrafluoroethylene binder which had a higher current production at -3.2 mA/cm^2 than previous carbon felt cathodes at -0.15 mA/cm^2 at a potential of -0.29 V. Commercial microbial fuel cells from Aquacycl were tested for their power production while operating with simulated blackwater achieved an average of 5.67 mW per cell. The small MFC with the C-N-Fe catalyst and one cathode was able to generate 8.7 mW. Imitating the Aquacycl cells, the new MFC was a scaled-up version of the small MFC where the cathode surface area increased from 81 cm^2 to 200 cm^2. While the MFC was operating with simulated blackwater, the peak power produced was 14.8 mW, more than the smaller MFC, but only increasing in the scaled-up MFC by 1.7 when the surface area of the cathode increased by 2.46. Further long-term application can be done, as well as operating multiple MFCs in series to generate more power and improve the design.

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CHAPTER 1

INTRODUCTION

An improvement in recent sustainable technologies, is being able to recover energy from wastewater through biotechnology. Wastewater is abundant wherever humans reside and is traditionally seen as a "waste." Wastewater is typically transferred from the source of generation to a wastewater treatment plant to remove high levels of Biochemical Oxygen Demand (BOD) and nutrients (N and P) that contribute to excessive growth of bacteria and algae in bodies of water. As fossil fuel use increases, the need for renewable resources to support the growth of populations is needed. The energy content present in wastewater can help offset the cost and energy associated with its treatment if successful energy-recovery technologies are implemented.

In remote and non-permanent settings, it is common to separate wastewater based on two major sources. Blackwater is often considered a mixture of toilet and kitchen wastewater, containing a high solids, nutrients, and BOD concentration: 91% total nitrogen, 71% COD, 80% phosphorous, and 82% potassium of total domestic wastewater (Kujawa-Roeleveld and Zeeman, 2006). Greywater is wastewater derived from sinks, showers, and laundries and contains lower BOD and nutrient loadings. In these settings, blackwater is a suitable source for energy recovery technologies, such as microbial fuel cells (MFC) to generate an electrical current. The amount of energy available in the United States from biomass is 7.5 EJ which supports 7% of the population's energy use using statistics from 2005 (Rittman et. al., 2008). Blackwater can be diverted straight from the source and be applied in MFCs.

MFCs are a bio-electrochemical technology that can use blackwater as the substrate. As the anode-respiring bacteria (ARB) consume electron donors, they emit electrons that are collected on the anode and sent to the cathode, creating a current. This occurs because of the potential gradient between the oxidation-reduction reactions on both the anode and the cathode. The electron acceptor is oxygen, which passes from the air-cathode into the cell (Ucar et al., 2017). The ARB attach themselves to the anode fibers and oxidize organics, where the following equation represents the process with acetate as the electron donor.

$$CH_3COO^- + 4H_2O \leftrightarrow 2HCO_3^- + 9H^+ + 8e^-$$

The extracellular electron transfer of the ARB in their membranes allows for the reaction to occur.

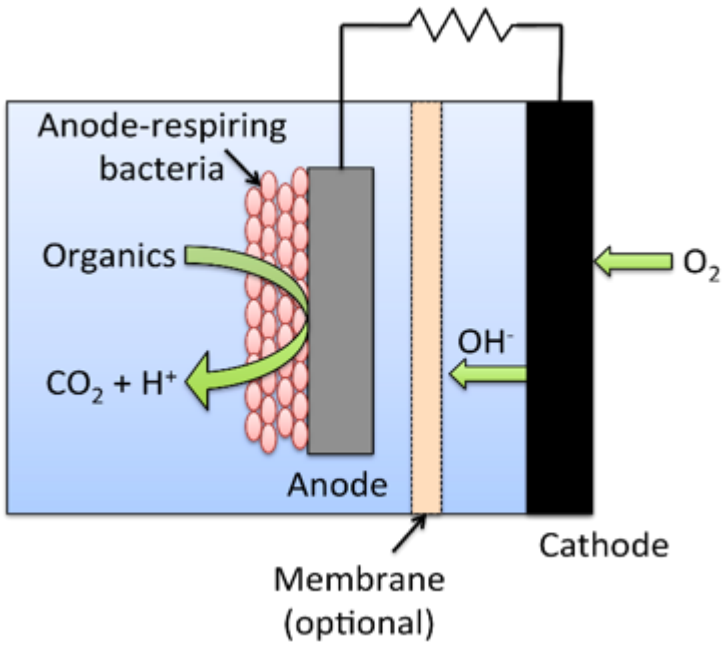

Figure 1: MFC set-up (from the Biodesign Institute).

For the anode and cathode to be efficient, it should be electrically conductive, have a high surface area, be resistant to corrosion, and be economical. Common anode materials include those made out of carbon and/or metal sheets (Santoro et al., 2017). The anode is the working electrode in order to set the cell at an ideal voltage where certain highly-conductive bacteria, such as *Geobacter*, can thrive and contribute to higher current densities (Torres et. al., 2009). Lower ranges of anodic voltage, -0.4 to -0.2 V vs. SHE, allow a higher concentration of ARB to accumulate on the anode instead of a diverse group of bacteria.

For the cathode catalyst, activated carbon has shown to improve the oxygen reduction reaction (ORR).

$$O_2 + 4H^+ + 4e^- \leftrightarrow 2H_2O$$

The air cathode allows oxygen to come in and react with protons to create water. While platinum has been a common material to use for the catalyst of a cathode as well as for support, it is more economically feasible to use nickel or stainless steel for the support and current collector while attaining the same or better current. The catalyst is then added to the support as C-N-Fe. When wanting to increase the size and power generation of a MFC, it is advantageous to use materials that work well at a cheaper price.

