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Voltage reversal during microbial fuel cell stack operation

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Abstract

Microbial fuel cells (MFC) can be used to directly generate electricity from organic matter, but the voltage produced by a single reactor is only ~0.5 V. Voltage can be increased by stacking cells, i.e. by linking individual reactors in series, as is commonly done with hydrogen fuel cells, to provide a higher voltage output. A two-cell air-cathode MFC stack tested here produced a working voltage of 0.9 V (external load 500 Ω) and had an open circuit voltage (OCV) of 1.3 V when operated in fed batch mode under substrate-sufficient conditions. When multiple cells are stacked together, however, charge reversal can result in the reverse polarity of one or more cells and a loss of power generation. We investigated the causes of charge reversal and the impact of prolonged reversal on power generation using a two air-cathode MFCs stack. When voltage began to decline at the end of a fed batch cycle, we observed voltage reversal with one cell producing a working voltage of 0.6 V, and the other cell having a reversed voltage of −0.58 V, producing only a minimal stack voltage of 0.02 V. The reason for the voltage reversal was shown to be fuel starvation, resulting in a loss of bacterial activity. Voltage reversal adversely affected bacteria on the anode of the affected cell, as shown by a relative decrease in cell performance following a cycle of starvation (no feeding). The control of voltage reversal will be crucial for long-term operation of MFCs in series. Rapid feeding of a cell can restore positive voltage generation, but the long-term impact of charge reversal will be inactivation of bacteria and it will require that the affected cells be short-circuited to maintain stack power production. A better understanding of the long term effects of voltage reversal on power generation by MFC stacks is needed in order to efficiently increase voltage production by using stacked MFC systems.

Keywords: Microbial fuel cell; Voltage reversal; Stack; Direct electron transfer

1. Introduction

Microbial fuel cells (MFCs) can be used to generate electricity from various forms of biodegradable organic matter, even human and animal wastewaters [1–4]. The power produced depends largely on the system architecture and internal resistance [5], bacteria on the anode, type of organic matter, chemical characteristics of medium (solution conductivity, pH and chemical concentration), and catholyte [6–8]. While some systems use ferricyanide or metal cathodes [9–11], it is generally accepted that oxygen will need to serve as the final electron acceptor for practical use of MFCs for power generation [5].

The maximum theoretical power generated in an MFC can be calculated based on the substrate and catholyte. For example, the open circuit voltage (OCV) for acetate oxidation linked with oxygen utilization at the cathode is ~1.1 V [5]. However, a single MFC typically produces an OCV less than 0.8 V, and a working voltage of ~0.5 V due to energy utilization by bacteria, electrode overpotentials, and high internal resistance. In order to increase overall voltage, MFCs can be stacked together (i.e., linked together in series) resulting in a nearly additive increase in total voltage [12–14]. However, there is very little information available on the performance of stacked MFCs, and no previous investigations with air-cathodes in mediatorless MFC systems. Shin et al. [14] tested an MFC stack containing biopolar plates with oxygen or ferricyanide catholytes, but they used a chemical mediator in their system (thionin). Mediators are generally toxic, so using them is impractical for economical electricity generation [2,6]. Aelterman et al. [13] found that 2.2 V (255 mA) could be generated using a six-cell MFC stack with ferricyanide as the catholyte (no mediator). While performance was sustained using continuous feeding, they also reported that in some tests at high

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current densities that they observed cell charge reversal, whereby the voltage in one cell abruptly reversed polarity. Aelterman et al. [13] did not further explore this situation, noting only that fuel starvation was thought to result in cell reversal.

In the case of a solid polymer fuel cell stack, various conditions can bring about a fuel cell being driven into voltage reversal by other cells. Voltage reversal generally occurs when one or more cells experience a more extreme level of one of these conditions compared to other cells in the stack. These conditions would be insufficient oxygen at the cathode, insufficient fuel, impedance differences, and a lack of catalyst. When the voltage reversal occurs undesirable electrochemical reactions may occur and adversely affect fuel cell components [22,23].

