Journal of Power Sources 257 (2014) 454–460

Contents lists available at ScienceDirect

Journal of Power Sources

journal homepage: www.elsevier.com/locate/jpowsour

Electrochemical study of multi-electrode microbial fuel cells under fed-batch and continuous flow conditions

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HIGHLIGHTS

- Electrochemical study of multi-electrode MFCs with different electrical connections.
- Polarization tests were needed to compare individual reactors with combined MFCs.
- Same power was produced by combined and individual MFCs in fed-batch.
- Slightly lower power was produced by combined MFCs in continuous flow.
- Parasitic current flow did not appreciably impact reactor performance.

ARTICLE INFO

Article history:
Received 14 September 2013
Received in revised form
5 November 2013
Accepted 22 November 2013
Available online 18 December 2013

Keywords:
Multi-electrode
Microbial fuel cells
Hydraulic connection
Electrical connection
Continuous flow

ABSTRACT

Power production of four hydraulically connected microbial fuel cells (MFCs) was compared with the reactors operated using individual electrical circuits (individual), and when four anodes were wired together and connected to four cathodes all wired together (combined), in fed-batch or continuous flow conditions. Power production under these different conditions could not be made based on a single resistance, but instead required polarization tests to assess individual performance relative to the combined MFCs. Based on the power curves, power produced by the combined MFCs (2.12 ± 0.03 mW, 200 Ω) was the same as the summed power (2.13 mW, 50 Ω) produced by the four individual reactors in fed-batch mode. With continuous flow through the four MFCs, the maximum power (0.59 ± 0.01 mW) produced by the combined MFCs was slightly lower than the summed maximum power of the four individual reactors (0.68 ± 0.02 mW). There was a small parasitic current flow from adjacent anodes and cathodes, but overall performance was relatively unaffected. These findings demonstrate that optimal power production by reactors hydraulically and electrically connected can be predicted from performance by individual reactors.

1. Introduction

A microbial fuel cell (MFC) is a device that converts chemical energy from biodegradable substrates to electrical energy via microbially-catalyzed redox reactions [1–3]. MFCs have been used to produce electricity while simultaneously treating many different types of wastewaters [4–7]. Studies on the scale-up of MFCs containing multiple electrodes have shown the importance of optimization of electrode spacing and increasing specific surface area (surface area of the electrode per volume of reactor) to improve performance [8,9]. Building larger reactors simply by increasing the electrode and reactor sizes (i.e., larger electrodes in larger tanks) can result in decreased volumetric power output compared to smaller bench scale reactors [8–11]. The use of many smaller electrodes in stacks of hydraulically-coupled reactors has therefore been proposed as a more effective method for scale-up [12–15].

Multiple MFCs should not be electrically connected in series as this can substantially reduce power production. Electrically connecting fuel cells or batteries in series normally will increase the voltage in proportion to the number of individual units. However, connecting MFCs in series can produce voltage reversal, resulting in little voltage gains or even elimination of power production [16,17]. Factors that result in voltage reversal include different internal resistances between the units, or unequal voltage production due to differences in substrate concentrations [16,18,19]. Instead of connecting the units electrically together in series to increase voltage, higher voltages can also be obtained by using DC–DC power conversion systems or by charging arrays of capacitors in parallel that are then discharged in series [20].

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http://dx.doi.org/10.1016/j.jpowsour.2013.11.085

Practical applications of MFCs will require operation under continuous flow conditions, but hydraulic flow through arrays of MFCs can adversely affect power production and COD removal relative to that expected from individual reactor performance [14,21,22]. Wastewater can be processed through multiple MFCs in one of two ways: sequentially through all reactors (hydraulically connected in series) [12,23]; or divided up to flow through each individual MFC (hydraulically in parallel) [17]. Series flow can minimize the substrate concentration change in each reactor (i.e., difference between inlet and outlet concentration) and this approach has been used in several studies [12,24,25]. Parallel flow will produce similar conditions in all reactors [10,17], but a low desired effluent COD concentration would result in a large substrate gradient in each MFC. This large change in COD, in a single reactor with multiple anodes wired together, has been shown to adversely affect power production [19]. The same phenomenon occurs when multiple MFCs are wired together under conditions where there is an ionic connection between the electrodes (i.e., the electrodes of different units share the same fluid chamber) [22]. To avoid ionic connections between adjacent electrodes, flow through a series of MFCs was arranged in one study so that the water cascaded (overflowed) from one MFC to another [13]. This separation of the MFCs avoided direct fluid connections, and thus severed solution ionic connections. Alternatively, ionic separation can be achieved by using large constrictions in the flow path (creating very high ionic resistances between adjacent cells), or the cells can be widely separated [25,26]. The optimal condition is to have no electrolyte connection between these reactors [18], but that would not be possible in larger MFCs that contain multiple anodes or cathodes as these electrodes all share the same electrolyte.

