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Domestic wastewater treatment using multi-electrode continuous flow MFCs with a separator electrode assembly design

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Abstract Treatment of domestic wastewater using microbial fuel cells (MFCs) will require reactors with multiple electrodes, but this presents unique challenges under continuous flow conditions due to large changes in the chemical oxygen demand (COD) concentration within the reactor. Domestic wastewater treatment was examined using a single-chamber MFC (130 mL) with multiple graphite fiber brush anodes wired together and a single air cathode (cathode specific area of 27 m^2/m^3). In fed-batch operation, where the COD concentration was spatially uniform in the reactor but changed over time, the maximum current density was 148±8 mA/m^2 (1,000 Ω), the maximum power density was 120 mW/m^2, and the overall COD removal was >90 %. However, in continuous flow operation (8 h hydraulic retention time, HRT), there was a 57 % change in the COD concentration across the reactor (influent versus effluent) and the current density was only 20±13 mA/m^2. Two approaches were used to increase performance under continuous flow conditions. First, the anodes were separately wired to the cathode, which increased the current density to 55 ±15 mA/m^2. Second, two MFCs were hydraulically connected in series (each with half the original HRT) to avoid large changes in COD among the anodes in the same reactor. The second approach improved current density to 73±13 mA/m^2. These results show that current generation from wastewaters in MFCs with multiple anodes, under continuous flow conditions, can be improved using multiple reactors in series, as this minimizes changes in COD in each reactor.

Keywords Microbial fuel cell · Scaling up · Separator electrode assembly · Continuous flow · Domestic wastewater

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Introduction

Activated sludge processes typically require 0.6 kWh for each cubic meter of domestic wastewater treated, with up to 50 % of this energy used for wastewater aeration (Rosso et al. 2008; McCarty et al. 2011). Aerobic wastewater treatment processes also produce large amounts of sludge, and the treatment and disposal of the sludge present many challenges due to economic, environmental, and regulatory factors (Wei et al. 2003; Aelterman et al. 2006). Microbial fuel cells (MFCs) are being investigated as an alternative to activated sludge as they produce energy from wastewater and they generate much less sludge (Rabaey and Verstraete 2005). MFCs are devices that use microorganisms to directly produce electrical current from biodegradable organic and inorganic compounds (Rabaey and Verstraete 2005; Logan et al. 2006). Many types of wastewaters have been successfully treated using MFCs, including domestic, animal, brewery, and food processing wastewaters (Oh and Logan 2005; Feng et al. 2008; Jiang et al. 2011; Puig et al. 2011).

There are several operational aspects that need to be considered in designing MFCs for domestic wastewater treatment. First, domestic wastewater contains relatively low concentrations of chemical oxygen demand (COD) compared to other solutions used in laboratory tests. Power production in MFCs typically follows a Monod-type relationship with COD concentration, and a low COD can therefore reduce power production (Min and Logan 2004; Torres et al. 2007). Domestic wastewater CODs in MFC studies have ranged from 160 to 850 mg/L (Min and Logan 2004; Puig et al. 2011) compared to 780–1,900 mg/L typically used in tests with chemicals such as acetate (Catal et al. 2008; Lefebvre et al. 2008; Liu et al. 2009; Tsai et al. 2009; Nam et al. 2010). Other wastewaters can contain higher COD concentrations than domestic wastewater, for example 2,250 mg/L for brewery wastewater (Feng et al.

2008) and 8,320 mg/L for animal wastewater (Min et al. 2005). Second, the low solution conductivity of domestic wastewaters (~1 mS/cm) results in lower power densities than those obtained with highly conductive and usually better buffered solutions that are typically used in laboratory tests (i.e., 7 mS/cm for 50 mM phosphate buffer and higher; Nam et al. 2010). Low solution conductivities increase the internal resistance, resulting in reduced power output. One way to lower the internal resistance is to decrease the spacing between the electrodes. For example, the internal resistance was decreased from 35 to 16 Ω by reducing the electrode spacing from 2 to 1 cm (Liu et al. 2005). Further decreases in electrode spacing, however, can reduce power due to oxygen diffusion through the cathode to the anode. If anode bacteria respire using oxygen as a terminal electron acceptor, current will usually decrease. This effect of oxygen on current generation can be reduced by using a separator electrode assembly (SEA) MFC configuration, where a cloth or porous material is placed between the electrodes. Some SEA configurations have produced large increases in power densities due to an electrode spacing of <1 cm (Fan et al. 2007; Zhang et al. 2009).