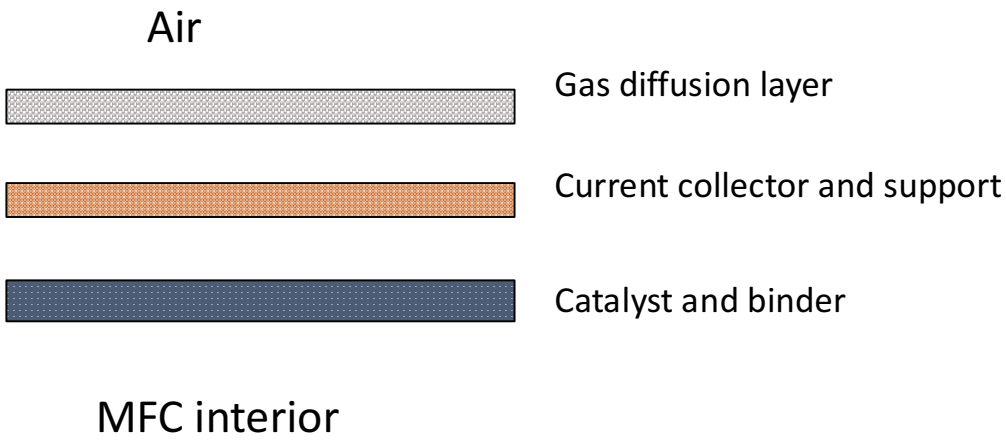

Figure 2: Structure of a Cathode.

In order for cathode performance to be measured, all of the cathodes made were placed in half-cells with 50 mM phosphate buffer. Linear sweep voltammetry (LSV) is applied to record the current density of the cathode at different voltages of the cell.

Throughout the project, progress has been made with creating cathodes from a new recipe using a Fe-N-C catalyst (Yang & Logan, 2016) that allows for an 8.75 times higher current than previous cathodes that were solely made out of carbon felt. Using a nickel mesh instead of a stainless-steel mesh for the structure of the cathode also slightly increased the productivity and reduced rusting while operating in the MFC. The cathode is the limiting factor within an MFC since there is overpotential and leaking that can occur depending on the material of the cathode. The distance between the cathode and the anode must be small to increase conductivity and reduce resistance.

Our MFC designs are compared against commercially available MFCs provided by Aquacycl (“Aquacycl”, n.d.). The BETT reactors have been commercialized for the treatment of high-strength wastes, including wastewater sludge and/or blackwater. This

modular design has been shown to be resilient and achieve high rates of BOD removal. While the main goal for the project is to maximize power production from blackwater, the Aquacycl reactors are designed to maximize the rate of treatment by using carbon-based electrodes that stimulate hydrogen peroxide production.

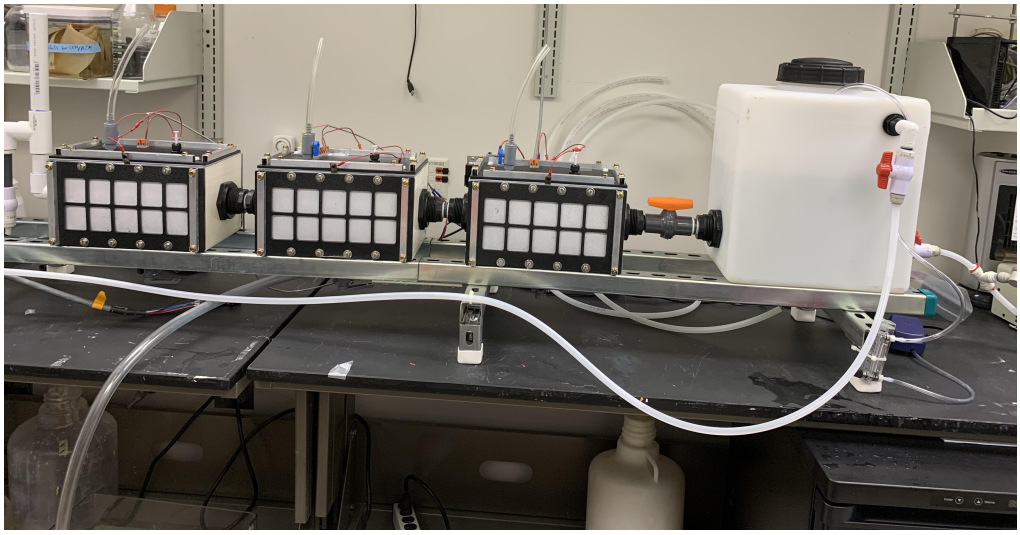

Figure 3: BETT cell set-up (photo by Ethan Howley).

The operation of an MFC includes feeding blackwater, recording COD, recording pH, and analyzing the current density and potential outputs. Simulated blackwater is made out of thickened sludge and fed every 2 days to keep the organics level in the MFC high, current density steady, and imitating constant circulation.

All previous working MFCs have also been constructed and operated with a single cathode and anode. This study includes the increase in the size of the MFC where the active surface area of one cathode increased from 81 cm^2 to 200 cm^2 as well as increasing the number of anodes and cathodes used in the MFC to two each. This was inspired by the Aquacycl commercial MFCs whose purpose is to treat the wastewater that flows through it. While it treated the blackwater, the cells were also tested for power

production in a series of three, yet produced less than the smaller MFC with the C-N-Fe cathode. The success of the smaller MFC led to the upscale of the MFC to act like the Aquacycl cells in series, but with cathodes made from the recipe proven to have higher power generation.

The goal is to increase the amount of power linearly equivalent to the increase in surface area, which is 2.46 the amount of power as the previous MFCs since two larger cathodes are in production. For three of the new MFCs to run in series, it will theoretically produce 100 mW of power.