The aim of this study was to better understand the reasons why power production decreases when multiple anodes are wired together, under conditions where there are large substrate concentration changes. To study how substrate concentration changes might affect power production in a multi-electrode reactor, we hydraulically connected four MFCs in series to simulate the operation of single MFC containing multiple anodes and cathodes. The electrical connections between the reactors were either set with completely individual circuits between the paired anodes and cathodes, or they were combined into a single circuit with all four anodes wired together and connected to four cathodes all wired together. Power production with this parallel electrical connection was compared to the summed power produced by the individually wired MFCs to determine how the electrical connections between the electrodes affected performance. These comparisons with the two different electrical connections were made using polarization data for MFCs operated in either fed-batch mode or continuous flow conditions with hydraulic flow in series through the four reactors. Continuous flow operation produced conditions that resulted in large substrate concentration changes across the multiple electrodes, allowing examination to how substrate changes affect overall performance. The individual potentials of the electrodes were measured using reference electrodes and individual current using resistances on the different anodes and cathodes, allowing a more comprehensive characterization of the multi-electrode MFCs.

2. Materials and methods

2.1. Reactor construction

Single-chamber, air-cathode MFCs were made of cube-shaped Lexan blocks, each having a single cylindrical chamber with a volume of 14 mL (7 cm^2 cross sectional area) as previously described [7]. Windows (20 mm length × 6 mm width) were cut in the center on the left and right sides of the block to allow hydraulic flow between MFC reactors aligned side by side (Fig. 1). This hydraulic connection of the individual cells enabled simulation of a single multi-electrode system. The total liquid volume of the four connected MFC reactors was 58 mL. Anodes were non-waterproof carbon cloth (#CCP40, Fuel Cell Earth, USA) with a projected surface area of 7 cm^2. Air cathodes (7 cm^2) made of waterproof carbon cloth (30 wt.%, #CC640WP30, Fuel Cell Earth, USA) had a catalyst loading of 0.5 mg-Pt/cm^2 on the water side, and four PTFE diffusion layers on the air side [27]. The electrode spacing was 2 cm (anode surface to cathode surface). The distance between the main liquid chambers of each MFC was 2 cm. A reference electrode (Ag/AgCl; +200 mV versus a standard hydrogen electrode (SHE); BASi) was inserted into the middle of each of the four MFCs to determine the anode and cathode potentials (Fig. 1b). Additional tests were conducted with four MFCs connected hydraulically in series using very thin needles (21 G × 1, BD™ sterile hypodermic needle, BD, USA) to reduce ionic connections between the reactors.

A tracer test was conducted using abiotic reactors to determine whether there was flow short circuiting. A KCl solution (1 mol L^{-1}) was used as the conservative tracer, with an input of 1 h duration. The tracer concentration at the outlet was measured using a conductivity meter over a period of 34 h. The experimental data was modeled using both dispersion model and CSTRs in series model [28].

2.2. Reactor operation

MFCs connected by individual circuits were compared to the four MFCs wired together (combined circuit) under fed-batch mode and continuous flow mode in terms of power production. For individual circuit connections, each MFC was connected through a separate external resistor (see Supporting information, Scheme S1a). For combined circuit connections, the four anodes were wired together using copper wires and then connected through a single external resistor to all the cathodes similarly wired together with copper wires (Scheme S1b). The four MFCs with individual electrical connections were designated as R1–R4 (duplicates R5–R8). For combined electrical connection, the MFC was designated as M1234 (duplicate M5678).