Primarily for these two reasons (low wastewater strength and conductivity), power densities produced using domestic wastewaters are usually lower than those obtained with easily degraded substrates such as glucose and acetate, under both fed-batch and continuous flow conditions (Kim et al. 2010; Pant et al. 2010; Jong et al. 2011; Velasquez-Orta et al. 2011; Wei et al. 2012). For example, single-chamber MFCs operated in fed-batch mode produced a maximum power density of 146 mW/m^2 with domestic wastewater compared to 494 mW/m^2 with glucose (Liu and Logan 2004). In the case of continuous flow operation, the maximum power density with domestic wastewater was only 72 mW/m^2, compared to 212 mW/m^2 with glucose, 286 mW/m^2 with acetate, and 220 mW/m^2 with butyrate (Min and Logan 2004). A comparison of power production using acetate or domestic wastewater in SEA-type MFCs showed that 1,300 mW/m^2 could be produced with a high concentration of acetate in a higher conductivity medium (COD=780 mg/L, 6.9 mS/cm) compared to 220 mW/m^2 produced using domestic wastewater (COD=390±89 mg/L, 1.8 mS/cm; 14 mL liquid volume, graphite fiber brush anodes, glass fiber separators, and activated carbon cathodes; Hays et al. 2011).

One additional challenge in the use of MFCs for domestic wastewater treatment that has not received sufficient attention is the performance of reactors containing many electrodes (Cusick et al. 2011; Jiang et al. 2011; Kim et al. 2011a). The packing density of these electrodes can be increased in order to minimize the hydraulic retention time (HRT) by using SEA configurations (Logan 2012). We recently developed and tested a compact MFC that could be easily scaled to larger reactor sizes (Ahn and Logan 2012). The MFC contained three brush anodes pressed against a separator to reduce electrode spacing and a single large cathode. The reactor was operated in fed-batch or continuous flow mode, but performance was examined using only a synthetic acetate-based medium (Ahn and Logan 2012). The maximum power density of this multi-anode MFC (975 mW/m^2, 50 Ω resistor) was lower than that reported with the single-anode reactor when it was operated in fed-batch mode (1,300 mW/m^2; Hays et al. 2011). However, the voltage substantially decreased from 0.68 to 0.21 V when this reactor was operated with continuous flow (8 h HRT; Ahn and Logan 2012). The reason for this decrease in voltage was thought to be a result of decreased average COD in the reactor. However, a more detailed analysis for the reason for this decrease in performance was not conducted, and no method was developed to increase voltage or power production in this MFC.

In this study, we examined the effect of COD changes on MFC performance by comparing current generation under fed-batch conditions (where the COD concentration is relatively uniform in the reactor at any point in time) with continuous flow conditions (where there is a COD gradient across the reactor at any instant in time due to the differences between the influent and effluent COD concentrations). The current production by the individual anodes was compared to that obtained with separate circuits (one for each anode) for the MFC operated in continuous flow. MFCs were also operated in series in order to show how smaller changes in COD could improve current and power generation.

Materials and methods

MFC construction Single-chamber, air cathode MFCs (130 mL liquid volume) were constructed as previously described (Fig. 1; Ahn and Logan 2012). Each reactor contained three brush anodes (25 mm diameter by 35 mm length) made from carbon fibers (PANEX 33 160K, ZOLTEK) wound into a titanium wire core (Logan et al. 2007), with the electrodes placed perpendicular to the flow path in continuous flow operation. The anode specific surface area was 2,600 m^2/m^3 based on fiber mass and liquid volume and 20 m^2/m^3 based on projected area. The anodes were connected together externally with a single copper wire, except as noted below. The MFC contained a single air cathode (30 wt% wet-proofed carbon cloth, type B-1B, E-TEK) with a platinum catalyst (0.5 mg/cm^2) on the water side and four diffusion layers on the air side (Cheng et al. 2006). The cathode was 7 cm long and 5 cm wide, with 35 cm^2 exposed to the air, resulting in a cathode specific surface area of 27 m^2/m^3. Two layers of a textile material (Amplitude Prozorb, Contec Inc.) were used as the separators between the anode and cathode to avoid short circuiting

