

Electricity generation from model organic wastewater in a cassette-electrode microbial fuel cell

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Abstract A new highly scalable microbial fuel cell (MFC) design, consisting of a series of cassette electrodes (CE), was examined for increasing power production from organic matter in wastewater. Each CE chamber was composed of a box-shaped flat cathode (two air cathodes on both sides) sandwiched in between two proton-exchange membranes and two graphite-felt anodes. Due to the simple design of the CE-MFC, multiple cassettes can be combined to form a single unit and inserted into a tank to treat wastewater. A 12-chamber CE-MFC was tested using a synthetic wastewater containing starch, peptone, and fish

extract. Stable performance was obtained after 15 days of operation in fed-batch mode, with an organic removal efficiency of 95% at an organic loading rate of 2.9 kg chemical oxygen demand (COD) per cubic meter per day and an efficiency of 93% at 5.8 kg COD per cubic meter per day. Power production was stable during this period, reaching maximum power densities of 129 W m^{-3} (anode volume) and 899 mW m^{-2} (anode projected area). The internal resistance of CE-MFC decreased from 2.9 (day 4) to 0.64Ω (day 25). These results demonstrate the usefulness of the CE-MFC design for energy production and organic wastewater treatment.

Keywords Microbial fuel cell · Energy recovery · Organic waste · Biomass · Wastewater

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Introduction

Microbial fuel cells (MFCs) are devices that exploit microorganisms to generate electricity from a variety of reduced materials, including organic matter (Logan et al. 2006). An MFC is a new type of renewable and sustainable technology for electricity generation since it recovers energy from renewable materials that can be difficult to dispose of, such as organic wastes and wastewaters. Researchers have also used MFCs to recover electricity from marine sediments (Reimers et al. 2001) and rice paddy fields (Kaku et al. 2008).

Extensive studies performed to identify methods that enhance the performance of MFC reactors have resulted in the production of more efficient laboratory-scale MFCs. These technologies include the use of air cathodes

(Liu and Logan 2004), stacked reactors (Aelterman et al. 2006), cloth electrode assemblies (Fan et al. 2007a), and modification of solution chemistry using a carbonate buffer (Fan et al. 2007b). Among these, the use of air cathodes (Liu and Logan 2004) is particularly noteworthy since it enables the efficient use of oxygen from air and avoids the need for aerating the water or using chemical catholytes such as ferricyanide that must be regenerated. Air cathodes have been optimized for the use in MFCs (Cheng et al. 2006), and the effects of shape and position on MFC performances have been evaluated using different reactor designs (Zuo et al. 2007; Fan et al. 2007a). These efforts resulted in highly efficient small-volume laboratory MFCs (~20 ml in anode volume) that produced electrical outputs of over $1,000 \text{ W m}^{-3}$ (Fan et al. 2007a, b). However, it is still a challenge for MFC researchers to construct large-scale MFCs that have both high power production and stable performance (Zuo et al. 2007). Liu et al. (2008) have recently reported the construction of a 500-ml MFC reactor with the maximum power density of 20 W m^{-3} (Liu et al. 2008).

In the present study, we designed a new type of MFC architecture suitable for use in a large-scale reactor. The reactor consisted of a series of cassette electrodes (CEs) composed of a flat cathode box (i.e., a flat electrode with two sides) sandwiched between two proton-exchange membranes (PEM) and graphite-felt anodes. The CE-MFC was assembled from a series of these CEs so that the complete unit could be inserted into an anaerobic digester. The use of this cassette configuration allows the design of the modular MFC reactor to be flexible in terms of size, shape, and number of units. We report here the performance of a laboratory-scale reactor (approximately 1 l in capacity) that was tested using a synthetic wastewater containing starch, peptone, and fish extract as the fuel.