The MFCs were each initially acclimated using an individual circuit connection with a 1000 Ω external resistor in fed-batch mode, with the growth medium replaced when the voltage decreased to <0.05 V. Polarization and power data were obtained for both the individual connections and the combined connections after the MFC reactors exhibited stable performance, defined as reproducible voltage output over at least three consecutive cycles. Following batch tests, the MFCs were switched to continuous flow operation, with medium flowing sequentially through reactors 1–4 (Fig. 1b). The hydraulic retention time (HRT) was set at 12 h using a flow rate of 0.08 mL min^{-1}. All the experiments were conducted in duplicate in a temperature controlled room at 30 °C.

The medium contained (per liter): 0.5 g CH_3COONa, 10 mL vitamins, and 10 mL minerals [12,20] in a 50 mM PBS buffer (0.31 g NH_4Cl, 2.45 g $NaH_2PO_4\cdot H_2O$, 0.13 g KCl, 4.58 g Na_2HPO_4; $pH = 7.1 \pm 0.2$, conductivity $\gamma = 7.6 \pm 0.2$ mS cm^{-1}). The medium was autoclaved and then placed in an ice bucket during tests to avoid degradation prior to being fed into the reactors in continuous flow tests. The fluid warmed during transfer into the MFC, avoiding temperature differences between tests.

2.3. Chemical and electrochemical analysis

Soluble chemical oxygen demand (sCOD) was measured using standard methods (method 5220, HACH COD system, HACH Company, Loveland, CO) [29]. All samples for sCOD measurement were filtered through 0.45 μm pore diameter syringe filters

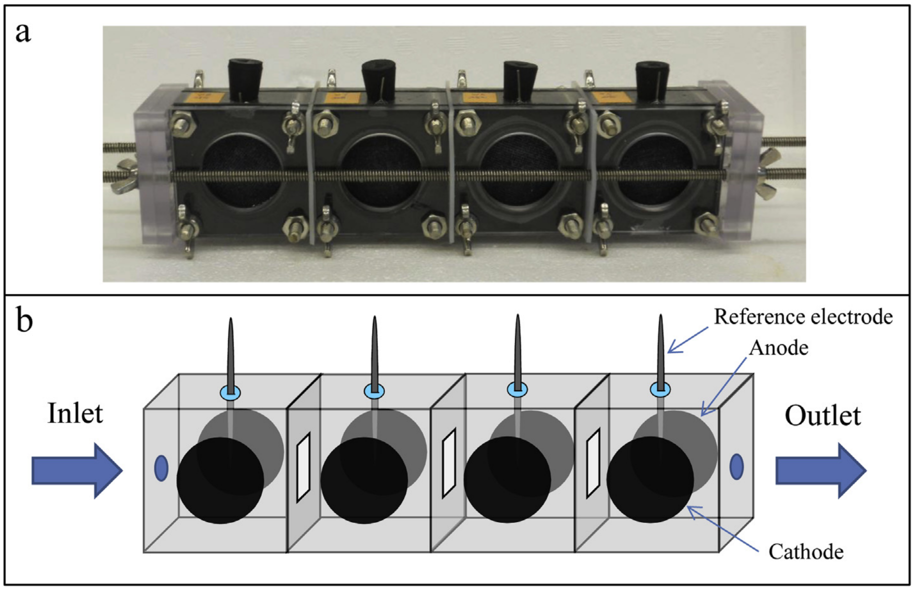

Fig. 1. Configuration of the four hydraulically connected MFCs: (a) photo showing the outside structure and (b) schematic drawing showing the hydraulic connection.

(polyvinylidene difluoride, PVDF, 25 mm size, Restek Corporation, USA). Conductivity and pH were measured using a probe (SevenMulti, Mettler-Toledo International Inc.).