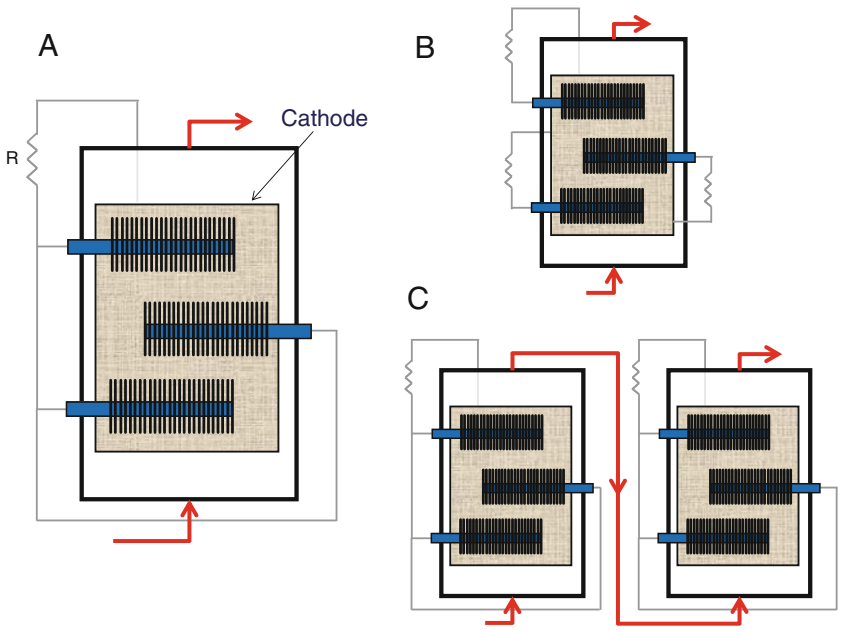

Fig. 1 Schematic presentation of operational configurations used in this study—**a** all anodes connected together, **b** each anode connected to individual circuit, and **c** two MFCs connected in series

between the electrodes and to reduce oxygen crossover. The textile separator was made from 46 % cellulose and 54 % polyester (thickness=0.3 mm; weight=55 g/m^2).

MFC operation Domestic wastewater, used as the inoculum and fuel, was collected from the primary clarifier of the Pennsylvania State University Wastewater Treatment Plant. The wastewater was stored in refrigerator and then kept on ice during reactor feeding tests to minimize COD changes of the wastewater feed over time. The composition of wastewater samples obtained from the treatment plant varied over the course of the study, ranging from 275±71 mg-COD/L in initial tests to 155±36 mg-COD/L in later tests, with overall average characteristics of 232±84 mg-COD/L, a biochemical oxygen demand of 111±44 mg-BOD_5/L, and a conductivity of 1.4±0.2 mS/cm. For the first 5 days of startup, the reactor was refilled with fresh wastewater every day. Then, the reactors (duplicates) were operated in fed-batch mode by refilling the chamber with fresh wastewater when the voltage decreased to <10 mV (defined as one fed-batch cycle of operation). MFCs circuits contained either a 100 or 1,000 Ω resistor (as indicated). Following fed-batch tests, MFCs were switched to continuous flow mode by feeding wastewater into the bottom of the reactor using a peristaltic pump (Masterflex, Cole-Parmer, Vernon Hills, USA). The flow rates were adjusted to obtain HRTs of 8 h (390 mL/day) or 16 h (195 mL/day), and these were slow enough to ensure the wastewater was warmed to ambient temperature in the constant temperature room (30 °C).

