Enhanced current and power density of micro-scale microbial fuel cells with ultramicroelectrode anodes

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Abstract

We present a micro-scale microbial fuel cell (MFC) with an ultramicroelectrode (UME) anode, with the aim of creating a miniaturized high-current/power-density converter using carbon-neutral and renewable energy sources. Micro-scale MFCs have been studied for more than a decade, yet their current and power densities are still an order of magnitude lower than those of their macro-scale counterparts. In order to enhance the current/power densities, we engineer a concentric ring-shaped UME, with a width of 20 μm, to facilitate the diffusion of ions in the vicinity of the micro-organisms that form biofilm on the UME. The biofilm extends approximately 15 μm from the edge of the UME, suggesting the effective biofilm area increases. Measured current/power densities per the effective area and the original anode area are 7.08 ± 0.01 A m⁻² & 3.09 ± 0.04 W m⁻² and 17.7 ± 0.03 A m⁻² & 7.72 ± 0.09 W m⁻², respectively. This is substantially higher than any prior work in micro-scale MFCs, and very close, or even higher, to that of macro-scale MFCs. A Coulombic efficiency, a measure of how efficiently an MFC harvests electrons from donor substrate, of 70%, and an energy conversion efficiency of 17% are marked, highlighting the micro-scale MFC as an attractive alternative within the existing energy conversion portfolio.

Keywords: microbial fuel cell (MFC), ultramicroelectrode (UME), micro-scale, microfabrication, energy harvesting, carbon-neutral

Online supplementary data available from stacks.iop.org/JMM/26/095016/mmedia

(Some figures may appear in colour only in the online journal)

1. Introduction

Fossil energy is projected to deplete in the next one to two centuries, motivating the exploration of renewable energy sources as alternatives. Bioenergy is an attractive candidate due to the abundant biomass on earth, its carbon-neutrality, and renewability. Microbial fuel cells (MFCs), which convert biomass directly to electricity, are electrochemical fuel cells harvesting electrons from organic substances, i.e. acetate, by the catalytic reaction of specific bacteria species, named anode-respiring bacteria (ARB) or electroactive bacteria, via their unique extracellular electron transport (EET). The direct electrochemical conversion of MFCs results in high energy conversion efficiency. Miniaturized MFCs have the features of a small footprint, and are light weight, and potentially low cost, making them suitable for applications such as passive radio frequency (RF) tags, ultra-low power wireless sensor networks, implantable medical devices, and scientific research on electroactive bacteria [1–6].

Despite the steady success of miniaturized MFCs over several years, the current and power densities of micro-scale MFCs reported to date, are magnitudes lower than commercially available power sources such as lithium ion batteries (~100 W m⁻²) [7]. Many approaches have been attempted to
enhance the current/power density of micro-scale MFCs since the first report of a micro-scale MFC by Chiao et al [8]. Chiao et al later used microfluidic channels to increase the surface area to volume ratio (SAV) [9], and Siu and Chiao implemented micropillars to further increase SAV [10]. Inoue [11] and Mink et al [12] presented high SAV anodes using carbon nanotubes; Jiang et al implemented conductive nanofiber as 3D anode to reduce the start-up time and improve the performance [13]; Qian et al [14] and Choi et al [15] shortened the distance between anode and cathode to reduce the internal resistance, thereby enhancing the power density. We have previously presented micro-scale MFCs with high current and power densities up to 2.5 A m$^{-2}$ and 0.83 W m$^{-2}$ [7, 16, 17], respectively, the highest reported for micro-scale MFC. However, they are still approximately one order of magnitude lower than those of macro-sized counterparts (30 A m$^{-2}$ and 6.8 W m$^{-2}$) [18].

An ultramicroelectrode (UME) is defined as an electrode which has at least one dimension (such as the width of band or radius of disk) smaller than 25 μm [19]. As the dimension of an UME approaches the diffusion layer thickness, which describes the region at the vicinity of an electrode where the concentration of organic substrate and buffer is different from that in the bulk, the diffusion of the substrate and buffer greatly enhances. UMEs have been used in many applications, including chemical sensing, material synthesis, and energy conversion, etc, where the performance significantly improves as the diffusion rate substantially enhances [20, 21]. Recently, Liu et al implemented gold line UMEs (with a width of 10 μm) in electroactive biofilm, and achieved a high current density of 16 A m$^{-2}$ [22]. Similarly, Pocaznoi et al used a fine, platinum wire, 25 μm in diameter, as an UME in electroactive biofilm, enhanced...
the diffusion of acetate, and thereby remarkably improved the current density up to 66 A m$^{-2}$ [23].