To investigate the potential for polarity reversal in MFCs, we constructed a two-chamber MFC stack using an air-cathode system. The system was operated in a manner that would allow direct investigation of cell reversal in relation to system performance. Microorganisms have reversible hydrogenase enzymes, allowing for a biotic route of charge reversal whereby electrons could be captured into the cell rather than discharged from the cell. Using a sterile MFC in the stack, we show that bacteria do not need to be present in the system for voltage reversal to occur.

2. Materials and methods

2.1. MFC stack and single chambered MFCs

The anode and cathode in a reactor were placed on opposite sides in a plexiglas chamber, separated by a small electrode spacing (2 cm) shown to reduce MFC internal resistance [20] (Fig. 1A). The working volume of a single cell was 85.5 mL (9.5 cm × 4.5 cm × 2.0 cm). A conductive flexible graphite sheet (11.5 cm × 5.5 cm × 0.2 cm) was used to cover the anode so that air or liquid did not reach the biofilm on the anode. The edges of the electrodes protruding from the sides of the reactor were sealed using a viton rubber sheet. The two-cell stack was constructed using two cells having the same structure with the anode of the first cell connected to the cathode of the second cell using a small graphite plate (1.0 cm × 1.0 cm × 1.0 cm) (Fig. 1B). This direct contact formed a type of bi-polar plate between the two electrodes, eliminating the potential for high contact resistance caused by using a wire to connect the cells [15]. The anode and cathode material used were carbon paper and Pt-coated carbon on one side of the carbon paper (0.5 mg Pt cm^{-2}; 10% Pt; De Nora North America Inc.), respectively. The surface area (geometric area) of each electrode was 43 cm^2. A reference electrode (Ag/AgCl, 0.195 V versus a normal hydrogen electrode, NHE) was placed into the chamber of each cell to determine individual electrode potentials. The stacked cells were normally operated in fed-batch mode, with substrate in fresh medium replaced in each cell at the beginning of the cycle. In one set of tests the two cells were operated in continuous flow mode, with the medium containing 0.1 M of acetate [16] passed sequentially through the reactors (i.e., from Cell I to Cell II) using Tygon® tubing using a peristaltic digital pump.

To investigate the effect of bacterial activity on voltage reversal, separate tests were conducted using two MFCs linked in series and operated in fed batch mode. The construction of these reactors was previously described [17]. Each reactor had an empty bed volume of 28 mL, and the electrodes placed on opposite sides of the reactor (4 cm spacing) (Fig. 1C). The electrode material was the same as that described above.

2.2. Inoculation and operation

Dewatered sludge from an anaerobic digester (State College, PA) was used as an inoculum for the stack MFC. Acetate or glucose was used as an energy source in a nutrient solution (pH