Voltage was recorded using a multimeter (model 2700; Keithley Instruments, Inc.) at 20 min intervals, with the power calculated as $P = IU$, and the current calculated using Ohm's law ($I = UR^{-1}$), where U is the measured voltage (V), and R the external resistance (Ω) [30]. For fed-batch tests, polarization data were obtained using the multiple-cycle method (two cycles each) by changing the external resistances from 4000 to 1000, 500, 200, 120, 80 and 40 Ω (individual connections), or from 1000 to 250, 125, 40, 30, 20 and 10 Ω (combined connections). In continuous flow tests, the external resistances were changed from 4000 to 2000, 1000, 500, and 300 Ω (individual connections), or from 1000 to 500, 250, 125, and 75 Ω (combined connections), with a single resistor used for a minimum of two days. All electrode potentials were reported versus the Ag/AgCl reference electrode. Potential gradients in the electrolyte phase were measured as potential differences between the reference electrodes in the four different MFCs. In some combined electrical connection experiments, an additional 10 Ω resistor was introduced in series with each anode and cathode in order to measure the current of each individual electrode.

3. Results

3.1. Power production

In fed-batch mode, the performance of the individual MFCs (R1–R4) relative to each other was the same, based on power curves obtained from reactors after being fed with fresh medium, with a maximum power of 0.53 ± 0.03 mW at a current of 1.63 ± 0.01 mA (200 Ω) (Fig. 2a). The average power curve for the four reactors was therefore produced by averaging the current and power of the four individual MFCs at each external resistance (i.e., the average). The maximum power that could be obtained from the four MFC reactors based on summation of the power of the individual reactors (2.12 ± 0.03 mW, 6.53 ± 0.04 mA, 200 Ω) was the same as that obtained with all electrodes wired together (2.13 mW, 6.51 mA, 50 Ω, M1234, Fig. 2b).

The power produced by the individual MFCs under continuous flow conditions relative to each other was quite different from that obtained in fed-batch tests, due to differences in substrate concentrations among the reactors. The power produced by the MFCs

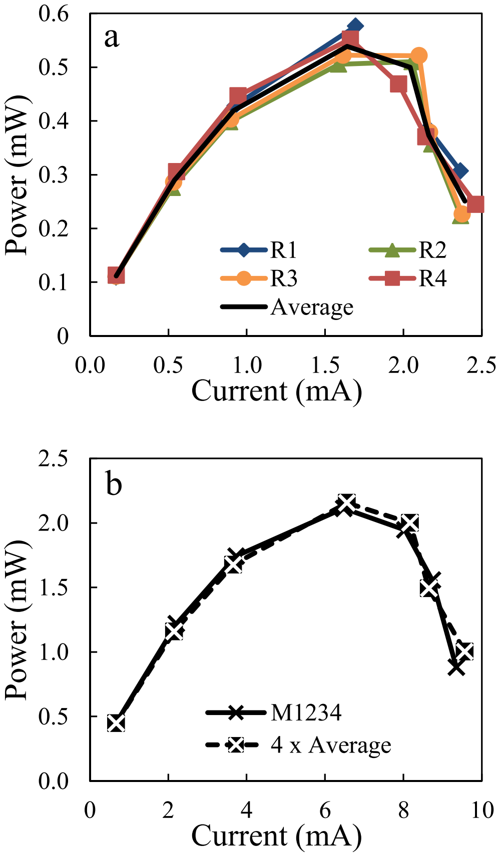

Fig. 2. Power production of the MFC reactors in fed-batch mode. (a) Individual connection showing the identical performance of the four MFC reactors (R1–R4). (b) Combined connection where the summed power of the four individual reactors (4× average) compared to that of the combined reactor (M1234). Results are shown for two separate sets of reactors (1–4, and duplicates 5–8 in Fig. S2).

decreased from inlet to outlet, with a maximum power for the individually-wired MFCs of 0.23 mW (1000 Ω, R1), 0.20 mW (1000 Ω, R2), 0.16 mW (2000 Ω, R3), and 0.10 mW (4000 Ω, R4) (Fig. 3a). The sCOD concentrations measured in the direction of flow decreased from the inlet concentration of 363 mg L^{-1}, to outlet concentrations of 253 mg L^{-1} (R1), 168 mg L^{-1} (R2), 112 mg L^{-1} (R3), and 86 L^{-1} (R4) (1000 Ω external resistance for each MFC). The reduction in power due to the lower substrate concentration was expected based on previous studies using individual reactors [23,31]. Tracer tests showed that the calculated HRTs using both models (16.5 h for CSTRs in series model and 15.2 h for dispersion model) were slightly larger than the theoretical value of 12 h (Fig. S2), indicating there was no flow short circuiting in the reactors. This lack of short circuiting is also supported by the large changes in COD along the direction of flow.