The UME effect can be effectively adopted in a micro-scale MFC through a microfabrication technique, as the geometries of the MFC can be precisely controlled in accordance with the scale of diffusion layer thickness. This work aims to adopt the UME effect in a micro-scale MFC using micro-fabrication techniques to substantially improve the current/power density of micro-scale MFCs. In the next section, materials and methods are presented, including the design, fabrication

Figure 3. Polarization curves for the MFCs: voltage and power density versus current density (a) and (b) of MFC with 100 μm UME, (c) and (d) of MFC with 220 μm UME, and (e) and (f) of MFC with plain gold disk anode (control).
and operation of the UME anodes, micro-scale MFCs, and as data analysis. Subsequently, experimental results are demonstrated, including the polarization curves, areal resistivity, and biofilm/efficiency characterization. Following a discussion of the micro-scale MFCs along with other types of energy converters, a conclusion is drawn.

2. Materials and methods

2.1. Micro-scale MFC fabrication and assembly

2.1.1. Ultramicroelectrode (UME) as an anode [24]. Figure 1 shows a concentric ring-shape UME. The UME serves as the anode of a micro-scale MFC. The UME on a glass slide has a ring width of 20 μm. In order to explore the UME effect, we adopted two UMEs, having this ring width of 20 μm, in which the gaps between the ring electrodes are 100 μm (figure 1(d)) and 220 μm (figure 1(e)), respectively, and a plain disk of 5040 μm in radius is implemented as a control.

Diffusion layer thickness (δ) can be described by:

\[
δ = \frac{D}{k_d} = \frac{nFDc}{l_i} = \frac{nFDc}{l_{i,\text{areal}}} \tag{1}
\]

where \(D\) is the diffusion coefficient (0.88 × 10^{-9} m² s⁻¹ for acetate), \(k_d\) is the heterogeneous diffusion rate constant, \(F\) is the Faraday constant (96 500 Cmol⁻¹), \(c\) is the concentration of substrate (acetate in this work), \(A\) is the geometric area of the electrode, \(n\) is the number of electrons in redox reaction, \(l_i\) is the limiting current, and \(l_{i,\text{areal}}\) is the areal limiting current density. When \(n\) is 8 and \(c\) is 25 mol m⁻³, areal limiting current density is computed at 165 A m⁻², projected based upon the prior design of using UME anodes, 66 A m⁻² at acetate concentration of 10 mM. Assuming the limiting current density is linearly proportional to the substrate concentration [19], we chose 165 A m⁻² of 25 mM acetate concentration, which gives \(δ\) of ~100 μm. The two UME gaps are chosen to be equal or larger than the diffusion layer thickness, to facilitate the mass transfer by the UME effect. The two UMEs and a disk-shaped plain gold disk anode are used to explore the UME effect.

The fabrication of micro-scale MFC which have UMEs starts with inlet/outlet formation. First, six through holes were drilled on a glass slide (46 mm × 26 mm × 1 mm, VWR International Inc.): one as the inlet, one as the outlet and the remaining four as screw holes. Then the glass slides were cleaned with piranha solution (concentrated H₂SO₄ and 30% H₂O₂, 3:1 volume ratio). Subsequently a layer of Cr/Au was deposited using a magnetron sputter (0.7 Pa, 150 mA for Cr and 45 mA for Au). A layer of Cr/Au (20/200 nm) was deposited using a magnetron sputter (0.7 Pa, 150 mA for Cr and 45 mA for Au).

2.1.2. Cathode. The fabrication of cathodes also started with inlet/outlet formation. First, six through holes were drilled on a glass slide (46 mm × 26 mm × 1 mm, VWR): one as the inlet, one as the outlet and the remaining four as screw holes. Then the glass slides were cleaned with piranha solution (concentrated H₂SO₄ and 30% H₂O₂, 3:1 volume ratio). Afterwards, a layer of Cr/Au (20/200 nm) was deposited using a magnetron sputter (0.7 Pa, 150 mA for Cr and 45 mA for Au).