Calculations and measurements The voltage (E) across the external resistor was measured every 20 min using a data acquisition system (Model 2700, Keithley Instruments) connected to a computer. Current (I) and power ($P=IE$) were calculated as previously described (Hays et al. 2011) and normalized by the cross-sectional area of the cathode (35 cm^2; Logan et al. 2006). Except as noted, polarization and power density curves were obtained by varying the external resistance (R_{ext}) of the circuit (20 min per resistor for fed-batch mode, and every 4 h for continuous flow mode). In one set of experiments, polarization data were obtained using linear sweep voltammetry (LSV; Biologic, VMP3) at a slow scan rate of 0.5 mV/s. The polarization tests were carried out three times after MFCs produced a stable maximum current. CE was calculated using the ratio of the total coulombs produced during the experiment to the theoretical amount of coulombs available from the substrate using CE=$C_{out}/C_{in}\times 100$ %, where C_{out} is the total coulombs actually transferred to the anode from the substrate and $C_{in}=(F\ b\ \mathrm{COD}_{in}\ Q\ \Delta t)/M$, where F is Faraday's constant, b=4 is the number of electrons exchanged per mole of oxygen, COD_{in} is the influent COD, Q is the flow rate, Δt is the time interval (HRT) used to calculate output charge, and M=32 is the molecular weight of oxygen (Logan et al. 2006). C_{out} was obtained by integrating the current over time. CODs were measured using high range (0–1,500 mg/L) HACH COD system with a spectrophotometer (DR/2010, Hach Co., Loveland, CO). Total COD was measured at the beginning and end of each batch cycle, while it was measured every 2 or 3 days for continuous flow mode operation. Internal resistance was characterized by electrochemical impedance spectroscopy (EIS) with a potentiostat (BioLogic, VMP3; He and Mansfeld 2009) over a frequency range of 100 kHz to 1 mHz with an AC signal amplitude of 10 mV.

Results

Reactor performance in fed-batch mode MFCs acclimated to the higher external resistance (1,000 Ω) demonstrated reproducible cycles of current generation more rapidly (within 12 days) than those acclimated to a lower resistance (100 Ω, 25 days; Fig. 2). This was consistent with previous findings that high external resistances shortened the lag phase for development of an exoelectrogenic biofilm (Hong et al. 2011; Zhang et al. 2011). The maximum current density during a single batch cycle was 148±8 mA/m^2 (1,000 Ω), with each fed-batch cycle typically 3.7 days long. A higher maximum current density of 310 mA/m^2 (0.13 V) was obtained using 100 Ω due to the lower circuit resistance. The average maximum power densities obtained from polarization curves obtained after five stable fed-batch cycles (day 50) were 120 mW/m^2 (3.3 W/m^3) for the MFCs acclimated at 1,000 Ω and 62 mW/m^2 (1.8 W/m^3) for MFCs acclimated to 100 Ω (Fig. 3).

COD removals were very good (>90 %) in all fed-batch tests with CEs of 22 % (100 Ω) and 15 % (1,000 Ω). These CEs are higher than those previously obtained in SEA MFCs with this source of domestic wastewater (5.6–7.2 %; Hays et al. 2011) likely due to the lower COD loading here than in that previous study, and thus shorter cycle times. CEs are expected to be higher under conditions that produce shorter cycle times (Oh et al. 2009). The fed-batch cycle time here was typically 3.7 days long, which was longer than that needed for acetate-fed MFCs (3.1 days; Ahn and Logan 2012) despite a lower COD loading with wastewater. A longer cycle time will increase the total mass of oxygen added into the anode chamber over the course of the fed-batch cycle and lower the CE (Oh et al. 2009). Other contributing factors to the lower CE of the wastewater could be due bacterial respiration using alternative electron acceptors in the wastewater, such as nitrate and sulfate, and methanogenesis (Chang et al. 2005).

Reactor performance in continuous flow mode When MFCs were operated in continuous flow mode, the overall performance

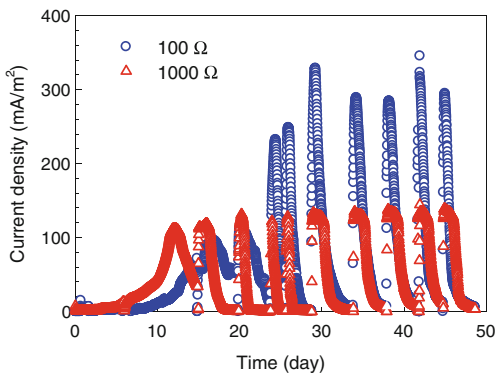

Fig. 2 Current production in a MFC in fed-batch mode with different external circuit loads

