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Graphite Fiber Brush Anodes for Increased Power Production in Air-Cathode Microbial Fuel Cells

BRUCE LOGAN,*,†,‡ SHAOAN CHENG,†,‡
VALERIE WATSON,† AND
GARETT ESTADT§
Department of Civil and Environmental Engineering,
The Penn State Hydrogen Energy (H2E) Center, and
Department of Chemical Engineering, Penn State University,
University Park, Pennsylvania 16802

To efficiently generate electricity using bacteria in microbial fuel cells (MFCs), highly conductive noncorrosive materials are needed that have a high specific surface area (surface area per volume) and an open structure to avoid biofouling. Graphite brush anodes, consisting of graphite fibers wound around a conductive, but noncorrosive metal core, were examined for power production in cube (C-MFC) and bottle (B-MFC) air-cathode MFCs. Power production in C-MFCs containing brush electrodes at 9600 m^2/m^3 reactor volume reached a maximum power density of 2400 mW/m^2 (normalized to the cathode projected surface area), or 73 W/m^3 based on liquid volume, with a maximum Coulombic efficiency (CE) of 60%. This power density, normalized by cathode projected area, is the highest value yet achieved by an air-cathode system. The increased power resulted from a reduction in internal resistance from 31 to 8 Ω. Brush electrodes (4200 m^2/m^3) were also tested in B-MFCs, consisting of a laboratory media bottle modified to have a single side arm with a cathode clamped to its end. B-MFCs inoculated with wastewater produced up to 1430 mW/m^2 (2.3 W/m^3, CE = 23%) with brush electrodes, versus 600 mW/m^2 with a plain carbon paper electrode. These findings show that brush anodes that have high surface areas and a porous structure can produce high power densities, and therefore have qualities that make them ideal for scaling up MFC systems.

Introduction

Microbial fuel cells (MFCs) are an emerging technology for producing electricity directly from biodegradable organic matter using bacteria (*1*−*3*). One of the most promising applications for MFCs is to use them as a method of simultaneously treating wastewater while accomplishing power generation (*4*−*6*). There remain several technical challenges to make this technology feasible for widespread application: power densities should be increased, the cost of construction materials needs to be reduced, and the architecture devised to produce power must be scaleable to allow treatment of large wastewater flows (*5*, *6*). The highest power density produced in an MFC of 4.3 W/m^2 was achieved by repeatedly feeding the reactor so that mediators produced by bacteria could accumulate in solution (the reactor fluid was not completely replaced) (*7*). Ferricyanide was used as a catholyte, but the use of this chemical is not sustainable and would therefore be impractical for large scale systems (*5*). For sustainable systems oxygen can be used as the electron acceptor at the cathode, but power densities with the cathode immersed in water have been low, ranging from maximum values of ∼45 mW/m^2 (*8*−*11*) using two-chambered systems to 177 mW/m^2 using sediment MFCs (*12*). Air-cathode MFCs, where the cathode is exposed to air on one side and water on the other, appear more practical due to lack of a need to aerate the water and the high power densities achieved in these systems (*5*).

A variety of anode materials have been used in MFCs, including plain graphite (*13*), carbon paper (*14*), carbon cloth, felt, or foam (*15*), reticulated vitreous carbon (RVC) (*16*, *17*), or graphite granules (*18*, *19*). There appears to be general trend of increased power production with higher surface area materials (*15*), but there have only been limited comparisons of power production under conditions that are identical except for the anode material. Increasing the surface area of the anode relative to that of the cathode can increase power when the system is not completely limited by a high internal resistance (*20*, *21*). Providing good access for bacteria and substrate is important for power generation. For example, moving a cloth anode away from the side of the reactor in one study increased power output, but power decreased when the electrode was again placed against that surface at the same distance from the cathode (*22*). Bonding materials to the anode that facilitate electron transfer and improve the adhesion of bacteria can increase power. For example, power was increased by bonding electron mediators such as neutral red (*23*), anthraquinone-1,6-dissulfonic acid (AQDS), or 1,4-naphthoquinone (NQ) to a graphite anode (*12*), or by adding metals such as Mn^{4+} (*23*), Fe_3O_4, or Fe_3O_4 and Ni^{2+} (*12*), but the final power densities were still low compared to more recent findings. The largest power density so far achieved using an air-cathode MFC of 1970 mW/m^2 was produced by ammonia-gas treatment of carbon cloth at elevated temperatures (*24*).