Under continuous flow condition, a comparison was made between the summed maximum power produced by the four individual MFCs and that of the combined MFCs with the electrodes wired together, as no averaged power curve could be calculated due to the different performance of the individual reactors. The maximum power produced by each individually-wired MFC was summed as 0.68 ± 0.02 mW (the average of the maximum power densities of R1–R4, and those for R5–R8). This was slightly higher (15%) than the maximum power produced by the combined MFCs with the electrodes wired together (0.59 ± 0.01 mW, averaged for M1234 and M5678) at an external resistance of 500 Ω (Fig. 3b). Additional tests conducted using reactors connected hydraulically with needles to break the ionic connections between the reactors also showed similar reactor performance between the individual circuit connections and the combined circuit connections as that of the MFCs hydraulically connected by windows, even under continuous flow conditions. The summed maximum power produced by the individual MFCs in these tests was 0.64 ± 0.01 mW, also slightly higher than that produced by the combined MFCs (0.59 ± 0.01 mW, 500 Ω) (Fig. 4). The maximum power in continuous flow was lower than that obtained in fed-batch tests (2.13 ± 0.03 mW) due to the lower substrate concentrations in the MFCs at steady state under continuous flow conditions, compared to that present in the reactors at the start of the fed-batch test [12].

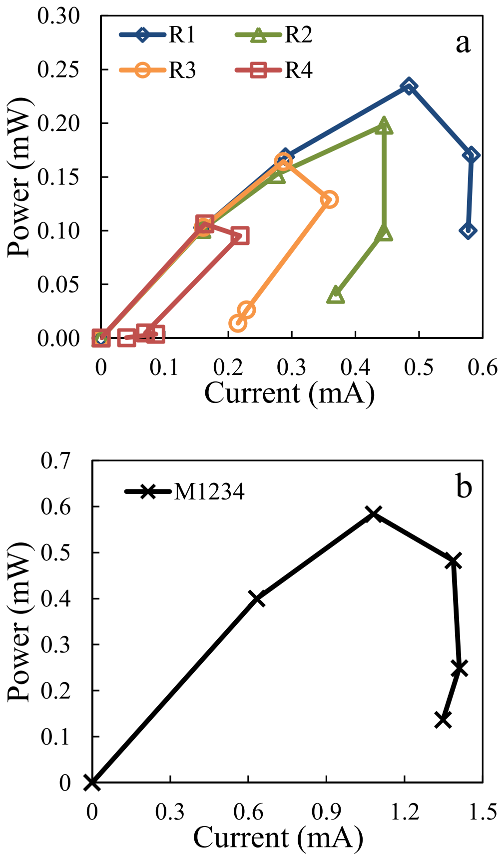

Fig. 3. Power production of the MFC reactors operated in continuous flow with the flow from R1 to R4. (a) Individual connection showing the different performance of the four MFC reactors (R1–R4). (b) Combined connection (M1234). Results are shown for two separate sets of reactors (1–4, and duplicates 5–8 in Fig. S3).

3.2. Effect of external resistance

The external resistances used for comparison of the combined system with the individual MFCs are particularly important for comparisons of power production. When the anodes and cathodes of multiple MFCs were electrically connected together to form a single anode and cathode connection, it was determined that the external resistance had to be reduced compared to that used for individual connections in order to obtain the maximum power. In fed-batch operation, the four combined MFCs produced their maximum power at 50 Ω, compared to 200 Ω needed for the MFCs with separately wired anodes and cathodes (Fig. 2). Similarly, the maximum power produced by the combined MFCs under continuous flow conditions was obtained at an external resistance of 500 Ω, compared to those (1000 Ω, 2000 Ω and 4000 Ω) for the individual MFCs (Fig. 3).