Materials and methods

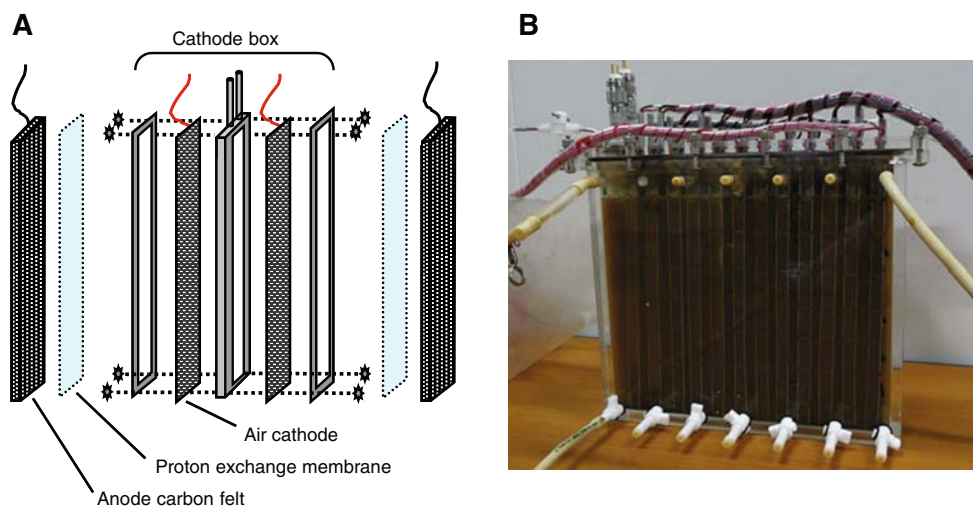
Reactor configuration

A cathode box for each cassette of the CE-MFC (Fig. 1a) consisted of a plastic frame ($4 \times 21.5 \times 1 \text{ cm}$) with the inside of the cathode box open to the atmosphere by two small air tubes (3 mm in diameter). Both sides of the cathode box consisted of air cathodes ($3 \times 19 \text{ cm}$) that were fixed to plastic frames using epoxy to avoid air or water leakage. Each side of the cathode box was covered by a PEM ($3 \times 20 \text{ cm}$, Nafion 117, Dupont Japan, Tokyo, Japan) and a graphite-felt anode ($3 \times 20 \text{ cm}$, 1.5 mm in thickness, Sohgo Carbon, Yokohama, Japan). The air cathode was made according to the method of Cheng et al. (2006) and had 4-polytetrafluoroethylene (PTFE) layers and 0.7 mg/cm^2 of Pt catalyst. The membranes and graphite-felt anodes were loosely attached to the cathode box, so that PEM prevented the anode and cathode from contact to each other. The anode solution could directly contact with air cathode surfaces. The CE-MFC reactor ($27 \times 6 \times 23 \text{ cm}$) contained 12 CEs (units A to L) and had a total liquid volume of 1 l (Fig. 1b). The air tubes of each CE were fixed to the cap of the reactor. The reactor had liquid sampling ports at the bottom, a gas-trap bag (1-6332-03; AS ONE, Osaka, Japan), an oxidation–reduction potential (ORP against the standard hydrogen electrode) probe (DKK-TOA, Tokyo, Japan), a pH control system (Marubishi Bioengineering, Tokyo, Japan), and a water-circulating system (EYELA, Tokyo, Japan). Air was circulated through each cathode box using an air pump (HOHMI, Tokyo, Japan).

CE-MFC operation

The CE-MFC was filled with an inorganic medium (Ishii et al. 2008a) purged of oxygen with nitrogen gas. The

Fig. 1 **a** Configuration of a single CE module. **b** A photo of the CE-MFC containing 12 CEs



inorganic medium (per liter) contained: 0.1 g KCl, 0.2 g NH_4Cl , 0.6 g NaH_2PO_4 , 1-ml vitamin solution (Sekiguchi et al. 2000), 1-ml trace element solution (Sekiguchi et al. 2000), and 1-ml Se/W solution (Sekiguchi et al. 2000). The reactor was inoculated with 50 g (wet weight) of soil obtained from a rice paddy field (Kamaishi, Japan) and fed 20 ml of a model organic waste (MOW) composed of starch (Cat. no. 191-03985, Wako Pure Chemical Industries, Osaka, Japan), Bacto peptone (Cat. no. 211677, Becton Dickinson, MD, USA), and fish extract (Cat. no. 054-03705, Wako) at a 3:1:1 ratio, producing a final chemical oxygen demand (COD) of 289 g l^{-1} . At various times when the COD had decreased, 20 ml of the anode solution was drained and replaced with 20 ml of fresh MOW solution. The reactor was operated at 30°C . Pure water was occasionally added to compensate for water loss from the air cathodes. Anodes and cathodes were connected to a potentiostat (HA-151, Hokuto Denko, Tokyo, Japan) by epoxy-encapsulated wires and operated at a fixed external resistance initially set at 100Ω , but decreased stepwise to 2Ω . Voltage (V) produced by the CE-MFC was monitored using the potentiostat and a PC program (MSR128-V5, M-System, Osaka, Japan). Polarization curves were obtained using the potentiostat machine and the PC program that changed voltage every 1 min in a stepwise manner.