The use of materials used in the laboratory, such as carbon cloth or graphite granules, presents substantial challenges for scale up. Carbon cloth (relative to granules) is expensive and would be difficult to use in the same configurations in large systems as those tested in some laboratory systems. Granules are heavy and could clog due to relatively low porosities. Biofilm reactors used for wastewater treatment require high structural strength to support the biofilm when used in structured, open-flow conditions such as trickling filters. In saturated flow systems, biofilm carriers such as sponges or blocks can also be used, but clogging of the material can be a problem and these types of materials would be impractical as supports in MFCs as they would not be in constant contact, as needed for electron transfer. We therefore examined a new type of anode architecture based on bundling high-surface-area graphite fibers into a central core, forming a highly conductive brush electrode having a high specific surface area (*25*). This brush architecture should be capable of being scaled up and used in larger-size reactors.

Materials and Methods

Electrode Materials. Brush anodes were made of carbon fibers (PANEX33 160K, ZOLTEK) cut to a set length and wound using an industrial brush manufacturing system into a twisted core consisting of two titanium wires. Two brush sizes were

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* Corresponding author phone: 814-863-7908; fax: 814-863-7304; e-mail: blogan@psu.edu.
† Department of Civil and Environmental Engineering.
‡ The Penn State Hydrogen Energy (H2E) Center.
§ Department of Chemical Engineering.

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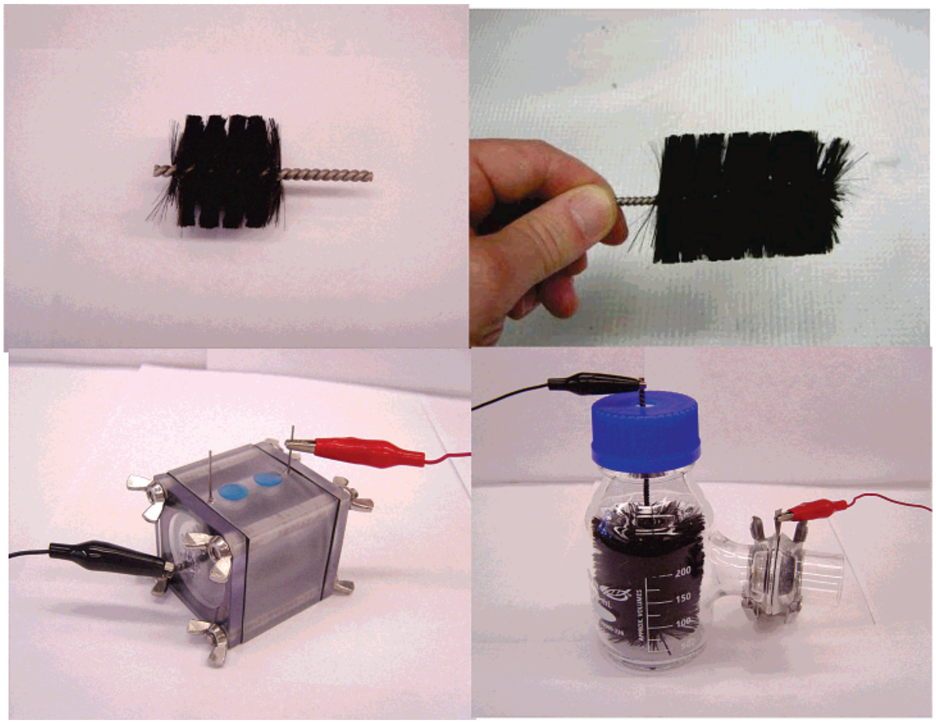

FIGURE 1. Graphite fiber brush anode electrode used in (A) C-MFC and (B) B-MFCs, and photographs of the reactors containing the brush electrodes: (C) C-MFC shown with the brush anode, and (D) B-MFC with brush anode and side port cathode.

used in this study: a small brush 2.5 cm in outer diameter and 2.5 cm long (Figure 1A); and a larger brush 5 cm in diameter and 7 cm long (Figure 1B). Based on mass of fibers used in a single brush, and an average fiber diameter of 7.2 μm, we estimated a surface area of 0.22 m^2 or 18 200 m^2/m^3-brush volume for the small brush (95% porosity), and 1.06 m^2 or 7170 m^2/m^3-brush volume for the larger brush (98% porosity). Except as noted, brush anodes were treated using ammonia gas as previously described (*24*). In some tests plain Toray carbon paper anodes (untreated and non-wet-proofed, E-TEK, having a projected area of 23 cm^2, both sides) were used for comparisons to brush anodes.