2.1.3. Assembly. The anodes, cathodes, proton exchange membrane (PEM, Nafion 117), silicone gaskets (250 μm thick, Fuel Store Inc.), nanoports (10–32 Coned assembly, IDEX Health & Science), bolts and nuts were assembled to build the micro-scale MFCs, as shown in figure 2. First, nanoports were glued to the inlet and outlet of anode and cathode glass slides by instant epoxy glue (Loctite). Second, two rectangular silicone gaskets with dimensions of 46 mm × 26 mm × 0.25 mm were patterned with a hole of 1.414 cm in diameter at the center, in order to define the anode and cathode chamber volume of 39.25 μl. Third, a rectangular PEM with a dimension of 46 mm × 26 mm was cut. Finally, the anode, cathode, conductive copper tape as interconnect, PEM and silicone gaskets were assembled, as illustrated in figure 2(a). Figure 2(b) shows the fabricated micro-scale MFC with a US quarter for size comparison.

2.2. Inoculum and electrolyte

The inoculum for the micro-scale MFC was obtained from an acetate-fed microbial electrolysis cell (MEC) that had a Geobacter-enriched bacterial community originally from anaerobic-digestion sludge [25]. The anolyte is 25 mM acetate, 82.6 mm², respectively. Three micro-scale MFCs are started for each type of anode.
0.1 mg Na$_2$WO$_4$ · 2H$_2$O, 0.2 mg NiCl$_2$ · 6H$_2$O, and 1 mg FeSO$_4$ · 7H$_2$O (per liter of deionized water) (pH 7.8 ± 0.2). For the start-up process, inoculum and anolyte are mixed with 1:1 volume ratio as the anolyte. The catholyte is 50 mM potassium ferricyanide in 100 mM PBS (pH 7.4). The anolyte and catholyte were supplied to the MFC through a syringe pump (PHD 2000, Harvard Apparatus).

### 2.3. Operation conditions

The voltage profiles of the MFCs were recorded using a data acquisition system (DAQ/68, National Instrument). Voltage drop across an external load resistor connected between the anode and cathode was recorded every minute using a Labview Interface (National Instrument). The MFCs run on continuous mode unless specifically mentioned. During the start-up process, the MFCs were operated at a flow rate of 0.5 μl min$^{-1}$ and at an external load resistor of 148 Ω. Once the start-up process was completed, flow rates were increased to 2 μl min$^{-1}$, 4 μl min$^{-1}$, 6 μl min$^{-1}$, and 8 μl min$^{-1}$. Upon changing the flow rate, the MFCs were left to reach a quasi-steady state, which usually occurs within approximately 12 h.

Then polarization curves were obtained by recording voltage across a series of resistors connected between the anode and cathode, ranging from 148 Ω to 1 MΩ. For each resistor, we waited for a period of 10–15 min until the voltage was stable. Coulombic efficiency (CE) and energy conversion efficiency measurements were performed by stopping the anolyte supply while maintaining the catholyte supply. By limiting the
2.4. Data analysis

The current through the external load resistor was calculated via Ohm’s law: \( I = \frac{V}{R} \), where \( V \) is the voltage across the resistor, measured by the data acquisition system. The MFC output power was calculated via Joule’s law: \( P = I^2 \cdot R \).

The current and power densities reported in this paper were all normalized on the unit of anode surface area and anode volume, and the corresponding areal and volumetric current/power density were calculated by \( I_{\text{areal}} = I/\text{A}, \quad P_{\text{areal}} = P/\text{A} \) and \( I_{\text{volumetric}} = I/V, \quad P_{\text{volumetric}} = P/V \), where \( A \) and \( V \) are the anode surface area and anode chamber volume, respectively. Polarization curves were obtained by measuring the voltage across a series of external loads, plotting current densities versus voltage and power densities. The highest areal and volumetric current/power densities were determined by locating the maximum current/power density in the polarization curves. Internal resistance of the MFCs was obtained by linearly fitting the linear region of the polarization curve, and areal resistivity was calculated by normalizing the internal resistance of each MFC to the anode area.

The CE is the ratio of total coulombs harvested by MFCs from breaking down organic substrates (in our case, acetate) to the maximum possible coulombs if all the organic substrates are consumed to produce current: \( CE = \frac{C_P}{C_T} \times 100\% \), where \( C_P \) is the total coulombs calculated by integrating the current over the time and \( C_T \) is the maximum available coulombs of the substrates, \( C_T = V \times b \times A \times e \times c \). \( V \) is the volume of anode chamber (m\(^3\)), \( b \) is the number of moles of electrons produced by oxidation of one mole of the substrate (\( b = 8 \text{ mol e}^- \text{ mol}^{-1} \text{ acetate} \)), \( A \) is Avogadro’s number \( (6.023 \times 10^{23} \text{ molecules mol}^{-1}) \), \( e \) is the electron charge \( (1.6 \times 10^{-19} \text{ C/ electron}) \), and \( c \) is the concentration of biomass in the anode chamber \( (c = 25 \text{ mol m}^{-3}, \text{ sodium acetate}) \).