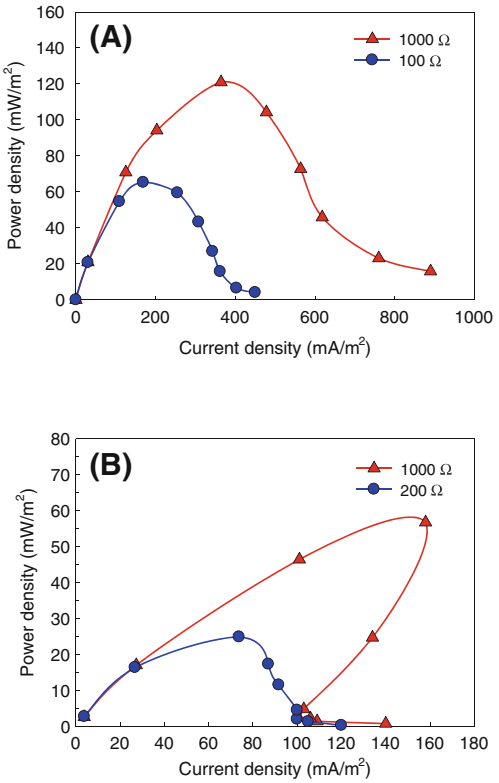

Fig. 3 Power density curves as a function of current density for MFCs in **a** fed-batch mode and **b** continuous flow mode (HRT=8 h) with different external circuit loads

substantially decreased compared to that obtained in the initial part of a fed-batch cycle (Fig. 4). A slightly higher external resistance (200 Ω) was used in continuous flow tests than fed-batch tests (100 Ω) in one of the MFCs in order to try to increase performance. This resistance was chosen as fed-batch polarization test results showed that the maximum power densities were produced at 200 Ω (1,000 Ω acclimated reactor) and 500 Ω (100 Ω acclimated reactor). In continuous flow tests (HRT=8 h), the voltage averaged only 0.12±0.06 V (145±80 mA/m^2) at 200 Ω and 0.41±0.05 V (103±13 mA/m^2) at 1,000 Ω. The COD removal was 52 % with CE=7 % (200 Ω). The variations in the current density over time (Fig. 4a) could have been due to changes in the composition of the wastewater source at different sampling periods from the wastewater treatment plant and changes in wastewater composition during storage in the refrigerator.

When MFCs were operated at a longer HRT=16 h (200 Ω), voltage and current generation was essentially eliminated (0.02 V±0.004; 0.25±0.08 mA/m^2). The change in COD was even larger at this HRT, with a total COD removal of 60 %, and the CE was only 0.6 %. The low CE is consistent with results showing little current generation despite these COD removals.

The maximum power densities under continuous flow conditions, based on polarization data, were 57 (1.5 W/m^3,

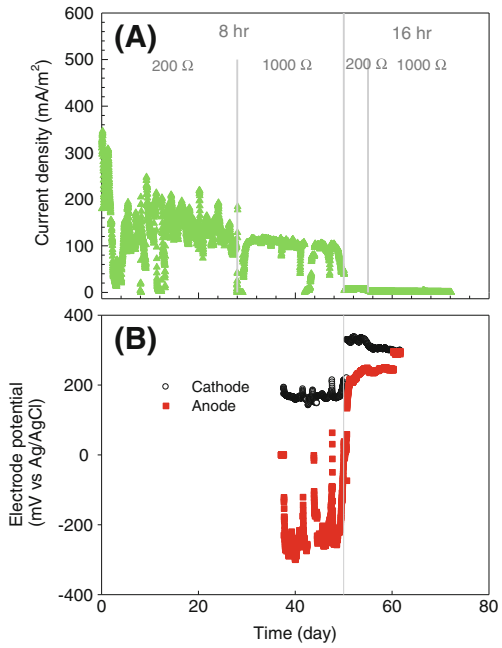

Fig. 4 **a** Current production and **b** electrode potentials during continuous flow mode operation of MFC fed with domestic wastewater at the indicated external resistances and HRTs