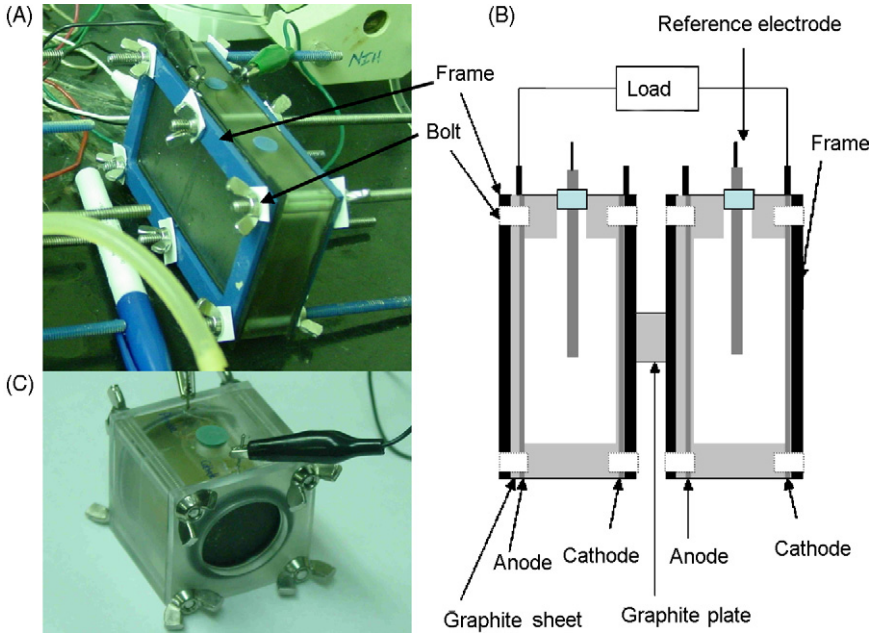

Fig. 1. (A) Photograph of a single cell used in MFC stack experiments, which holds a liquid volume of 85.5 ml. (B) Schematic of a two-cell stack illustrating how the anode and cathode were linked by a central graphite plate. (C) A smaller 28-ml single-chambered MFC that can be linked to another MFC in series.

7.0) [16]. During start-up, each cell was filled with the nutrient solution and inoculated with sludge (2 g) and glucose (1 g L^{-1}) in an anaerobic glove box. The sampling ports of each chamber were then sealed with rubber stoppers, and the reactor was removed from the glove box and operated using a 30 Ω resistor (except as stated otherwise). Voltage was intermittently recorded across the resistor using a multimeter. The stack was operated in a fed-batch mode and repeatable cycles of power generation were observed. When the voltage decreased to below 30 mV, 1–3 mL of acetate (1 M) was added to each individual cell using a syringe. The 28 ml MFC was inoculated and operated in the same manner.

To obtain a polarization curve, a different external load was used for a complete batch cycle [18], with the resistance varied over a range of 2–100,000 Ω. The voltage was recorded and the current and power calculated for each cycle. All MFCs were operated in a constant temperature room (30 °C).

2.3. Analysis

Cell voltage of the system was monitored using a precision multimeter and a data acquisition system (2700, Keithly, OH, USA). All data were automatically recorded by a computer every 30 min. Power (P) was calculated according to $P = IV$ ($I = V/R$), where I is the current (A), V the voltage (V), and R the resistance (Ω). Power was normalized based on the cross-sectional area (projected) of the anode. The cell voltage (V_{Cell}) and anode potential (V_{An}) voltages were measured with respect to a Ag/AgCl reference electrode, and the cathode potential (V_{Cat}) was calculated based on the voltage difference as $V_{Cell} = V_{Cat} - V_{An}$. Impedance data were measured by electrochemical impedance spectroscopy using a PC4/750 potentiostat (Gamry Instruments) with the anode as the working electrode. The cathode was used as counter electrode and reference electrode. Impedance measurements were conducted at open circuit voltage over a frequency range of 10^5 down to 0.1 Hz with a sinusoidal perturbation of 10 mV amplitude [19].

3. Results

3.1. Power production using the two-cell MFC stack

The two-cell stack produced stable and repeatable power cycles within 300 h of inoculation, as shown in Fig. 2 by repeatable cycles of power generation with the reactor operated at external resistances of 30 Ω (0.4 V maximum voltage) and 500 Ω (0.9 V maximum voltage). The open circuit voltage produced using the two-cell stack was 1.27 V (OCV).

The polarization curves of the individual cells show nearly identical OCVs of the two cells (0.65 and 0.64 V) (Fig. 3A). The maximum power produced by the stack was 3.9 mW, or a power density of 460 mW m^{-2} (normalized by the total anode surface area of 86 cm^2; current density of 872 mA cm^{-2}) at a total stack voltage of 0.53 V (30 Ω resistor) (Fig. 3B). This is a volumetric power density of 23 W m^{-3} (total reactor volume). This power density is comparable to 506 mW m^{-2}, or 23 W m^{-3} [20], with single-chamber air-cathode MFCs operated in this manner using