CHAPTER 2

METHODS

Cathode:

The C-N-Fe catalyst used in the experiment is derived from (Yang & Logan, 2016) with modifications to the cathode preparations to accommodate larger designs. To prepare the catalyst, 6.0 g of Carbon Black Vulcan XC-72 from Fuel Cell Earth, 1 g of iron(III) chloride anhydrous $FeCl_3$ from Ward's Science, 1 g of 1,10-phenanthroline from Sigma-Aldrich, and 75 mL of water were stirred in a beaker at 60 °C for thirty minutes. The solution was then placed in an oven at 95 °C until dry. The dried catalyst was then placed in five Coorstek 60121 crucibles which were closed by wrapping stainless steel around the lid and bowl. They were then placed in the chamber of a high-temperature vacuum tube furnace OTF-1200X for pyrolysis. The chamber was flushed with nitrogen gas for around 5 minutes so that no oxygen remained before heating. To begin the heating ramp, the temperature of the chamber was held at 75°C for 5 minutes, a 30-minute ramp to get to 400°C, 30 minutes to get to 800°C, and finally at 800°C for 15 minutes. To cool down the chamber, it was set to lower to room temperature in 30 minutes. The catalyst was removed from the crucibles and mixed in a beaker with 0.01 M hydrochloric acid. After mixing for 30 minutes, the catalyst was left to settle out and the solution on the top was removed with a pipette. After the excess liquid is removed, the catalyst is left to dry in a fume hood. Once dry, the powder was crushed using a Coorstek 60310 mortar and pestle. Then they were sieved through a nickel 200 wire mesh.

In order to make the catalyst bind to the nickel mesh, around 1 gram of catalyst powder was added to a beaker along with around 10 milliliters of pure ethanol. The

beaker sat in a sonicator, and while sonicating, polytetrafluoroethylene (PTFE) was added to the beaker. The ratio of catalyst to PTFE is from Yang & Logan (2016) where 1:1 is 1 g of catalyst to 0.67 mL of PTFE. For the catalyst layer on the inside of the cell, a 1 to 1.5 ratio of catalyst to PTFE is needed so 1 gram of catalyst and 1 mL of PTFE. For the catalyst layer on the outside of the cell, a 1 to 6 ratio is used so 1 gram of catalyst to 4 mL. No less than 8 mL and no higher than 12 mL of ethanol is recommended for ratios of 1:1 to 1:6.

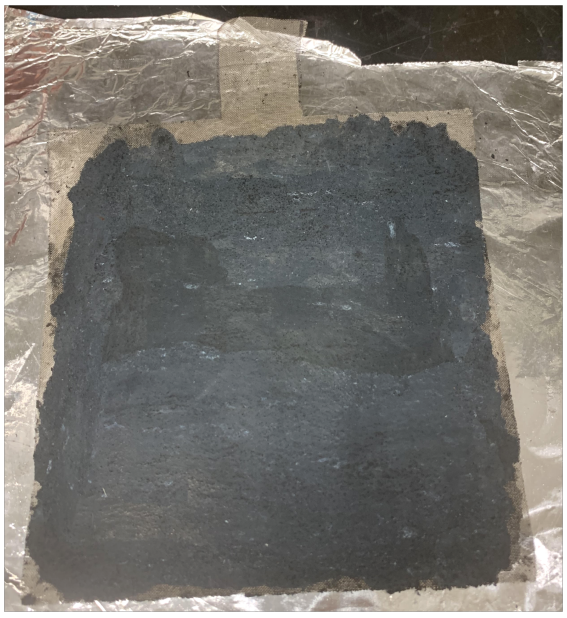

Figure 4: Image of Cathode with C-Fe-N catalyst and PTFE binder.

Linear Sweep Voltammetry (LSV):

To run initial LSV tests, the MFC to be tested were assembled with only the cathodes. Each MFC has an anode chamber volume of 500 mL. Each cathode does an LSV measurement individually. The chamber was filled with a 50 mM phosphate buffer. A nickel mesh was placed in the buffer without contact to other electrodes as well as an

AgCl reference electrode. The cathodes are connected as the working electrode and the nickel mesh as the counter electrode. While consistently mixing the buffer on a stir plate, the range of the sweep is from cathodic open circuit to -0.35 V vs Ag/AgCl at 1.00 mV/s. When taking sweeps consecutively, the cell rested for 5 minutes at open potential between sweeps.

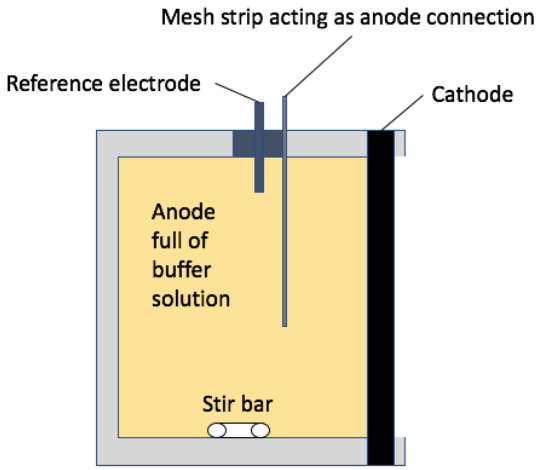

Figure 5: Half-cell set-up for LSV.

MFC Anode design:

The anode was prepared with carbon fibers and a titanium plate that has been cut to have 6 by 6 two-centimeter squares (*Figure 6*). A bundle of 2,000 carbon fibers were weaved through the squares diagonally. Once the ends were tied off, then the coating on the fibers were removed by going through the following process: soak in 1 molar nitric acid for three hours, then soak in 1 molar acetone for 12 hours, then soak in 1 molar ethanol for 12 hours, then rinse off with deionized water.