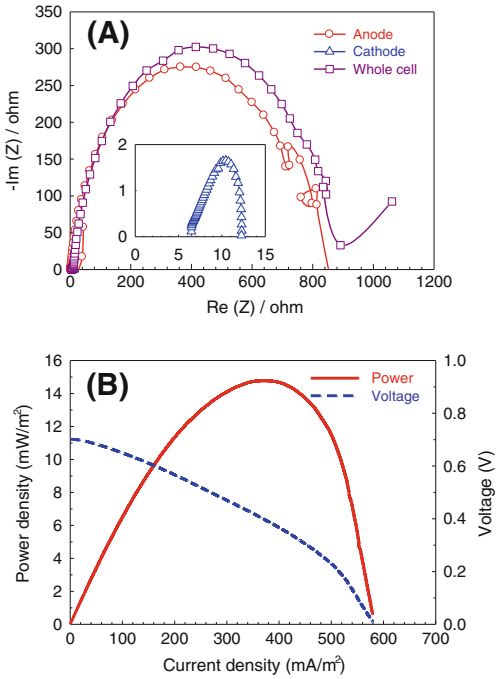

Fig. 5 **a** Impedance spectra under working cell conditions and **b** polarization curve for the MFC with domestic wastewater (HRT=8 h, R_{ext}=200 Ω)

acclimated at 1,000 Ω) and 25 mW/m^2 (0.7 W/m^3, acclimated at 200 Ω; Fig. 3b). Power overshoot (type D), where the power density curve doubles back to lower current densities as the voltage is increased, was observed for the MFC acclimated to the higher resistance. This observation of power overshoot is consistent with previous findings that MFCs acclimated to high resistances often exhibit power overshoot at higher current densities (lower resistances) in polarization tests (Hong et al. 2011). It is not known why power overshoot occurred during continuous flow operation, but not during fed-batch mode. It may be that operation with more consistent conditions produced by continuous flow mode created more instability in the response of the biofilms when varying resistances and current densities during the polarization results. Power overshoot may also be a consequence wiring anodes together when there are large changes in COD, as further explored below.

Internal resistance analysis Total internal resistance was measured under continuous flow conditions (HRT=8 h) by two methods: using the linear portion of the polarization curve obtained using LSV (Logan 2008) and EIS analysis. Total internal resistance was 2,580 Ω based on polarization data and 1,800 Ω from the EIS analysis (Fig. 5a). Based on EIS results, the anode charge transfer resistance (860 Ω) was much larger than that of the cathode (5 Ω). The ohmic resistance was 6 Ω, which was only slightly higher than that obtained with an acetate solution having a much higher conductivity (2.2 Ω, 6.9 mS/cm).

Total power production measured by LSV was much lower (15 mW/m^2, Fig. 5b) than that obtained by changing the resistances (25 mW/m^2). Typically, the use of LSV results in higher maximum power densities than those obtained using different resistances (Velasquez-Orta et al. 2009), so the method used to obtain power densities was not a contributing factor here to low power production. During this period of continuous flow operation, the wastewater COD concentration was lower (160 mg/L) than it was for polarization tests conducted by varying the resistances (220 mg/L). This suggests that the lower power density obtained in the LSV test was due to decreased COD concentration of the wastewater.

Methods to increase current production under continuous flow conditions The dependence of power production on COD concentration, combined with the EIS analysis that showed high resistances with domestic wastewater, suggested that the low current densities were due to poor anode performance (Fig. 4b). The known dependence of anode potential on COD concentration (Ditzig et al. 2007) suggested that creating conditions that minimized changes in COD around an anode would be critical for improving performance. To test this hypothesis, two additional operational configurations were examined: (1) uncoupling the anodes from each other and (2) creating more uniform COD concentrations in the reactor. For the first approach, the anodes were connected through three individual circuits

and resistors (1,000 Ω) to the cathode (Fig. 1b). The first anode (bottom, influent) produced a voltage of 0.11±0.02 V, with lower voltages obtained for the second anode (0.08±0.02 V) and third anode (0.03±0.02 V; top, effluent; Fig. 6). Together, this resulted in a combined current density of 55±15 mA/m^2 (Fig. 7). This current was 2.8 times higher than that obtained with all the anodes wired together across a single resistor (20±13 mA/m^2, 0.08±0.05 V, R_{ext}=1,000 Ω; Fig. 6).