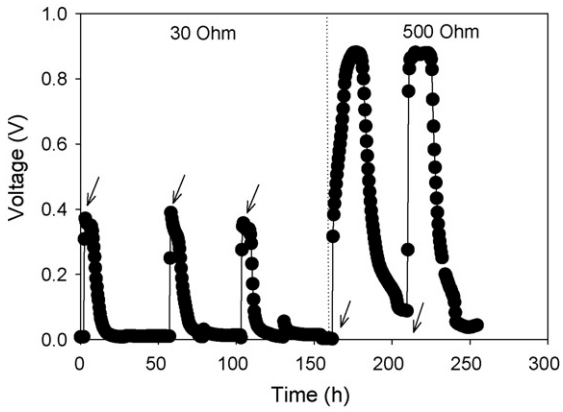

Fig. 2. Multiple fed-batch cycles showing voltage generation in two-cell MFC stack at the indicated external resistances of 30 and 500 Ω (the arrows indicate injections of 1 mL of 1 M acetate into both cells).

solutions of this ionic strength. Min and Logan [4] operated a flat plate MFC and obtained 286 mW m^{-2}, which is lower than that obtained here.

The slope of the polarization curve was linear over the range of ~200–1000 mA cm^{-2}, but at higher current densities the slope decreased suggesting mass transport limitations at an electrode (Fig. 3A). There was evidence of voltage reversal at high current densities, as the voltage of Cell II dropped to −0.15 V at a current density of 1197 mA cm^{-2}. At this current density

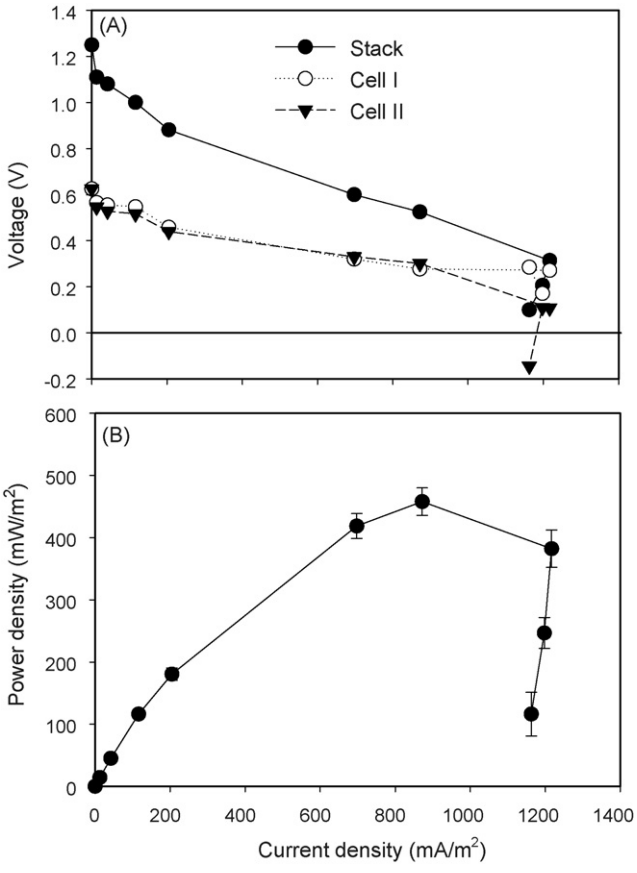

Fig. 3. (A) Stack and individual cell polarization curves for the two-cell stack. (B) Power density curve obtained for the stack.

the power output by the two-cell stack was limited by Cell II, indicating that the performance of Cell I was better than that of Cell II. The difference could be due to greater electrogenic activity by the bacteria in Cell I, greater performance of the cathode in Cell I, or slight differences in substrate concentrations that altered bacterial activity.

The internal resistance of the stack was 15 Ω. This value is much lower than 161 Ω (single-chambered MFC; [20]) and 1286 Ω (two-chambered MFC; [21]) produced in other reactors using a similar medium (i.e., at similar solution conductivities). This internal resistance is larger than 3.9 Ω measured for a single cell in the stack developed by Aelterman et al. [13] that used a ferricyanide catholyte instead of oxygen.