Random bundles of ammonia-treated graphite fibers were also used in some tests, consisting of one to four tows of fibers with each cut to a fixed length of 10 cm. The mass of each tow was ~0.1 g, with a projected surface area calculated as 0.020 m^2 per tow for 10 μm diameter fibers (Granoc-Nippon) and 0.035 m^2 per tow for the 6 μm diameter (#292 Carbon Fiber Tow, Fibre Glast, Ohio).

The cathodes were made by applying platinum (0.5 mg/cm^2 Pt) and four diffusion layers on a 30 wt % wet-proofed carbon cloth (type B-1B, E-TEK) as previously described (*26*). In some experiments, the cathodes were prepared using the same method (*26*) but contained 40% cobalt tetramethylphenylporphyrin (CoTMPP, 1.2 mg/cm^2) as the catalyst instead of Pt (*27*).

MFC Reactors. Two types of single-chambered MFCs were used to examine power production using brush electrodes: cube-shaped MFCs (C-MFCs) which are designed to maximize power production; and bottle-type MFCs containing a single side port (B-MFC) that were created for examining power production by pure and mixed cultures in an easily produced and inexpensive system. C-MFCs were constructed as previously described (*28*) except the anode that normally rested against the closed end of the reactor was replaced by a small brush electrode positioned in a concentric manner in the core of the cylindrical anode chamber (Figure 1C). The brush end was fixed in the chamber (4 cm long by 3 cm in diameter; liquid volume of 26 mL) so that the end was 1 cm from the cathode (3.8 cm diameter, 7 cm^2 total exposed surface area). The metal end of the brush protruded through a hole drilled in the reactor that was sealed with epoxy (Quick Set Epoxy, LOCTITE). CoTMPP was used as the catalyst in all C-MFC tests.

B-MFCs were made from common laboratory media bottles (320 mL capacity, Corning Inc., NY), and were autoclavable even if fully assembled (Figure 1D). A large brush electrode was suspended in the middle of the bottle containing 300 mL of medium, with the top of the brush ~6 cm from the bottle lid. The wire from the bush was placed through the lid hole and sealed with epoxy. In tests using carbon paper anodes (2.5 cm by 4.5 cm, 22.5 cm^2 total), the electrodes were placed ~6 cm from the bottle lid and connected to a titanium (99.8% pure) wire through a hole in the lid that was sealed with epoxy. The 4-cm long side tube was set 5 cm from the reactor bottom, with a 3.8 cm-diameter cathode held in place at the end by a clamp between the tube and a separate single tube 4 cm long, providing a total projected cathode surface area of 4.9 cm^2 (one side of the cathode). In tests using random bundles of fibers as the anode, the fibers were held by a pinch clamp connected to a wire that was passed through a hole in the lid and sealed with epoxy.

Reactor Inoculation. C-MFCs were inoculated using pre-acclimated bacteria from another MFC (originally inoculated with primary clarifier overflow) that had been running in fed batch mode for over 6 months. The reactor was fed a medium containing 1 g/L of acetate in 50 mM phosphate buffer solution (PBS; Na_2HPO_4, 4.09 g/L and $NaH_2PO_4 \cdot H_2O$, 2.93 g/L) or 200 mM PBS, NH_4Cl (0.31 g/L), and KCl (0.13 g/L), and metal salt (12.5 mL/L) and vitamin (5 mL) solutions (*29*). Feed solutions were replaced when the voltage dropped below 20 mV, forming one complete cycle of operation. C-MFCs were operated in a temperature-controlled room at 30 °C.