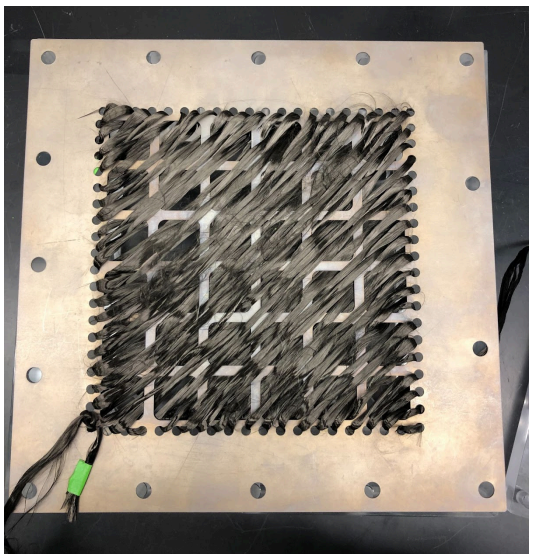

Figure 6: Image of Anode electrode with carbon fiber woven on a titanium plate.

MFC Membrane:

An AMI-7001 anion exchange membrane from Membranes International was used for most experiments. To prep the membrane for the MFC, it was soaked in a 5% NaCl solution for 24 hours to allow it to expand.

MFC build:

The MFC build of the up-scaled cell consisted of a center anode chamber (1.1 L) with an anode/membrane/air cathode assembly on each side (200 cm^2 each). The cathode was placed with the lower PTFE content towards the anode, while the higher PTFE content exposed to air. The MFC is put together in the following order: acrylic end, sponge for support, cathode, membrane, anode, acrylic center, anode, membrane, cathode, sponge, and the other acrylic end. Silicone rubber gaskets are placed in between each rigid component. The chamber of the MFC allowed influent and effluent through

two tubes on the same side. A circular access point on the top of it was sealed with a rubber stopper that the reference electrode and a gas outlet were attached to.

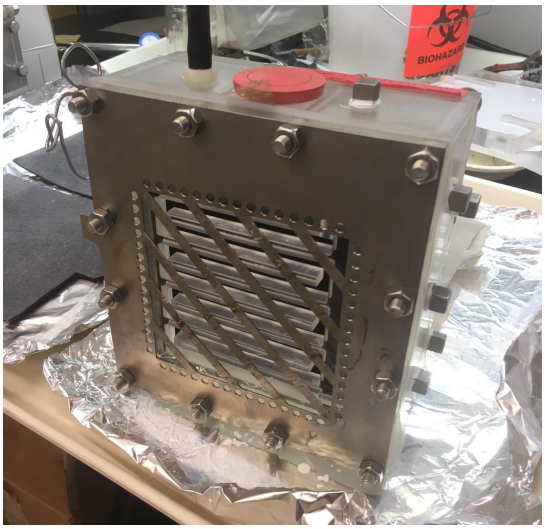

Figure 7: Image of an MFC (photo by Ethan Howley).

Catholyte insertion:

Two small GC (S&L Nylaflow (R) pressure tubing type ⅛”H MFG# 10/18/10 2) 1.5 inch-long tubes at the bottom and top corners of the MFC between the membrane and the cathode were placed to inject about 5 mL of 100 mM sodium hydroxide into the area between to ensure that the area was wet.

Set up for chronoamperometry:

The MFC operation was performed under a constant anode potential to maintain conditions appropriate for microbial growth. The MFC was connected to the potentiostat where the reference electrode was an Ag/AgCl electrode (BASi Instruments), the working electrode was the anode, and the cathode is connected as the counter electrode. The working electrode is set at -300 mV and measurements are taken every minute.

Feeding schedule:

The MFC had a feeding schedule where 120 mL of the blackwater in the anodic chamber was removed and 120 mL of fresh simulated blackwater was added every 2 days, for a hydraulic retention time of ~ 8.33 days. To simulate blackwater, mixed primary and secondary sludge is taken from the Mesa Northwest Reclamation Plant (Mesa, AZ). Then 268 mL per liter of mixed sludge is combined with 0.84 grams per liter of sodium bicarbonate, 0.77 grams per liter of ammonium chloride, and 732 mL per liter of water. The sodium bicarbonate brings up the pH of the mixture to stay neutral around 7 in order for the bacteria to grow optimally within the cell. The COD concentration of the simulated blackwater is around 10,000 mg/L. The amount of thickened sludge was increased to 270 mL after COD analysis of the formula in order for the fresh simulated blackwater to have 10,000 mg/L of COD available to achieve the desired power output.

CHAPTER 3

RESULTS AND DISCUSSION

In order to measure cathode overpotentials, the LSVs are run and compared. Higher current density shows a higher capacity for the MFC to generate power. A carbon felt cathode with PTFE as the gas diffusion layer is shown in *Figure 10* and demonstrates a density curve from -0.4 V to 0.1V. The current density values are low between zero and -0.4 mA/cm^2. The voltage at which to take note of the current density is around -0.3 V since that is the set voltage for which the MFC operates.

A newer cathode made with nickel and the C-N-Fe catalyst with PTFE binder has a current density curve with a greater range shown in *Figure 10*. At -0.3 V the current density is at -3.2 mA/cm^2 showing higher current being conducted with this new cathode structure.

Three BETT reactors were provided by Aquacycl for evaluation. Each reactor consists of a 1-gallon anode chamber with several carbon fiber brushes used as anodes and carbon cathodes on each side of the chamber. The details of the electrodes were not provided by the company. All three reactors were connected in series (*Figure 8*) along with an 8 L holding tank. The holding tank has an air sparger to oxidize sulfides in the wastewater and minimize their precipitation at the cathodes. The reactors were agitated by blackwater recirculation using a peristaltic pump at 50 mL/min. The feeding of BETT reactors, using the blackwater recipe described, was performed through pumping from a refrigerated feed tank (50 L).