3.2. Voltage reversal in MFC stacks

When operated in continuous flow mode (i.e., with continuous flow from Cell I into Cell II), the system produced continuous power and all cell voltages remained positive. The stack voltage ranged from 0.8 to 0.9 V when the stack was fed adequate acetate (0.1 M) at a hydraulic retention time of 3 h in both cells (external load of 500 Ω).

When the system was operated in fed-batch mode, positive cell voltages were maintained in both cells only at the beginning of each cycle (Fig. 4). At an external load of 30 Ω, for example, the voltages at the beginning of the cycle were 0.27 V (Cell I) and 0.20 V (Cell II). At the end of every batch cycle, however, it was observed that the voltage in Cell II reversed (Fig. 4). After 5 h of operation, for example, the voltage of Cell I was 0.6 V while the voltage in Cell II decreased to −0.6 V.

Voltage reversal is well known to occur in hydrogen fuel cells when one of the cells suffers loss of the fuel or exhibits a much larger resistance than other cells in the stack [22,23]. We therefore examined whether voltage reversal here was due to differences in substrate concentrations in two cells, as suggested by others [13]. First, we showed that substrate concentration affected voltage generation (Fig. 5). Voltage output by the two-cell stack was relatively stable at 0.5 V at an acetate concentration of >12 mM, but the voltage decreased in proportion to substrate concentration at lower acetate concentrations. This

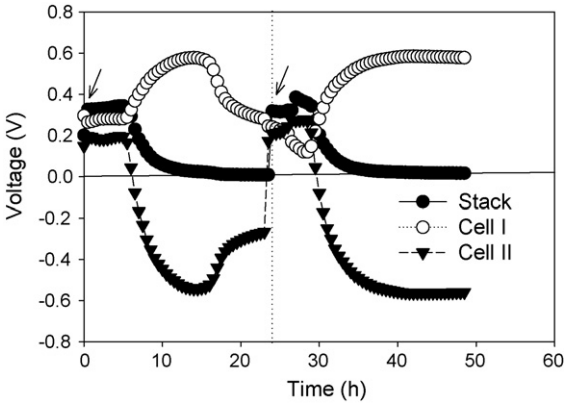

Fig. 4. Stack and individual cell voltages over time in the two-cell stack (arrows indicate addition of 1 ml of 1 M acetate; external load was 30 Ω).

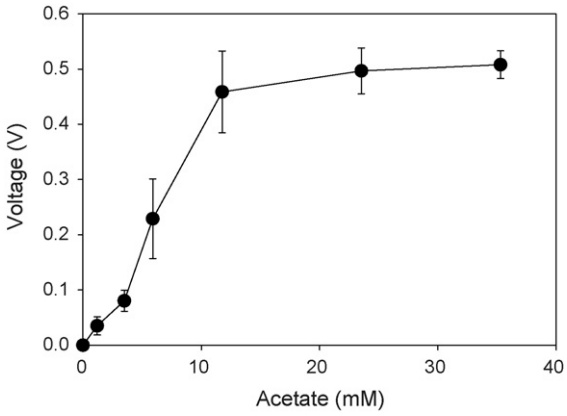

Fig. 5. Stack voltages as a function of acetate concentration in the two-cell stack (external load was 30 Ω).

effect of substrate concentration on voltage is consistent with that previously observed with a single-cell MFC [17,24].