B-MFCs were inoculated using fresh primary clarifier overflow (unless stated otherwise) in a 1 g/L glucose medium prepared as described above with 50 or 200 mM PBS. In one separate set of tests the reactor was inoculated with the same

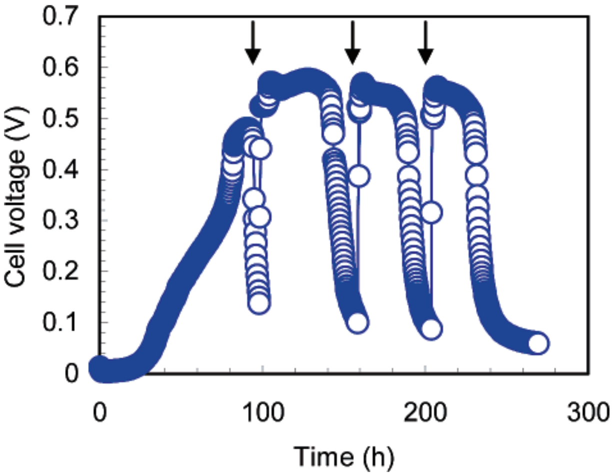

FIGURE 2. Initial four cycles of power production in a C-MFC with a brush anode (50 mM PBS, 1000 Ω resistor; arrows indicate when reactor was fed fresh medium).

pre-acclimated bacterial solution used to inoculate the C-MFCs. All B-MFCs were operated on laboratory bench tops at ambient temperatures of 23 ± 3°C.

Analyses. The voltage (V) across an external resistor (1000 Ω except as noted) in the MFC circuit was monitored at 30 min intervals using a multimeter (Keithley Instruments, OH) connected to a personal computer. Current (I), power ($P = IV$), and Coulombic efficiency (CE) were calculated as previously described (*9*), with the power density normalized by the projected surface area of one side of the cathode, and volumetric power density normalized by the volume of the liquid media. Internal resistance, R_{int}, was measured using electrochemical impedance spectroscopy (EIS) with a potentiostat (PC 4/750, Gamry Instrument Inc., PA), with the anode chamber filled with PBS and substrate. Impedance measurements were conducted at the open circuit voltage (OCV) over a frequency range of 10^5 to 0.005 Hz with sinusoidal perturbation of 10 mV amplitude (*22*). Polarization curves were obtained by measuring the stable voltage generated at various external resistances and then used to evaluate the maximum power density (*5*). The C-MFCs were run for at least two complete operation cycles at each external resistance, where each cycle takes ~2 days. The B-MFCs require much longer cycle times (~21 days), and therefore polarization data were taken after 15 min at each external resistance at the beginning of a single operation cycle. The internal resistance, defined as the sum of all ohmic resistances including electrolyte and contact resistances, for both C- and B-MFCs was determined using Nyquist plots of the impedance spectra from the real impedance Z_{re} where it intersects the X-axis (imaginary impedance $Z_{im} = 0$) (*18, 31−32*).

Results

Power Production using C-MFCs. Voltage generation cycles of C-MFCs with brush anodes were reproducible after 4 feeding cycles with fresh media, producing a maximum voltage of 0.57 V and a CE = 41% with the 1000 Ω resistor (Figure 2). Based on polarization data, the maximum power produced was 2400 mW/m^2 at a current density of 0.82 mA/cm^2 ($R_{ext} = 50$ Ω), or 73 W/m^3 when power was normalized by the reactor liquid volume (Figure 3). CEs ranged from 40 to 60% depending on the current density (Figure 3). The internal resistance was $R_{int} = 8$ Ω for the brush C-MFC (200 mM PBS), versus $R_{int} = 31$ Ω for a carbon cloth C-MFC (200 mM PBS, 4 cm electrode spacing) (Figure 4 and Table 1).

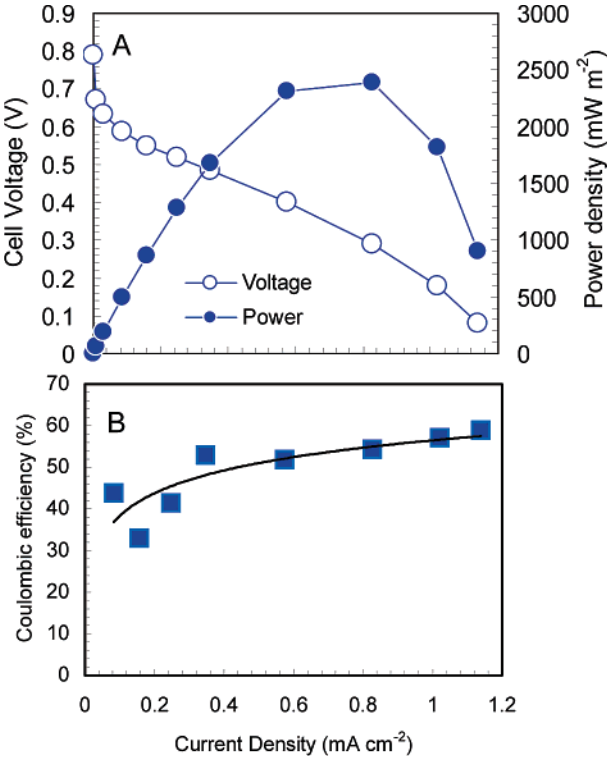

FIGURE 3. (A) Power density and cell potentials, and (B) Coulombic efficiency for C-MFC with brush anode.