Chemical analyses

COD was measured using a COD reactor and a COD 0–1,500-ppm range kit (Hach, Laveland, CO, USA). Soluble COD was measured using the supernatant of a sample (1 ml) centrifuged at $10,000\times g$ for 10 min (4°C). Particulate COD was measured using the precipitate suspended in 1 ml of water. The total COD (T-COD) was calculated from the sum of soluble and particulate CODs.

Headspace gas composition (methane, hydrogen, carbon dioxide, nitrogen, and oxygen) was analyzed using a gas chromatograph (GC-14A; Shimadzu, Kyoto, Japan) equipped with a thermal conductivity detector and a molecular sieve 5A 60–80/Porapack Q 80–100 column (Shimadzu) as described previously (Ishii et al. 2005). A daily gas production rate was determined by measuring volume of gas collected in a gas bag. Organic acids (pyruvate, malate, succinate, lactate, fumarate, acetate, propionate, and butyrate) were measured by liquid chromatography using a LC-6A HPLC system (Shimadzu) equipped with an electroconductivity detector (CDD-10AVP; Shimadzu), a Shim-pack SCR-102H column (8 mm i.d. \times 300 mm; Shimadzu), and a guard column (SCR-102H, 6 mm i.d. \times 50 mm; Shimadzu) according to the manufacturer's instructions. Organic acids were eluted with 5 mM *p*-toluenesulfonic acid solution at a flow rate of

0.8 ml/min at 45°C . Before acidic eluents were introduced to the detector, they were reacted with 20 mM bis(2-hydroxyethyl)-iminotris(hydroxymethyl) methane aqueous solution containing 5 mM *p*-toluenesulfonic acid and $100 \mu\text{M}$ ethylenediamine tetraacetic acid.

Calculations

MFC performance was evaluated after the voltage produced was stable (from day 28 to 48) according to methods described by Logan et al. (2006). An organic removal efficiency (ORE [%]) was calculated from the influent (T-COD_{in}) and effluent COD (T-COD_{out}) as $\text{ORE} = [\text{T-COD}_{\text{in}} - \text{T-COD}_{\text{out}}] / \text{T-COD}_{\text{in}}$. Current ($I = V/R$) and power ($P = IV$) were calculated using the measured voltage (V) for the set resistance (R). Coulombic efficiency was calculated based on total COD removal ($\text{T-COD}_{\text{in}} - \text{T-COD}_{\text{out}}$) and the measured current using: $1 \text{ g of COD} = 0.125 \text{ mol of}$