The second operational configuration that was used to increase performance was to hydraulically connect the reactors in series, so that the effluent from the first MFC was fed into the second MFC (all anodes in each MFC were connected to the cathode via a single resistor circuit; Fig. 1c). It was reasoned that this flow condition would provide more constant COD conditions among the anodes in the two reactors. The total HRT was set at 8 h for the two MFCs in series (4 h per reactor). The first MFC produced an average current density of 85±13 mA/m^2 and second MFCs produced 60±13 mA/m^2

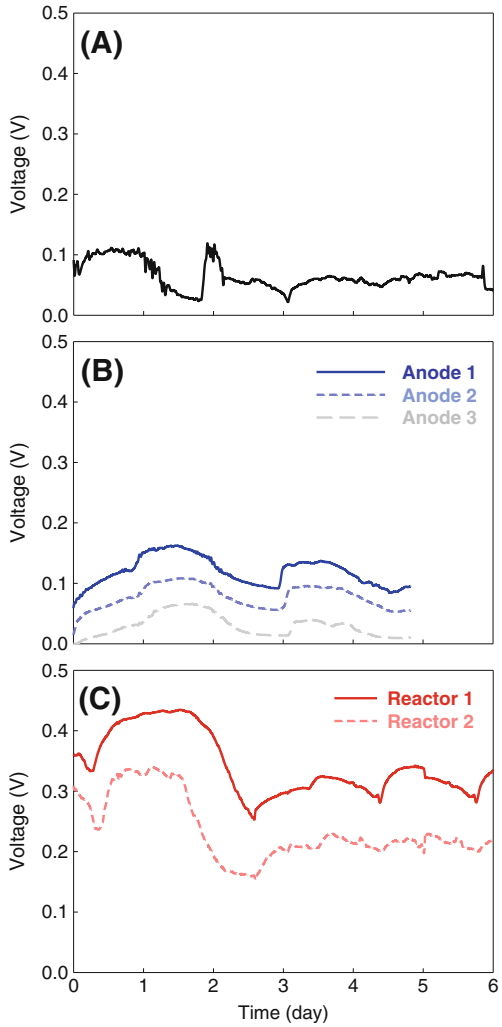

Fig. 6 MFC cell voltages in **a** control, **b** multi-electrode, and **c** in series mode operation at an HRT=8 h with a 1,000-Ω resistor

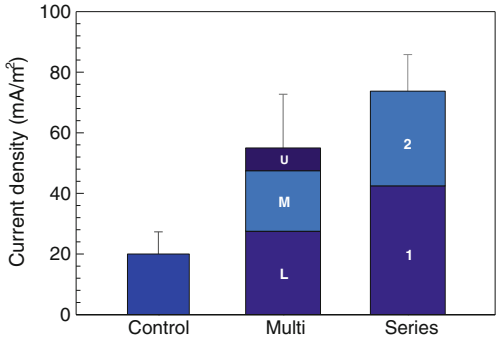

Fig. 7 Comparison of overall current production in different operational modes: control, all anodes connected together; multi, anodes connected to individual circuits; series, two MFCs hooked in series to produce the same HRT as that used for the control and multi tests (HRT=8 h; *L* lower, *M* middle, *U* upper anodes in multi-circuit mode; *1* first MFC and *2* second MFC in the sequenced reactors)

for a total of 73±18 mA/m^2 (Fig. 6). This is 1.3 times higher than that obtained when each anode was connected to an individual resistor and 3.7 times higher than obtained with all the anodes wired together across a single reactor (Fig. 7). The change in the COD concentration was 29 % in the first reactor and 31 % in the second reactor, for an overall change in COD of 60 % during continuous flow operation. This total change in COD was similar to that obtained using a single MFC operated at an HRT=8 h, where the total change in COD in the reactor was 57 %.