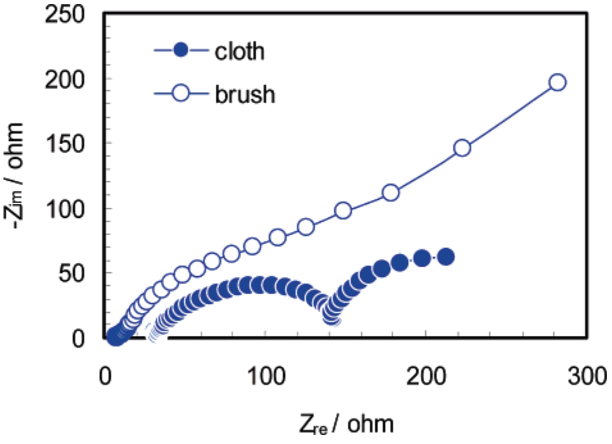

FIGURE 4. Nyquist plots corresponding to the impedance spectra of the C-MFCs measured between the cathode and anode (two-electrode mode) in 200 mM PBS (the MFC was discharged to 0.57 V at 1000 Ω and the external circuit had been disconnected for 2 h). The internal resistance is obtained as the value of the *x*-intercept.

Power Production using B-MFCs. Brush electrodes used in B-MFCs produced up to 1430 mW/m^2 (2.3 W/m^3), compared to 600 mW/m^2 (0.98 W/m^3) using carbon paper electrodes in a 200 mM PBS solution (Figure 5). Using a lower ionic strength solution reduced power production to 570 mW/m^2 (0.93 W/m^3) with a brush anode, and 300 mW/m^2 (0.50 W/m^3) with a carbon paper anode. This effect of solution conductivity is consistent with previous studies showing that power increases with ionic strength (up to the tolerance of the bacteria) due to a reduction in ohmic resistance (*5, 32*). The internal resistance of the brush B-MFC was 50 Ω, with values for the other reactor conditions summarized in Table 1. Cotton plugs are often used in shake flask experiments to keep bacterial suspensions aerobic but sterile (i.e., the plug allows oxygen transfer but filters out bacteria). Placing cotton plugs into the side tube did not affect power production, a situation which would be helpful for conducting experiments

TABLE 1. Power Production and Internal Resistances of MFCs Containing Various Components (200 mM PBS)

reactor type	anode	internal resistance (Ω)	maximum power	
			(mW/m²)	(W/m³)
C-MFC	small brush	8	2400	73
C-MFC[a]	carbon cloth	31	1070	29
B-MFC	large brush	50	1200	2.0
B-MFC	large brush[b]	49	1430	2.3
B-MFC	large brush, untreated	58	750	1.2
B-MFC	carbon paper	65	600	0.98

[a] 4 cm electrode spacing. [b] Using an inoculum from a previously acclimated MFC.