Each BETT reactor was connected to a variable resistor. Each week, the resistor was changed to match the maximum power output from a j-V curve. The resulting power generation is seen in *Figure 9*.

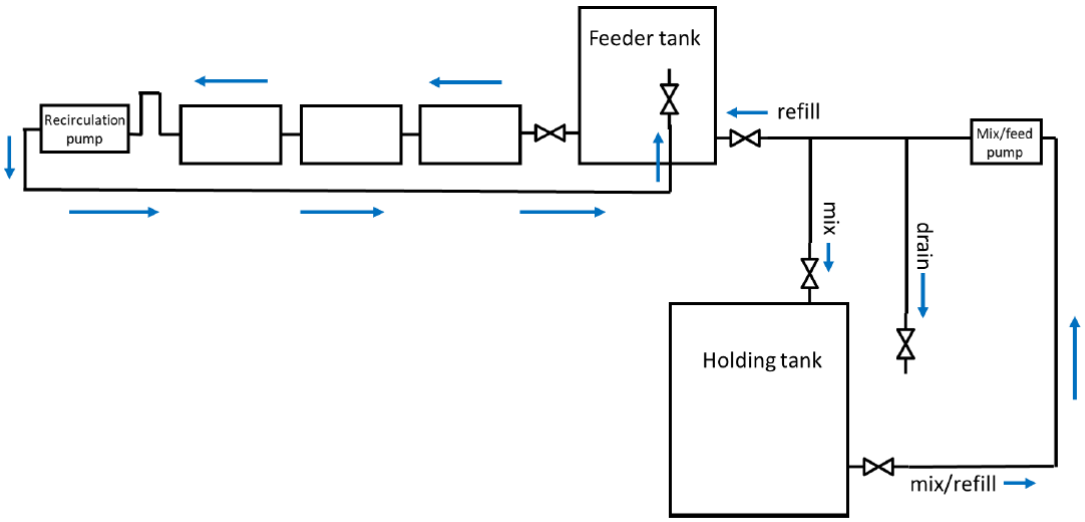

Figure 8: BETT reactors set up in series. The recirculation pump is run continuously, while refill/drain lines are used to feed the reactor from the holding tank containing blackwater at 4 ^{o}C.

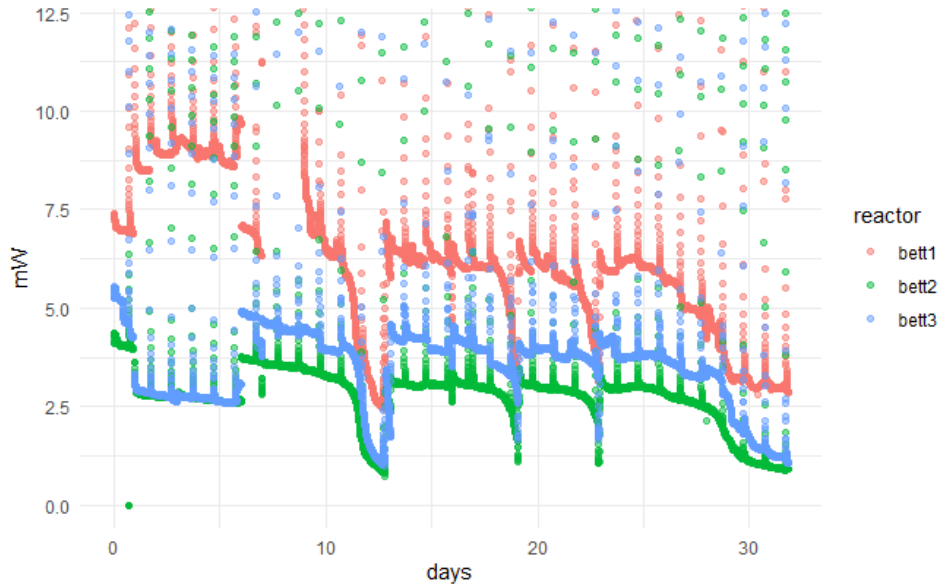

Figure 9: Power generation of BETT cells under steady-state conditions (graph by Ethan Howley).

While running the MFC, the average power that the cell was able to accomplish was 8.7 mW. Compared to the commercial BETT cells, which on average were able to generate 5.67 mW from all three cells. When comparing the two using the surface area of the cathodes, the smaller MFC has much greater power capabilities.

A newer cell was made by increasing the size of the cathode and the MFCs themselves. The cathodes are increased from 81 cm^2 to 200 cm^2 of surface area available. Imitating the commercial cells, the bigger MFC will also include two cathodes on opposite ends of the cell. Now the MFC can hold 1.1 L of simulated blackwater.

From the LSV in *Figure 10*, the bigger cathode can carry a current density which is lower than that of the smaller cathode. The current density for the bigger cathode does decrease because of the increase in surface area, which is expected.