Energy conversion efficiency is the ratio of the total energy harvested by an MFC to the maximum possible energy that the biomass can produce (standard molar enthalpy (heat) of biomass): \( \eta = \frac{E_P E_T}{\Delta H^*} \times 100\% \), where \( E_P \) is the energy harvested by MFC, calculated by integrating output power over the time and \( E_T \) is the maximum possible energy of the biomass \( E_T = V \times c \times \Delta H^* \). \( V \) is the volume of anode chamber (m\(^3\)), \( c \) is the concentration of biomass in the anode chamber \( (c = 25 \text{ mol m}^{-3}, \text{ sodium acetate}) \), and \( \Delta H^* \) is the standard molar enthalpy (heat) of formation \( (872.711 \text{ KJ mol}^{-1} \text{ for acetate}) \) [26].

2.5. SEM imaging of biofilm

The MFCs were disassembled, rinsed in PBS, and biofilm on the UME anodes were fixed in 2% glutaraldehyde solution overnight at 4 °C (Glutaraldehyde solution, Grade I, 25% in H\(_2\)O, Sigma Aldrich). Samples were then dehydrated by serial, 10 min transfers through 50%, 70%, 90%, and 100% ethanol. Samples were examined using a field emission scanning electron microscopy (FESEM) (Hitachi S-4700-II). Voltage of the electron gun was set to be 15 kV.

3. Results

3.1. Polarization curves

All MFCs completed their start-up process in 3.5 d, and the current density during the start-up process is shown in figure S1 (stacks.iop.org/JMM/26/095016/mmedia). Then, flow rates were increased and polarization curves were obtained, as illustrated in figure 3. As flow rate increases, the current and power densities increase for all three types of anodes, which is due to the improved mass transfer of acetate and H\(^+\) carrying buffer into the biofilm, and is in accordance with previous studies [16]. At a higher flow rate (i.e. 8 \( \mu \text{L min}^{-1} \)), the current and power density decreases; we believe this might be due to the detachment of biofilm at a high flow rate. The plain gold disk MFC for the control group demonstrates a maximum current and power density of 17.7 \( \pm \) 0.03 A m\(^{-2}\) and 7.72 \( \pm \) 0.09 W m\(^{-2}\). The MFC with a 100 \( \mu \text{m} \) gap UME anode marked a highest current and power density of 19.0 \( \pm \) 0.18 A m\(^{-2}\) and 1.98 \( \pm \) 0.05 W m\(^{-2}\). The MFC with a 100 \( \mu \text{m} \) gap UME anode marked a highest current and power density of 19.0 \( \pm \) 0.18 A m\(^{-2}\) and 1.98 \( \pm \) 0.05 W m\(^{-2}\). The MFC with a 100 \( \mu \text{m} \) gap UME anode marked a highest current and power density of 19.0 \( \pm \) 0.18 A m\(^{-2}\) and 1.98 \( \pm \) 0.05 W m\(^{-2}\). The MFC with a 100 \( \mu \text{m} \) gap UME anode marked a highest current and power density of 19.0 \( \pm \) 0.18 A m\(^{-2}\) and 1.98 \( \pm \) 0.05 W m\(^{-2}\). The MFC with a 100 \( \mu \text{m} \) gap UME anode marked a highest current and power density of 19.0 \( \pm \) 0.18 A m\(^{-2}\) and 1.98 \( \pm \) 0.05 W m\(^{-2}\). The MFC with a 100 \( \mu \text{m} \) gap UME anode marked a highest current and power density of 19.0 \( \pm \) 0.18 A m\(^{-2}\) and 1.98 \( \pm \) 0.05 W m\(^{-2}\). The MFC with a 100 \( \mu \text{m} \) gap UME anode marked a highest current and power density of 19.0 \( \pm \) 0.18 A m\(^{-2}\) and 1.98 \( \pm \) 0.05 W m\(^{-2}\).
220 \( \mu \)m gap UME marked a highest current and power density of 15.03 \( \pm \) 0.09 A m\(^{-2}\) and 6.75 \( \pm \) 0.13 W m\(^{-2}\), which is 2.8-fold and 3.4-fold, respectively of the corresponding performance of the plain gold disk MFC. The performance enhancement between the two UMEs are similar. This enhancement is primarily due to the UME anodes, allowing a higher mass transfer of acetate and H\(^{+}\) carrying buffer into the biofilm, which is in agreement with previous studies [22, 23]. We also compared quasi-voltammetry curve for the micro-MFCs, as shown in figure S2 (stacks.iop.org/JMM/26/095016/mmedia). Both UME anodes mark higher current density. At a higher flow rate (i.e. 8 \( \mu \)l min\(^{-1}\)), areal resistivity values of 165 \( \pm \) 8 \( \Omega \) cm\(^{2}\) and 171 \( \pm \) 12 \( \Omega \) cm\(^{2}\) were at least one order of magnitudes lower than that of prior studies of micro-scale MFCs.