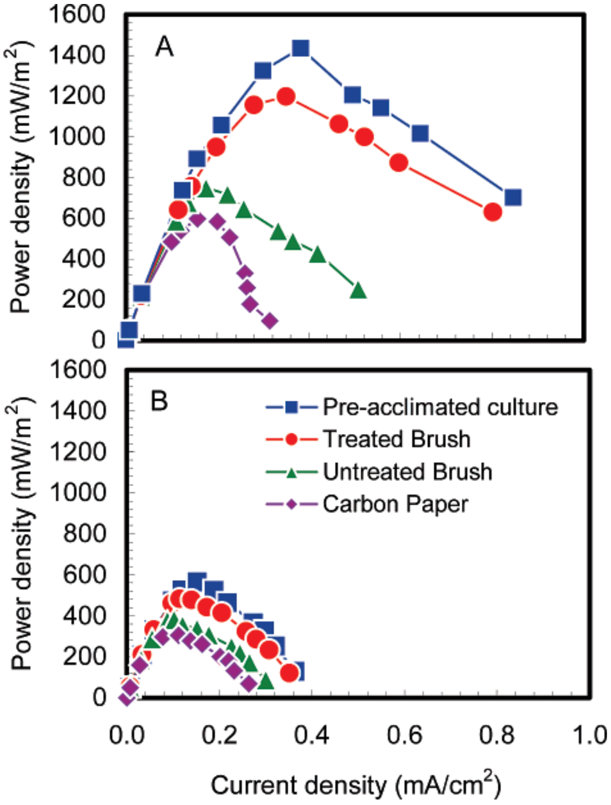

FIGURE 5. Power density curve for B-MFCs in (A) 200 mM PBS, and (B) 50 mM PBS.

with pure cultures as this could help reduce the potential for bacterial contamination through the cathode.

To confirm that treatment of the brush electrodes with ammonia gas was an effective method of reducing the acclimation time and increasing power, additional tests were conducted using untreated brush anodes. Power production reached a maximum of 750 mW/m² with the untreated anode, which is 37% less than that obtained with ammonia treatment (Figure 5). Peak power production for the first cycle took 330 h, compared to 136 h with the treated electrodes, consistent with previous findings that the ammonia treatment reduces acclimation time (*24*). Power production with the brush electrodes was also substantially higher than that produced with an untreated carbon paper electrode, which produced a maximum of 600 mW/m².

Power Production using Random Fibers. The use of random or unstructured graphite fibers was also examined using B-MFC reactors. The maximum power production was 1100 mW/m² (0.11 g of 6 μm diameter fibers) (Figure 6). There did not seem to be any consistent trend in power generation with brush surface area or loading. In tests with the 10 μm diameter fiber, power ranged from 690 to 850 mW/m² for mass loadings of 0.09 to 0.35 g. Power production using the 6 μm diameter fibers ranged from 770 to 1100 mW/m² (Figure 6). It is apparent that the two graphite materials differed in power production, but it is not known whether that was a result of surface chemistry or fiber size. The lack of a consistent effect of brush mass on power suggests that the random orientation was an important factor in power production. The fibers were not well dispersed in the reactor and tended to clump together, and this may have hindered bacterial access to the fiber surfaces as well as the diffusion of substrate into the brush interior. Thus, it appears from this preliminary analysis with random fibers that the use of fibers in structured (brush) anodes will be more useful for power generation in MFCs. Further testing with structured materials of various sizes, lengths, and set orientations will help to establish if there is an optimal loading or brush architecture that can minimize the mass of fibers needed to maximize power.

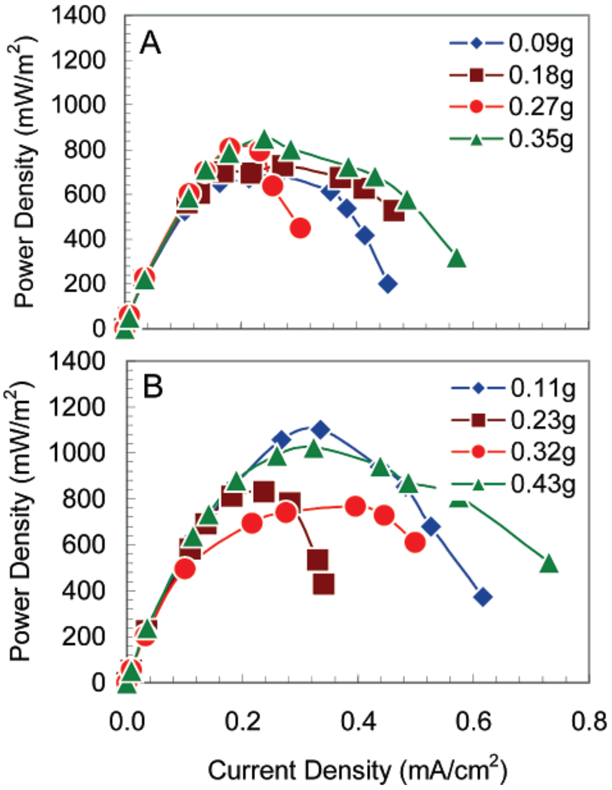

FIGURE 6. Power density curves using varied loadings of randomly distributed (A) 10 μm and (B) 6 μm graphite fibers as the anode material.