Second, we demonstrated that low substrate concentration would produce voltage reversal by intentionally starving one of the cells over a cycle by not feeding it substrate. As shown in Fig. 6, in the first cycle when both cells were fed substrate (acetate), positive voltages were initially produced in the two cells at the beginning of the cycle producing a total stack voltage of 0.38 V. However, the voltage in Cell I was lower than that of Cell II at the beginning of this first cycle. Thus, after this initial period Cell I demonstrated voltage reversal with Cell II having

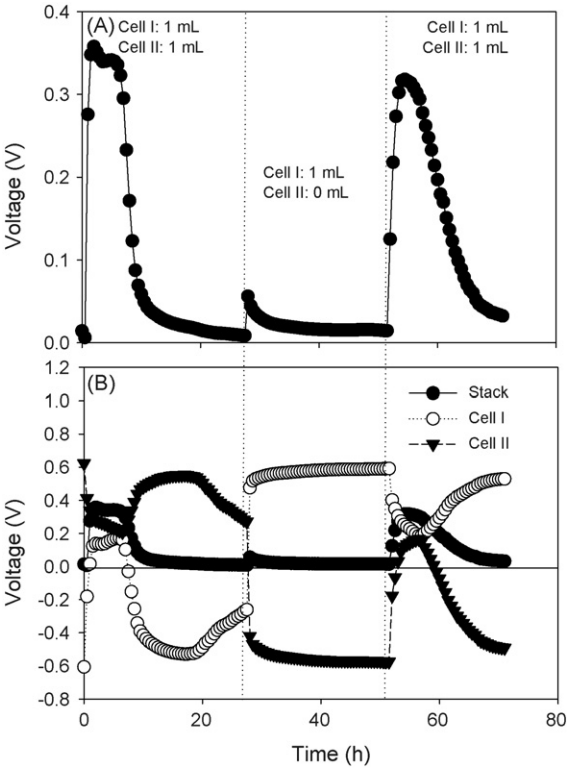

Fig. 6. Stack voltages (A) and individual cell voltages (B) over time in the stack under different conditions (at time 0, 1 mL of 1 M acetate was added to Cell I and II, at time 28 h, 1 mL acetate to Cell I and no substrate to Cell II, and at time 53 h, acetate was added to both Cell I and II. The external load was 30 Ω).

a positive voltage throughout the first cycle. Thus, the weaker cell at the beginning of the cycle is the one that will undergo voltage reversal (also supported by results shown in Fig. 4). We next demonstrated that we could make Cell II undergo charge reversal by not feeding it (i.e., having it produce less voltage than Cell I). In the second cycle, we starved Cell II (no substrate), making it the weaker cell compared to Cell I, and thus, Cell II showed voltage reversal. The maximum stack voltage for this second cycle was now only 0.06 V.

Third, we fed both cells again to see which cell would undergo charge reversal. In this third cycle (Fig. 6), we observed that Cell II initially produced less voltage than Cell I, and that it then showed charge reversal. By starving Cell II over a whole feeding cycle we made it the weaker cell in the third cycle, and we produced voltage reversal in it even though both cells were fed the same amount of substrate. This shows that prolonged starvation adversely affected Cell II, making it the weaker cell, and that the weaker cell will be the one to undergo charge reversal.