These results on the maximum power at different resistances suggested that polarization and power curves should be conducted to compare the combined electrode system with the individual MFCs, instead of measuring the power production under a single resistance. For example, when a 2000 Ω resistance was used for the four MFC reactors electrically combined in one circuit, the power was 0.24 ± 0.01 mW, compared to 0.65 ± 0.04 mW obtained by summation of the power produced by the four individual reactors at 2000 Ω (Fig. 5). However, when the resistance was decreased to 500 Ω, the power produced by the four combined MFCs increased to 0.64 ± 0.01 mW (Fig. 5). Additional examples at different external resistances in both fed-batch operation and continuous flow were provided in Supporting Information (Figs. S6 and S7).

The reduced external resistance used for the combined MFCs to achieve comparable power to that produced through summation of power of the individual MFCs, indicated a decrease in internal resistance for the combined MFCs. Theoretically, for n batteries with same electromotive force (Emf) and internal resistance, an external resistance used for the combined circuit to obtain the same power production to the summation of individual circuits should be $1/n$, compared to that used in the individually-wired electrode, where n is the number of electrodes combined together. This rule can be applied for the multi-electrode MFCs operated in the fed-batch operation, because four reactors had the same performance. However, in continuous flow tests the four MFCs had different internal resistances (Fig. 3a) due to the large substrate gradient in the direction of flow. When the four MFCs were combined to form one circuit, the apparent internal resistance also decreased, but the value was dependent on the individual Emfs and internal resistances. Future studies on the systematic analysis of the internal resistances of the individual MFCs and the combined MFCs, either by impedance tests or modeling, would be interesting and helpful for a better understanding of changes in reactor performance in multi-electrode systems.

3.3. Current profiles for combined electrodes

When multiple anodes or cathodes are connected together, parasitic current could affect the performance of an adjacent

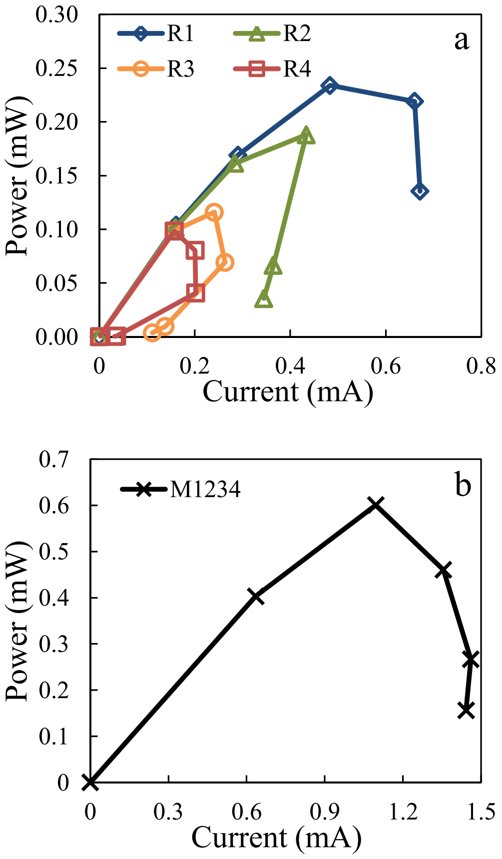

Fig. 4. Power production of the MFC reactors hydraulically connected using needles in continuous flow with the flow from R1 to R4. (a) Individual connection showing the different performance of the four MFC reactors (R1–R4). (b) Combined connection (M1234). Results are shown for two separate sets of reactors (1–4, and duplicates 5–8 in Fig. S5).