Discussion

Current and power densities produced by the multiple-anode MFCs under continuous flow conditions, when the anodes were wired together, were substantially lower than those produced during fed-batch mode operation (Fig. 4). One important difference between these tests is that a fed-batch cycle lasted 3.7 days, so that the COD concentration was uniformly high at the beginning of the cycle. However, in the continuous flow tests with HRTs of 8 or 16 h, there was a 52 % change in the COD across the reactor. It was found in a previous study with a single anode (Ahn and Logan 2010) and domestic wastewater that continuous flow operation actually produced slightly higher power densities (422 mW/m^2) than those obtained under fed-batch operation (334 mW/m^2). However, in that study, the HRT was only 13 min, and thus there was only a 26 % change in the COD during flow through the reactors. Thus, the large COD changes that occurred during continuous flow operations with a long HRT help to explain the lower power production obtained here in continuous flow tests compared to fed-batch tests. However, the very low power densities obtained in continuous flow tests must also have been due to the use of multiple anodes combined with these large changes in COD.

By changing the electrical wiring so that anodes were not connected to each other, but instead individually connected to the cathode, total current production increased 2.8 times. This demonstrated that performance was limited by connection of the anodes to each other under conditions where the anode potentials could be much different. It is known that electrically connecting MFCs in series will produce voltage reversal (Oh and Logan 2007), resulting in much lower power output than that expected from adding up the power produced by the individual reactors. Voltage reversal is easily produced in MFCs electrically connected in series by having different COD concentrations in the reactors (Oh and Logan 2007; Kim et al. 2011b). During continuous flow operation here, the influent and effluent COD concentrations were much different, similar to the conditions that produce voltage reversal because of the large differences in COD between the electrodes. As a result, with all three anodes connected together, the average potential produced was much lower than that expected. However, by separating the anodes into different circuits, the overall system performance could be improved, as seen by improved current production.

The most effective method to improve performance of the MFCs was to reduce the HRT in the reactor and create more uniform COD concentrations across the multiple electrodes. With the two MFCs connected hydraulically in series (each with a 4-h HRT) with the same amount of wastewater treated as two MFCS each at an HRT=8 h, current production was increased by 3.7 times.

The maximum power density obtained here in fed-batch mode tests (120 mW/m^2) was still lower than that obtained using smaller MFCs (334 mW/m^2, 28 mL) that did not have a separator, with domestic wastewater from the same treatment plant (Ahn and Logan 2010). However, the initial CODs and solution conductivities in the previous study (440–490 mg/L, 1.80±0.54 mS/cm) were higher than those measured here (232±84 mg/L, 1.4±0.2 mS/cm), and both of these wastewater characteristics would have improved performance. The lack of a separator would also have improved performance (Ahn and Logan 2010), although having a separator allows for more compact reactor designs. In tests with domestic wastewater using smaller SEA-type MFCs that had separators and similar CODs (390±89 mg-COD/L), the maximum power densities were only slightly larger (130–240 mW/m^2; Hays et al. 2011) than those obtained here. One possible reason for slightly lower power densities here could be the different electrode specific surface areas in the reactors, as total cathode surface area is known to limit performance (Freguia et al. 2007; Cheng and Logan 2011). However, the cathode specific areas were similar in the two studies (27 m^2/m^3 here versus 25 m^2/m^3 in the smaller MFCs), and the anode specific surface area based on projected area here (20 m^2/m^3) was actually slightly higher than that in the smaller MFCs (18 m^2/m^3). The total anode surface area based on the individual fibers was much lower here (2,600 m^2/m^3) than that used in the smaller MFCs (4,107 m^2/m^3), but other data suggest that this difference was not important when brush projected area relative to the cathode was kept constant (Hutchinson et al. 2011). Thus, this suggests that these differences in power production were due to natural variations in the composition of the wastewaters over time.

In conclusion, we showed that it is important to avoid large changes in COD concentrations across multiple anodes when they are wired together. The most effective method for achieving good performance of the multi-electrode MFC was to use multiple reactors hydraulically linked in series, with shorter HRTs per reactor, as this produced more consistent COD concentrations within the reactors. Even though MFCs will produce different voltages based on different average CODs in the reactors linked in series, the power from these MFCs can be efficiently combined using circuits that incorporate capacitors (Kim et al. 2011b). Therefore, when large arrays of MFCs are used for wastewater treatment, the reactor should be arranged with hydraulic flow in series through the reactor, with short HRTs per reactor. Operating arrays of MFCs under these conditions will enable both efficient wastewater treatment and maximum harvesting of electrical power generation. Additionally, more studies are needed to increase the CEs to minimize energy losses to alternate electron-accepting processes in the MFC.

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