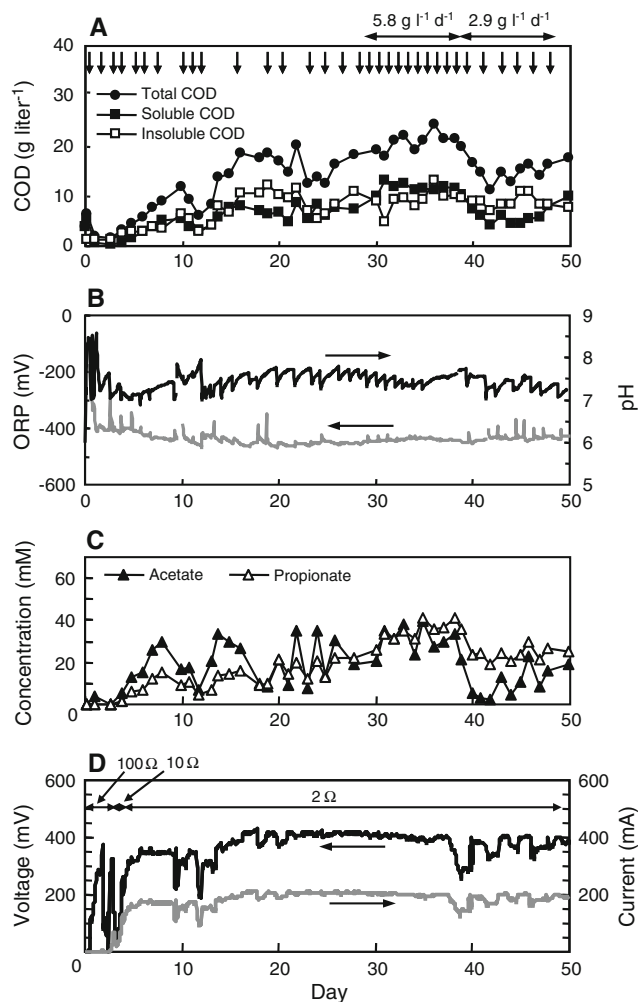


Fig. 2 Time course study of operational parameters of the CE-MFC: **a** MOW loading and COD values; **b** oxidation–reduction potential (ORP) and pH values for the anode solution; **c** acetate and propionate concentrations in the reactor; **d** external resistors used at different times and voltage and current generation

Table 1 Summary of MFC performance

Parameter	Value for:	
	Organic loading rate of 5.8 g l ⁻¹ d ⁻¹ (days 28 to 38)	Organic loading rate of 2.9 g l ⁻¹ d ⁻¹ (days 38 to 48)
T-COD _{out} (g l ⁻¹)	20.5	15.1
Organic removal efficiency (%)	93	95
Current (mA)	202	183
Maximum power density per anode volume (W m ⁻³)	129	117
per anode projection area (mW m ⁻²)	899	797
per anode projection area (mW m ⁻²)	946	839
OCV (V)	0.56	0.56
SCC (mA)	530	505
R _{int} (Ω)	0.64	0.74
Coulombic efficiency (%)	28	48
Methane production rate (mmol day ⁻¹)	4.0	0.11

electron; 1 A=5.39×10²³ electrons per day (Logan et al. 2006). Polarization and power density curves were calculated as previously described, and internal resistance (R_{int}) was estimated from the slope of a polarization curve (Logan et al. 2006). Methane production rate was estimated as described elsewhere (Ishii et al. 2005) using gas composition and gas volume data.

Results

The ORP rapidly decreased to approximately -400 mV following inoculation and was relatively stable at this value for the rest of the operation time (Fig. 2b). For the initial few days, the pH was unstable and it was necessary to adjust the pH by adding an NaOH solution, but after day 4 the pH remained between 7 and 8 (Fig. 2b). Adding MOW, however, resulted in a rapid pH decrease of 0.5 units or less, but it gradually returned to the original level. Acetate and propionate were consistently detected in the reactor solution, while other organic acids were either below the detection limit or less than 5 mM (Fig. 2c). Thus, the reduction in pH was likely due to the rapid formation of these two volatile fatty acids, followed by their removal.

Voltage generation was detected after day 2, and the fixed external resistance was reduced from 100 to 10 Ω on day 3, and then to 2 Ω on day 4 to maximize power production (Fig. 2d). Power generation was stable (approximately 80 W m⁻³ [anode volume] and 558 mW m⁻²

[anode projection area]) from day 15, and the effluent T-COD_{out} was approximately 15 g l⁻¹ (Fig. 2a). On day 28, the organic loading rate was increased to 5.8 g l⁻¹ day⁻¹ by adding MOW solution once a day, after which the T-COD_{out} increased and stabilized at 20.5±1.5 g l⁻¹ (mean±SE). The organic removal efficiency during this period was approximately 93%, and the electrical output did not increase during this period.

On day 38, the loading rate was decreased to 2.9 g l⁻¹ day⁻¹ (MOW was added once every 2 days), resulting in the T-COD_{out} value being restabilized at 15.1±1.8 g l⁻¹ [mean±SE]. Thereafter, however, the electric output slightly decreased. Methane production rates were 4.0±0.2 and 0.11±0.06 mmol day⁻¹ (mean±SE), at loading rates of 5.8 and 2.9 g l⁻¹ day⁻¹, respectively. Hydrogen was not substantially produced during MFC operation (<0.001 mmol day⁻¹). Operational parameters estimated at these loading rates are summarized in Table 1.