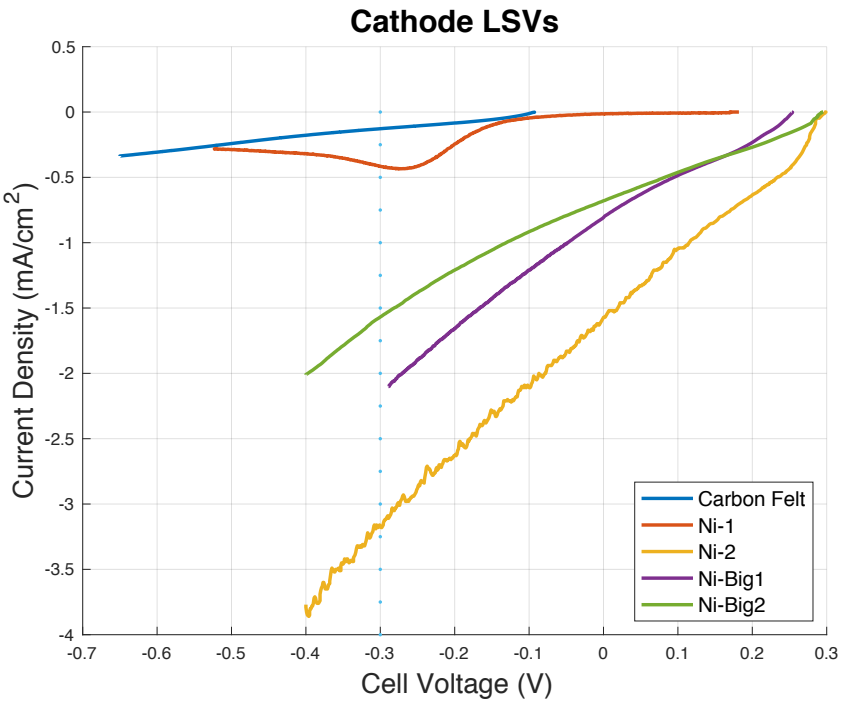

Figure 10: Cathode LSVs. Carbon felt with a PTFE gas diffusion layer, (Ni-1) nickel mesh with catalyst and PTFE on the inside with a PTFE gas diffusion layer, (Ni-2) nickel mesh with catalyst and PTFE on both sides with a 1:1 ratio on the inside and 1:3 ratio on the gas diffusion layer, and (Ni-Big1 and Ni-Big2) scaled-up nickel mesh with catalyst and PTFE on both sides with a 1:1.5 ratio on the inside and 1:6 ratio on the gas diffusion layer.

The cathodes that were made with the C-N-Fe catalyst and PTFE binder work better than those previously made out of carbon fiber. The current density of the 200 cm^2 and 81 cm^2 cathodes at -290 mV are -2.10 mA/cm^2 and -3.11 mA/cm^2 respectively. When multiplying by the area of the bigger and the smaller cathode, the current going through them are 420.0 mA and 251.9 mA. The surface area increased by 2.46, yet the current only increased by 1.67.

A reason for why the current is not equivalently increased, may be because of the difference in mass of the two cathodes. There was more catalyst and binder mixture rolled onto the larger cathode in order to ensure against leaking. For this same reason, a

higher amount of PTFE was used in the mixture, which can increase resistance in the cathode. There was also higher water pressure on the cathodes which led them to bulge out from the MFC. The larger MFC held 1.1 L of blackwater while the smaller MFC held 0.5 L. In order to keep the cathodes from bulging and creating holes in the structure, a sponge was placed between the cathode and the acrylic support on the outer ends of the MFC.

While the MFCs are in operation, the cathode is under different conditions than the half-cell with phosphate buffer and acts differently.

A problem that arose around 5 months after starting is the corrosion of the cathode. Although the pH level of the MFC was around 7 for the entire length of the experiment, the cathode degraded over time. There was green residue on the cathode due to the nickel being oxidized into Ni^{2+}. At one point of the experiment, the cell started working in a reduction state rather than an oxidation state which may have created conditions in which the carbon and the nickel react with each other. Visible leaking was noted to be coming from the cathodes after the membrane was removed, which also decreased the potential of the cathode.

When the bigger MFC was running with simulated blackwater, the current and potential of the cell was measured using chronoamperometry. At first, the anionic membrane was used between the cathode and anode in order to allow selective ions to react at the cathode. It also kept the cathode from being damaged from solids in the blackwater. This does increase the resistance which in turn reduces the current.

$$Current\ (I) = \frac{Voltage\ (V)}{Resistance(R)}$$

The current density resulting from running the MFC in this state can been seen in *Figure 10* where the peak current density was at 0.95 mA/cm^2. The current density levels were stable over the course of 86 days, but slowly decreased to 0.01 mA/cm^2 with regular feeding of blackwater. When this occurred, the cell was fed 100 mM Acetate solution to provide electron donors for the bacteria on the anode and revive them. When feeding the acetate, the current density increased to a peak of 0.26 mA/cm^2. When the cell stopped being fed acetate and continued feeding of blackwater it continued to produce current density at a stable rate over 41 days.

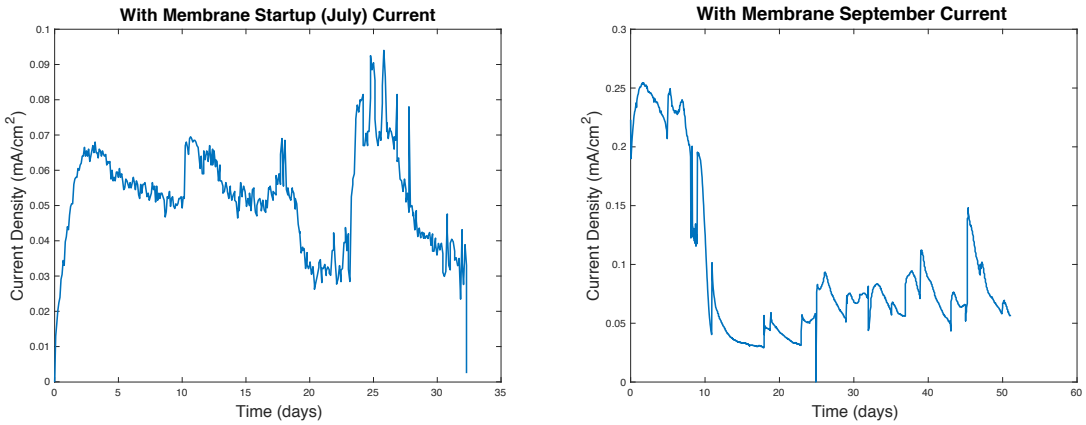

Figure 11: Current density of MFC with membranes in place. (Left) Current density of the MFC for the first 33 days. (Right) Current density when acetate was added and stopped after 10 days.