Discussion

The high-surface-area brush electrode treated with ammonia gas produced a maximum power density of 2400 mW/m². To our knowledge, this is the highest power density achieved for power normalized to the projected surface area of an electrode (cathode). We previously achieved 1970 mW/m² using a smaller cube-type reactor (14 mL total volume) that had a smaller electrode spacing (2 cm) than that used here (4 cm) (*24*). However, the smaller volume in that study (half of that used here) did result in a higher volumetric power density (115 W/m³ versus 73 W/m³ achieved here).

While other high specific surface area materials have been used as the anode, such as carbon foams (*15*), such materials could become filled with bacteria and difficult to unclog. Brush electrodes have been previously investigated as cathodes immersed in seawater as a part of chemical fuel cells for sea vessels (*33*). Power production was not adversely affected by biofilm growth on the surface, and system performance actually improved over time with biofilm development (*34*).

The small size of the individual graphite fibers (7.2 μm) and their low resistance (0.00155 Ω cm, reported by the manufacturer), coupled with a good dispersed distribution of the filaments in brushes, make them ideal as anodes for small or large scale MFC applications. Brushes for larger applications can be made in any size or packing density, but additional work will be needed to optimize brush architecture (such as fiber density, length, and winds per length) to maximize power generation while minimizing the mass of material used. The specific surface areas using the brush electrodes were 18 200 (C-MFC), and 7170 m^2/m^3 (B-MFC), and with the reactor volumes used here produced 9600 (C-MFC), and 4200 m^2/m^3 (B-MFC) normalized to the liquid volume. We do not know the minimum specific surface area needed to maximize power, but surface areas used here are substantially greater than 100 m^2/m^3 typically used in fixed film processes such as trickling filters.

There are several reasons why graphite brush electrodes should permit the use of much higher specific surface areas in MFCs than those typically used with other types of biofilm reactors. Aerobic reactors such as trickling filters are designed to have specific surface areas on the order of 100 m^2/m^3 to maintain air flow through the reactor, and to have an open structure that avoids biofouling by sloughed biofilm. However, oxygen is not directly needed for MFC anode biofilms, and thus there is no need for air flow through the brushes. Biofouling issues are substantially different for exoelectrogenic biofilms than for aerobic biofilms as the bacteria in the MFC at the bottom (the electrode surface) are metabolically quite active. In contrast, bacteria in aerobic biofilms are active at the top and die off at the bottom of the biofilm, causing massive sloughing events that can clog the reactor. It appears from our own operational data that MFCs may be less susceptible to such sloughing events, clogging, or fouling as carbon cloth and paper electrode MFCs have operated continuously for years without apparent fouling affecting power generation. However, larger scale systems and actual wastewaters will need to be tested to fully ascertain the stability of the brush electrode systems with respect to clogging.

The microbial ecology of exoelectrogenic bacteria is an important subject that is only just beginning to be examined (*35*). To study these exoelectrogenic bacteria we need to be able to grow them as pure cultures in high-performance systems. The B-MFC tests are well suited for such comparisons as these reactors are relatively easy to construct out of readily available materials (standard media bottles and side arms), all materials are autoclavable, and the system has a relatively low internal resistance compared to two-bottle systems. Power production with the single-bottle system was not affected when the cathode tube was fitted with a cotton plug, making it possible to minimize the potential for contamination through the cathode. These characteristics of single-bottle systems should help researchers obtain more useful data with pure cultures. For example, in a previous test power generation was compared with pure and mixed cultures but there was no difference in maximum power for the two inocula likely as a result of the high internal resistance of the system (*36*). However, in a different system with a low internal resistance, pure cultures produced substantially less power than the original mixed culture (*7*). Additional work is ongoing in our laboratory to compare power production of mixed and pure cultures under identical conditions using these reactors.

The use of graphite brush electrodes in bottle and cube reactors should allow comparisons of not only pure and mixed culture inocula, but also of the effects of various substrates and solution chemistries under conditions where the surface for bacterial growth is provided in excess. For large-scale reactor design, it will be important to optimize specific surface areas needed for bacterial growth to maximize power output but minimize the amount of graphite materials used, especially in relation to available cathode surface area. A method for providing high specific surface areas for cathodes is described in a companion paper in this issue (*37*). Linking these brush anode and tube cathode systems will provide a method for testing reactor performance at different scales.

Acknowledgments

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