In order to evaluate changes in electrochemical properties of the CE-MFC, polarization and power density curves were obtained at several points during operation (Fig. 3). The maximum power density and R_{int} were calculated based on these curves as summarized in Table 1 (data obtained from days 28 to 48). The shapes of the polarization and power density curves were essentially unchanged after day 25, even though the loading rate was varied between 2.9 and 5.8 g l⁻¹ day⁻¹ (Fig. 3). R_{int} decreased from 2.9 (day 4) to 0.64 Ω (day 25) according to the enrichment of electricity-generating microbes. Coulombic efficiency was calculated based on organic removal rates (an organic loading rate multiplied by an organic removal efficiency) and a mean current, and values obtained for the reactors operated at the organic loading rates of 5.8 and 2.9 g l⁻¹ day⁻¹ were 28% and 48%, respectively.

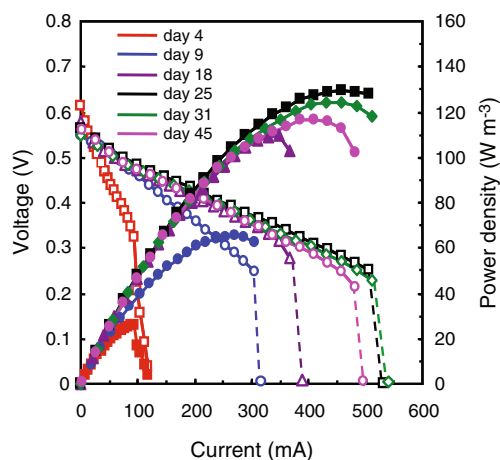


Fig. 3 Polarization and power density curves of CE-MFC at various time points. Lines with open symbols are polarization curves, while lines with closed symbols are power density curves. Curves for different days are indicated with different colors as indicated in the figure

Table 2 Performance of each CE unit on day 48

CE unit	R_{int}^a (Ω)	Max. power density (W m^{-3})
A	5.0	14.1
B	7.8	10.9
C	5.4	11.9
D	5.0	16.0
E	4.6	17.7
F	5.5	14.9
G	5.4	12.8
H	5.1	15.8
I	4.8	17.4
J	4.4	18.4
K	5.1	15.5
L	5.1	16.5
Mean \pm SE	5.3 \pm 0.2	15.1 \pm 0.7
Estimated total performance ^b	0.43 ^c	182 ^d
Measured total performance	0.60	115

^a Internal resistance of each CE unit

^b Total CE-MFC performance estimated from the data for the 12 CEs

^c Estimated according to the following equation: $1/R_{\text{int}} = \sum 1/r_{\text{int}}(i)$

^d Sum of the power densities of units A to L

To examine performance of each individual cassette, we measured polarization and power density curves for each of the 12 CEs (data not shown) on day 48 and calculated the R_{int} and maximum power density (Table 2). We found comparable performance for each CE, with a mean of $15.1 \pm 0.7 \text{ W m}^{-3}$ (a range of 10.9 to 18.4 W m^{-3}). The internal resistances averaged $5.3 \pm 0.2 \Omega$ (range of 4.4 to 7.8Ω). When these values were used to estimate total performance of CE-MFC (by summing performance values for each CE), these estimates were somewhat superior to the values experimentally determined (Table 2). We postulate that this was because the anodes of one CE were connected to those of neighbor CEs, and electrons harvested at neighbor anodes also contributed to the current generation from one CE. It is suggested that the total performance of CE-MFC is dependent on a number of CE units.