The MFC then had the anion membrane removed with intent to increase the current density of the cell. Fresh sludge was added to the cell after removing the membrane and putting cloth in its place. The current density increased right after the membrane was removed, but after a day, decreased when the cell went into a state of reduction (*Figure 12 - left)*. Feeding the MFC the usual amount of blackwater every two days did not change the cell back into being in an oxidation state, so 100 mL of thickened

sludge was added to increase the percentage of organics as well as adding new bacteria and the cell was left in open circuit. After 13 days, the cell began producing current again, but it was lower than the current at the initial removal of the membrane as well as when the membrane was in place. The current density declined to 0 mA/cm^2 98 days after reviving the cell.

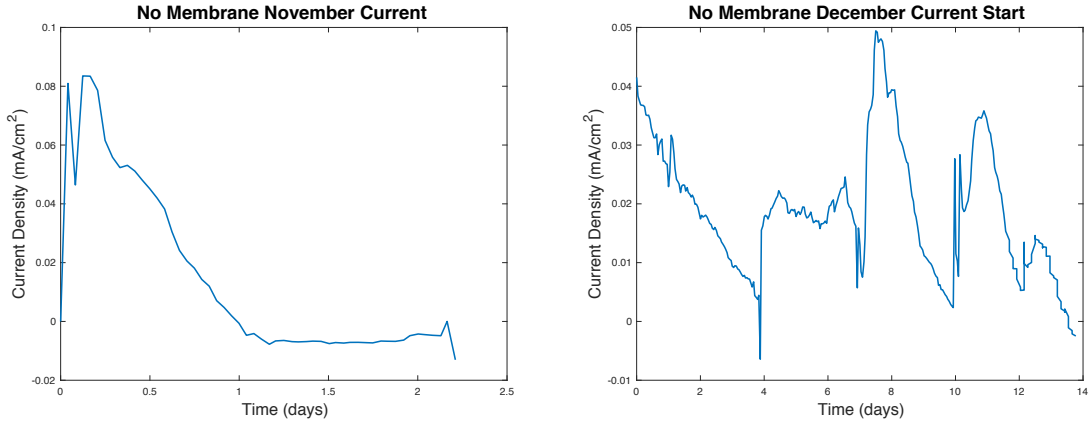

Figure 12: Current density of MFC with no membranes in place. (Left) Membrane was removed and after 0.5 days the current density dropped below zero. (Right) Current density of the revived MFC after it was left at open circuit and 100 mL of sludge was added.

The anionic membrane within the cell should facilitate the transfer of anions from the anodic chamber to the cathodic chamber (Ramirez-Nava et al., 2021). It also keeps organic material from reaching the cathode. The selectively permeable nature of the membrane can be increase the resistance of the cell which would decrease the current. When the decision was made to remove the membrane, it would have allowed for a higher current to come out of the cell. This did happen immediately after removing the membrane, but the membrane was also protecting the anaerobic conditions of the anodic chamber. When the membrane was removed, the anaerobic bacteria that were growing on the anode had a sudden change of environment. The cell had to stabilize with new

bacteria that could grow in these conditions, and were able to do so once 100 mL of thickened sludge was added to the MFC.

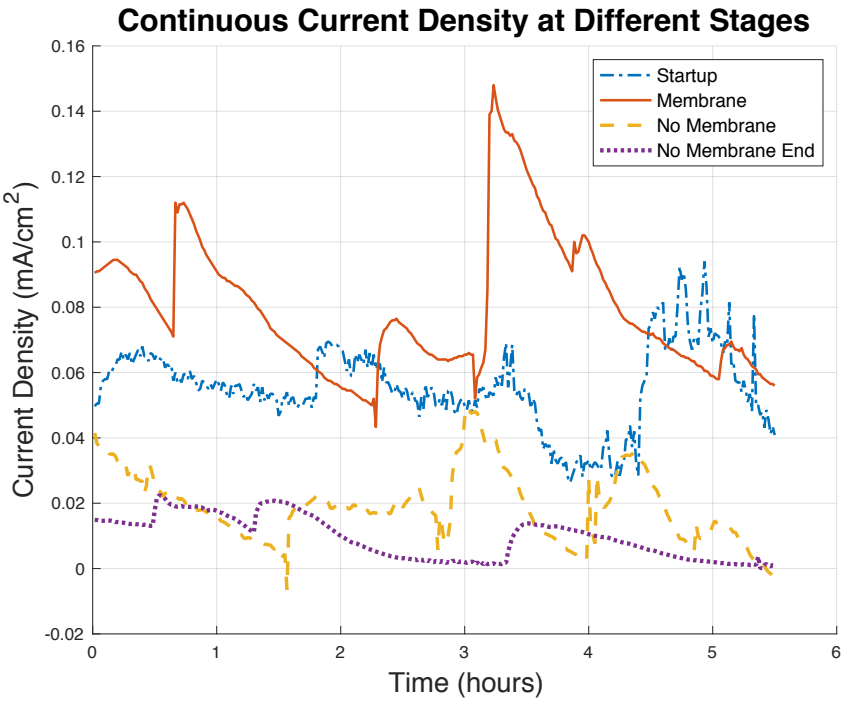

Figure 13: Comparing current densities of MFC at different stages of operation: startup with a membrane, steady-state with a membrane, removal of the membrane, end of run with no membrane.