electrode. This parasitic current could arise from differences in potentials between adjacent electrodes that were produced, for example, by differing substrate concentrations among the electrodes. In continuous flow tests, the sCOD concentration was reduced during flow from R1 to R4. Thus, the anode potentials in the direction of flow increased from −0.39 ± 0.01 V (R1) to −0.32 ± 0.02 V (R2), −0.23 ± 0.02 V (R3), and −0.15 ± 0.01 V (R4) (results using a 125 Ω external resistance). Based on measurements of voltages from each anode and cathode (obtained using a small 10 Ω resistor in each electrical connection to monitor current), the R4 anode (R4A) produced very little current (0.014 mA), compared to the current flowing into the opposing R4 cathode (0.18 mA, R4C) (Fig. 6). This imbalanced current flow between the opposing electrodes indicated that there was an ionic current from the anode of the adjacent MFC (R3A) to the opposing cathode (R4C). Similarly, there was also ionic current from the R2 anode (R2A) to the R3 cathode (R3C) as indicated by an unbalanced current between R3A (0.24 mA) and R3C (0.37 mA). The parasitic current flow here was primarily due to the poor performance of the last anode (R4A), as it had very little current production likely due to the low effluent substrate concentration. As a result of the parasitic current flow, more current was produced by R2A (1.09 mA) and R1A (1.25 mA) than those by their opposing cathodes (R2C, 0.90 mA; R1C, 1.18 mA). As a result of this improved anode current flow compensating for the reduced anode current flow, overall reactor performance was not significantly affected.

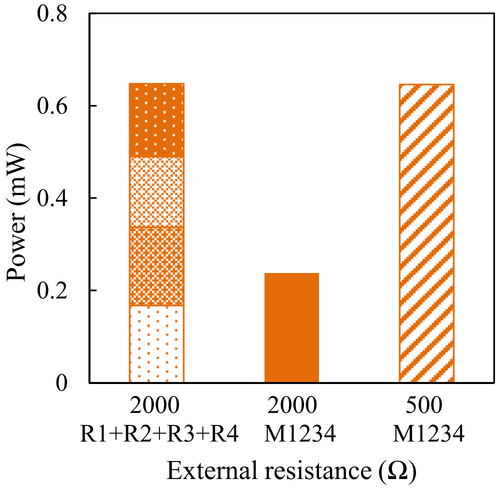

Fig. 5. Power output of the MFC reactors at individual connection (R1–R4, 2000 Ω external resistance) and combined connection (M1234, 2000 Ω and 500 Ω external resistance) in continuous flow. Results are shown for two separate sets of reactors (1–4, and duplicates 5–8 in Fig. S7b).

The effect of low current generation by an anode was further examined by disconnecting anodes (R1 and R3) under non-substrate limiting conditions (1 g L^{-1} sodium acetate) in continuous flow tests (300 Ω external resistance). The four cathodes were always wired together during these tests, while either four working anodes or only two working anodes (R2 and R4) were wired together and connected to the cathodes. When one anode was connected to two cathodes (one opposed, and one adjacent) the current was 1.18 ± 0.01 mA (0.81 ± 0.02 mA by the opposed cathode and 0.37 ± 0.01 mA by the adjacent cathode). This current was slightly higher than obtained by the same anode with a working adjacent anode which produced 1.08 ± 0.01 mA. Parasitic current was also observed from the current producing anodes (R2A and R4A) to the adjacent cathodes (R1C and R3C) opposing the anodes with no current production, resulting in higher anode current production.

4. Discussion

These experiments with multiple reactors connected electrically in different ways revealed two important aspects of MFC operation. First, the performance of an electrode could not be properly assessed without conducting polarization tests on each individual

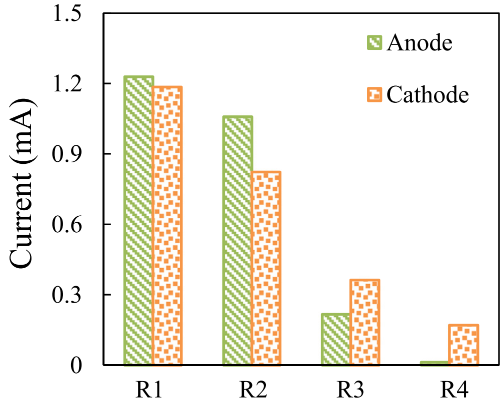

Fig. 6. Current output of the anodes and cathodes at combined connection (M1234, 125 Ω external resistance) in continuous flow. Results are shown for two separate sets of reactors (1–4, and duplicates 5–8 in Fig. S8).