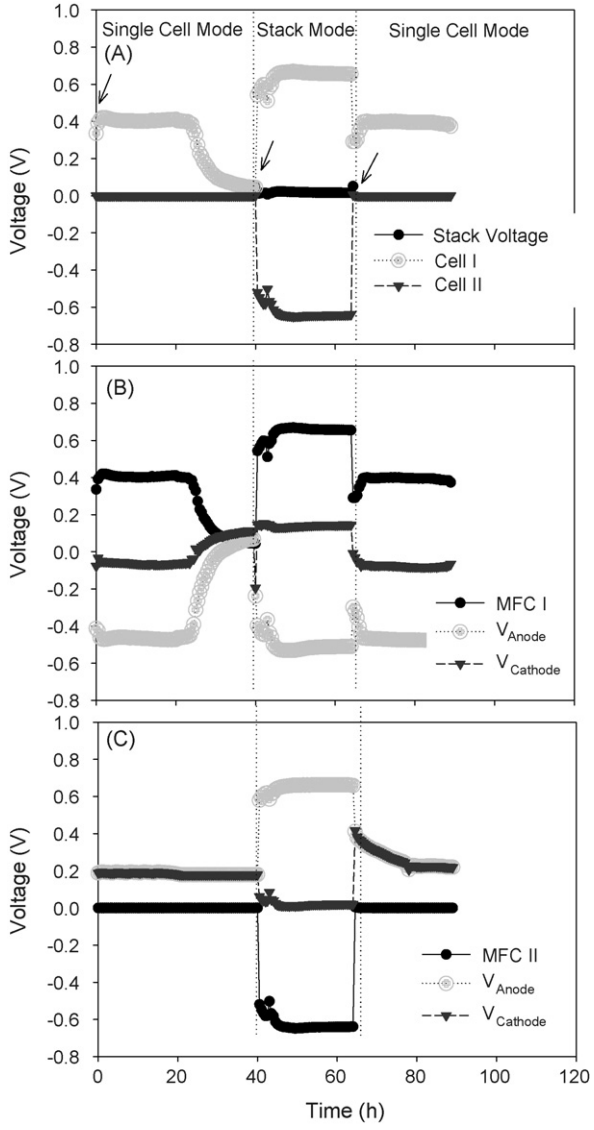

Fig. 7. Stack voltages (A) and individual voltages of working MFC (MFC I) (B) and autoclaved MFC (MFC II) (C) in single cell and stack mode. The arrows indicate injections of 1 mL of glucose (final concentration 714 mg L^{-1}). External load is 1000 Ω.

3.3. Voltage reversal in two MFCs connected in series

To determine whether bacteria were needed to produce voltage reversal, we conducted additional tests using two 28 ml single-chamber MFCs linked in series (connected by a wire) where the first reactor was an active MFC, but the second one was sterile (Fig. 7). This series mode of operation is the same as Aelterman et al. [13] whose stack was composed of a series of reactors connected by copper wires. The first MFC in the series was inoculated and produced stable power within 100–150 h. The individual potential for the biotic MFC I over a 40 h cycle were $V_{Cat} = -0.06$ V and $V_{An} = -0.46$ V, for a total cell voltage of 0.40 V, which is typical for this cell (Fig. 7B). This is a working power density of 228 mW m^{-2}, with a maximum power density of 371 mW m^{-2} produced with an external load of 150 Ω. The cell and anode voltages of the sterile Cell II were $V_{Cell} = 0.0$ V and $V_{An} = 0.18$ V (Fig. 7C).

The working MFC (MFC I) was then connected to a sterile MFC (MFC II) in series using a wire between the cathode of MFC I and anode of MFC II (Fig. 7) after 40 h. As soon as MFC I and II were connected, the cell voltage of MFC I was 0.66 V while the cell voltage of MFC II decreased to −0.64 V, demonstrating voltage reversal in the sterile Cell II (Fig. 7A). This voltage change produced in Cell II is the same as that observed for the two cells stack (Fig. 6). When the two cells were separated after 65 h, the original voltage was recovered in Cell I indicating power generation in this cell was not adversely affected by charge reversal in Cell II. Thus, we concluded that bacteria were not needed to produce the observed voltage reversal in MFCs connected in series since voltage reversal occurred in the absence of bacteria.

4. Discussion

MFCs can be linked in series or stacks, producing power densities similar to that obtained with using individual cells but allowing total voltage to be increased to a more useful value. In a stack it is the number of cells that contributes to the final voltage, while the surface area of each cell determines the final current. We achieved a maximum volumetric power density of 23 W m^{-3} and an open circuit voltage of 1.3 V (OCV) using a two-cell stack. Aelterman et al. [13] obtained a higher volumetric power density of 59 W m^{-3} (308 W m^{-3} based on the void volume in the anode chamber packed with graphite granules), but they used ferricyanide as the catholyte. The total stack voltage in their system was 4.16 V (OCV), or 0.69 V per cell in the six-cell stack, which is slightly larger than that achieved here. Shin et al. [14] did not report power normalized to the reactor volume, but they indicated a maximum power density of 230 mW m^{-2} (normalized by the electrode projected surface area) using oxygen (versus 1300 mW m^{-2} using ferricyanide) in their mediator-type stacked MFC. This power density with oxygen is less than that found here (460 mW m^{-2}) despite the

fact that they used a chemical mediator. Wilkinson [12] built a 6-cell stack MFC assembly, but no specific data was provided on the power densities produced by his MFC.