The COD of the blackwater going into and coming out of the cell was monitored, and based on the original blackwater recipe, the COD going in was around 6,000 mg/L. By doubling the amount of thickened sludge in the simulated blackwater recipe, the input COD was able to reach 10,000 mg/L.

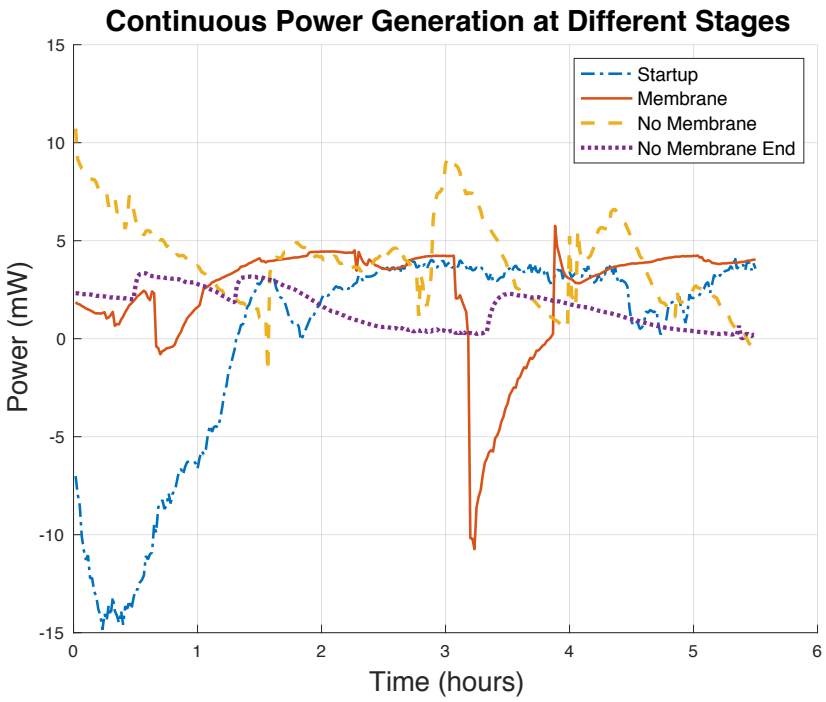

Figure 14: Comparing power generation of MFC at different stages of operation.

Figure 14 shows the power generated in a time snap-shot of each scenario. High levels of power around 14 mW were achieved at startup and after the MFC was fed acetate, both scenarios had the membrane in place. There were lower levels after the membrane had been removed.

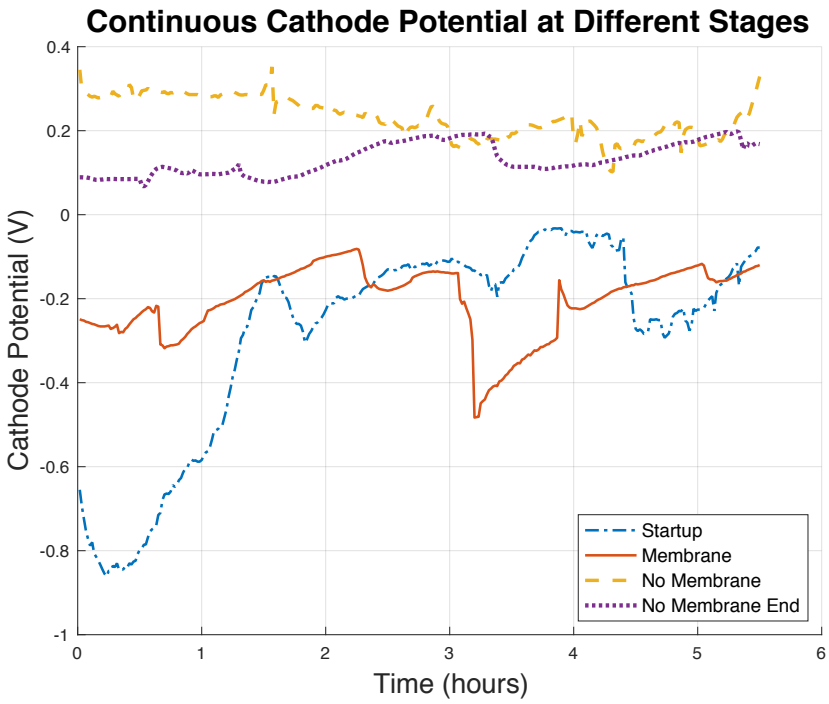

Figure 15: Comparing cathode potentials at different stages of operation.

The cathode potential in *Figure 15* shows how well the cathode is performing. If the overpotential of the cathode is high, then the potential of the cathode becomes more negative. This makes the cathode potential become closer to the anode potential of -300 mV. There is more power production when the difference between the anode and cathode potentials is larger as more energy is released as electrons transfer between the two. Removing the membrane had reduced the resistance as the cathode potential increased (became more positive), but when compared to the impact that it had on the cell as a whole, it was more harmful than beneficial.

CHAPTER 4

CONCLUSION AND FUTURE RECOMMENDATIONS

The scaled-up MFC was able to generate more power than the smaller ones, but there were problems with the design that limited its capabilities. Leaking continues to be a time-consuming problem, as the cathodes must be leak tested over 24 hours multiple time before using in an MFC. Once the cathodes have shown to not leak within this time period, it could start to leak over the use of the MFC. Preventing leaking is a priority in future experiments.

An alternate experiment would be starting a cell without a membrane in case the low power production was due to the cathode or anode being damaged in the process of removing the membrane. It would also start the MFC in an environment that does not change where bacteria are accustomed to the levels of oxygen coming through the cathode.

Future experiments should monitor the power generation of three scaled-up MFCs in series to mimic the BETT cells and compare output. New carbon-based cathode development that can both treat wastewater and generate energy would be beneficial and cost effective.

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