electrode. If only a single resistance was used for testing the power produced by the whole reactor or an individual electrode, the performance of that individual electrode connected separately from other electrodes would appear to be much better than it was when connected with other electrodes. This result was due to the different electrical loads on that electrode when the same resistance was applied for each individual electrode versus the combined electrodes from multiple reactors. Comparisons of power curves for individual versus combined electrodes here indicated that the resistance used for the combined MFCs should be lower than that used for the individual reactors to achieve comparable power production due to a decrease in internal resistance when the anodes and cathodes were connected electrically in parallel (Section 3.2). Previously it was reported that anode performance was reduced when all anodes were connected together in a reactor containing a single cathode [19]. However, this comparison was based on using the same external resistance (1000 Ω) for the individual electrodes and the combined electrodes. Our results here suggest that the resistor used for the combined anodes should have been much lower than the 1000 Ω used for the individual anodes. The best way to compare individual electrode performance is therefore to conduct polarization tests on each individual anode. It was shown here that a power curve based on the summed power produced by the four individual reactors was essentially identical to that of the MFC with the four anodes wired tougher and the four cathodes wired together in fed-batch operation (Fig. 2b). While in continuous flow, the summed maximum power from the power curves of the four individual MFCs was slightly higher (15%) than that from the power curve of the combined MFC (Fig. 3).

A second important finding was that the substrate concentration differences at the anodes, produced under continuous flow conditions, had more of an effect on the performance of multiple reactor systems than parasitic current between the electrodes. In continuous flow operation, the four individual MFCs can have different Emfs and internal resistances at the substrate concentration where they achieved their maximum power. A loss in maximum power could result when MFCs with different Emfs and internal resistances are combined into a single circuit because of the differences in these systems. Therefore, there is no single external load that could be chosen that would allow all the individual reactors to produce their maximum power.

Parasitic current flow between adjacent electrodes, determined from individual electrode measurements and by disconnecting electrodes, did not appreciably affect the overall performance of the multi-electrode MFC, although it did alter the current produced by the individual electrodes. The anode near the exit of the flow from the MFC (R4) produced very little current when wired to other anodes. However, while the R4 anode (R4A) generated little current, the adjacent anode (R3A) generated a higher current as a result of an ionic flux supported by both its opposing cathode (R3C) and the adjacent cathode (R4C). This resulted in greater current generation by R3A than would be possible in the absence of R4C, as further shown in tests comparing performance of anodes with and without the adjacent anode electrically connected to the circuit. As a result of this improved anode current flow compensating for the reduced anode current flow, overall reactor performance was not affected. However, large changes in the flow rate through the reactor, such as very long HRTs, could further alter anode potentials. The effect of HRT on performance was not examined here.

Research by others suggested that completely separating reactors, so that there was no fluid connections between them, might further improve power production compared to the case here with all electrodes exposed to the same fluid [13,17,18]. However, additional tests using reactors connected hydraulically by very thin needles showed the same performance as those connected by windows, in terms of power production for different electrode connections (Figs. 3 and 4), although a very large solution resistance created by the needle bridge had reduced the parasitic current to a level below the detection limit ($<10^{-3}$ mA). This further suggested that parasitic current would not affect MFC performance when the electrodes sharing the same electrolyte were wired together, and thus completely separating reactors might not improve power production.

5. Conclusions

The best way to compare the performance between the combined MFCs and the individual reactors is to conduct polarization tests. Changes in substrate concentration with flow through the reactors can result in differences in internal resistance among the reactors, which can preclude accurate comparison based on a single external resistance. When compared on the basis of polarization data, the MFCs with the electrodes wired together showed no differences in power production from that obtained by summing power from the individually wired reactors in fed-batch operation. Even in continuous flow conditions, where the substrate concentration significantly affected the anode potentials and MFC performance, the maximum power of the combined MFC was only slightly lower than that summed of the four individual reactors. Parasitic current flow measured for MFCs hydraulically connected in series and electrically in parallel showed, no appreciable impact on reactor performance, and thus there was no need for electrolyte isolation between adjacent reactors under such flow conditions.

Acknowledgments

The authors thank David Jones for help with the analytical measurements. This research is supported by Award KUS-I1-003-13 from the King Abdullah University of Science and Technology (KAUST).

Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jpowsour.2013.11.085.

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