Discussion

It was shown here that a new type of MFC could successfully treat a wastewater, achieving a removal efficiency of 93% at an organic loading rate of $5.8 \text{ g l}^{-1} \text{ d}^{-1}$. These values are equivalent to those for conventional anaerobic digesters (Chynoweth and Isaacson 1987). In addition, the Coulombic efficiency values were 28% to 48%, which are equivalent to or higher than electricity-generation efficiencies of processes including anaerobic methanogenic digestion and gas turbine (Chynoweth and Isaacson 1987). Together with the ability of this CE-MFC to generate electricity without the need of supplemental

facilities (e.g., a gas turbine when using methane), the results of the present study support the potential of CE-MFC systems for the treatment of organic wastewaters. However, more efficient methanogenic digesters continue to be developed (e.g., a digester with organic loading rates of more than $80 \text{ g l}^{-1} \text{ day}^{-1}$ [Tatara et al. 2005]), making it important that further advances in MFC are made to continue to make these systems competitive with modern anaerobic digesters.

The Coulombic efficiencies show that large portions (52 to 72%) of electrons released from the organic matter were not captured for current generation. Possible alternative electron sinks include methanogenesis (Ishii et al. 2008b), aerobic respiration, and biomass formation. With respect to methanogenesis, since 8 mol of electrons are needed for generating 1 mol of methane from carbon dioxide, the methane production rates (Table 1) measured here are equivalent to a current of 1 mA (at the loading rate of $2.9 \text{ g l}^{-1} \text{ day}^{-1}$) or 36 mA (at $5.8 \text{ g l}^{-1} \text{ day}^{-1}$). The possible electron sources and sinks, summarized in Table 3, indicating that methanogenesis did not largely contribute to loss of electrons. It is more likely that a large percentage of the electrons were lost to aerobic respiration sustained by oxygen diffusion through the air cathode (Cheng et al. 2006). In our reactor, oxygen was never detected in our gas analyses under stable operational conditions (data not shown), and the ORP was always highly negative and stable at approximately -400 mV . These two results suggest that any oxygen that leaked into the reactor through the air cathodes was immediately utilized by microbial respiration (probably at the surfaces of the air cathodes). Although oxygen respiration rates could not be measured here, based on data presented in a previous report (Cheng et al. 2006) we estimate oxygen transfer through the 4-PTFE air cathode as $1.4 \mu\text{mol h}^{-1} \text{ cm}^{-2}$. With 4 mol of the electrons needed for each mole of oxygen, and using the total air cathode area of $1,360 \text{ cm}^2$, the oxygen-dependent electron consumption rate for the CE-MFC is calculated to be equivalent to an electric current of 206 mA. This was approximately half of the electron source at the loading rate of $2.9 \text{ g l}^{-1} \text{ d}^{-1}$, revealing that the electricity generation and oxygen respiration evenly shared the electrons released from the organic matter at this loading rate. In case of the $5.8\text{-g l}^{-1} \text{ day}^{-1}$ loading rate, 30% of electrons are still unaccounted for by this analysis. Another possibility is that at this loading rate biofilm organisms actively grew and/or produced reduced intracellular organic materials (Freguia et al. 2007), which remained in the CE-MFC. Recently, it was shown that placing a cloth between the anode and cathode can greatly improve Coulombic efficiencies of laboratory MFCs with only a small drop of power density (Fan et al. 2007a). This technique may be useful for increasing Coulombic efficiencies in CE-MFCs in future tests.

Table 3 Electron balance of the CE-MFC reactor expressed as electric current

Electron source and sink	Value (mA) for	
	2.9 g l ⁻¹ day ⁻¹	5.8 g l ⁻¹ day ⁻¹
Source		
Organic oxidation ^a	385	755
Sink		
Electric output	183 (48) ^b	202 (28)
Methanogenesis	1 (0.2)	36 (5)
Others ^c	201 (52)	513 (67)

^a Estimated from an organic loading rate, an organic removal efficiency, and the COD–electron conversion ratio (see text)

^b Percentage to the total electron sink that is equal to an organic removal rate

^c Including biomass formation, instrumental loss, and use of other electron acceptors. Values were estimated by subtracting values for electric output and methanogenesis from the total electron sink

In conclusion, the present study shows that the CE system is useful for obtaining a scalable MFC with relatively high performance. The CE modules can be made to be detachable, and thus the performance of each module can be individually examined, allowing for the development an MFC reactor that is easy to maintain and control. We anticipate that the performance of CE-MFCs will be further improved by incorporating other advances made in MFC materials and designed into the cassette type architecture examined here.

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