During continuous stack operation voltage reversal did not occur due to high substrate concentrations in both cells and short hydraulic retention times (HRTs). The feed concentration of acetate in these tests was 100 mM and the HRT was 3 h. Although we did not measure acetate concentrations in either cell the rates of substrate removals observed in other tests should not have reduced the substrate concentration to below 96 mM. As can be seen in Fig. 5, voltage output is stable when the acetate concentration is >12 mM (with 30 Ω external resistance). Therefore, we did not observe a voltage reversal as the acetate concentrations likely remained well above 12 mM in both cells. Aelterman et al. [13] operated a six-cell MFC stack and had the flow fed individually into each cell to ensure sufficient substrate in each cell. They tested volumetric loading rates of 1.62 and 2.17 g COD L^{-1} d^{-1} (0.58 and 0.78 g L^{-1} of sodium acetate at a HRT of 8.86 h). When external resistances were higher than 360 Ω (with 1.62 g COD L^{-1} d^{-1}), voltage reversal did not occur, but at lower external resistances voltages started to diverge suggesting that low external resistances produced voltage reversal. The volumetric loading rate used here was much higher than that of Aelterman et al. [13], i.e. 51.2 versus 1.62 g COD L^{-1} d^{-1}, respectively. Also, the external load (500 Ω) used in this study was higher than the value indicated by them to be a threshold for voltage reversal. Therefore, we did not expect (and did not observe) voltage reversal in our stack system under continuous flow conditions with the feed from Cell I into Cell II.

The main challenge to obtaining useful power from MFC stacks is to avoid voltage reversals. Voltage reversal is produced when voltage in the cells is not matched, as we have shown can occur as the result of substrate starvation. This voltage reversal adversely affects the bacteria in the biofilm experiencing the voltage reversal, as shown by a subsequent reduced performance of the stack cell.

The problem of voltage reversal can be addressed in several different ways. First, a cell demonstrating voltage reversal could easily be short-circuited, thereby eliminating it from reducing stack performance. Several researchers have used diodes connected in parallel in the circuit in hydrogen fuel cell stacks because diodes have a low ohmic resistance and are able to prevent charge reversal [22]. Thus, when one or more of the cells show exhibit defective operation, they can be automatically short-circuited using this simple and economical approach [23]. Second, fuel starvation must be avoided by making sure that there is sufficient substrate provided to the anode and that there is oxygen at the cathode. Third, continuous operation of a MFC stack (versus fed batch cycles) can be used in laboratory studies to avoid the potential for low substrate conditions at the feed. When the system examined here was operated in a continuous mode operation, voltage reversal has not found. Finally, the system should be operated at lower current densities. As shown in Fig. 3A, at high current density of 1197 mA m^{-2}, voltage of Cell II was reversed while at low current densities, sign of voltages of Cell I and II were all positive. Aelterman et al. [13] also observed voltage reversals at higher current densities tested in their system.

5. Conclusions

Stacking individual air-cathode MFCs, using either wires or a conductive plate, successfully increased voltage of two-cell stacks. However, voltage reversals occurs when one cell does not generate sufficient voltage relative to other cells. It was shown that fuel starvation in an active cell, or a lack of power generation in the absence of bacterial activity in a cell (abiotic conditions) produced voltage reversal. A better understanding of the effects of voltage reversal on the bacterial biofilm, and the factors that lead to voltage reversal, will help to enhance the utility of using MFCs in stacks. Future studies should examine methods to control voltage reversal and to temporarily isolate cells in the stack until stable power generation can be restored.

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