### Table 2. Summary table of this work in comparison with prior designs.

<table>
<thead>
<tr>
<th>Reporters</th>
<th>[4]</th>
<th>[9]</th>
<th>[11]</th>
<th>[44]</th>
<th>This work</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anode volume (( \mu l ))</td>
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<td>1.5</td>
<td>4.5</td>
<td>350</td>
<td>39.3</td>
</tr>
<tr>
<td>Anode area (cm(^{2}))</td>
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<td>2.25</td>
<td>0.01</td>
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</tr>
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<td>Gold</td>
<td>Gold</td>
<td>Gold</td>
<td>UME/gold</td>
</tr>
<tr>
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<td>Lactate</td>
<td>Acetate</td>
<td>Acetate</td>
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</tr>
<tr>
<td>Catholyte</td>
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<td>Ferricyanide</td>
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</tr>
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<td>Naion</td>
<td>Naion</td>
<td>Naion</td>
<td>Naion</td>
</tr>
<tr>
<td>Inoculum</td>
<td>Geobacter sulfurreducens enriched</td>
<td>Shewanella oneidensis MR-1</td>
<td>Geobacter sulfurreducens enriched</td>
<td>Geobacter sulfurreducens enriched</td>
<td>Geobacter sulfurreducens enriched</td>
</tr>
<tr>
<td>Internal resistance (k( \Omega ))</td>
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<td>NA</td>
<td>0.9</td>
</tr>
<tr>
<td>Areal resistivity (k( \Omega ) \cdot cm(^{2}))</td>
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<td>22.5</td>
<td>NA</td>
<td>0.167</td>
</tr>
<tr>
<td>P(_{\text{areal}}) (W m(^{-2}))</td>
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<td>0.047</td>
<td>0.12</td>
<td>7.72</td>
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<td>P(_{\text{volumetric}}) (W m(^{-3}))</td>
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<tr>
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<td>0.49</td>
<td>10.6</td>
<td>0.12</td>
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<td>Energy conversion efficiency (%)</td>
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<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>15–17</td>
</tr>
</tbody>
</table>

3.2. Areal resistivity

Internal resistance and areal resistivity were calculated based on the polarization curves (figure 4). According to Ren et al, areal resistivity, which is the internal resistance normalized to a unit surface area, is effective for comparing different MFCs [3] and thus we compared this. As flow rates increased from 2 \( \mu \)l min\(^{-1}\) to 6 \( \mu \)l min\(^{-1}\), the areal resistivity for all MFCs decreased, in accordance with the increase of the power and current density. At a higher flow rate (i.e. 8 \( \mu \)l min\(^{-1}\)), areal resistivity of all three MFCs increased; similar to the power density, this is possibly due to the detachment of biofilm. The areal resistivity values of 165 \( \pm \) 8 \( \Omega \) cm\(^{2}\) and 171 \( \pm \) 12 \( \Omega \) cm\(^{2}\) were at least one order of magnitudes lower than that of prior studies of micro-scale MFCs.

3.3. Biofilm characterization

Figure 5 illustrates SEM images of the biofilm on the UME and control anodes. Geobacter formed a thick and dense biofilm on the UME anodes. The width of the metal ring (20 \( \mu \)m) and biofilm (50 \( \mu \)m) were marked. The biofilm extended out of the UME laterally by \( \sim \)15 \( \mu \)m on both sides. Outside of this area, Geobacter spread sparsely on the glass slides. This biofilm profile is similar to the SEM images of Liu et al [22], and the extension limit of 15 \( \mu \)m agrees well. It was believed that when Geobacter are located far away from the anode, they cannot form a thick and dense biofilm due to the limitation in long range EET [22, 27, 28]. The sparse Geobacter on the glass slide may rely on residual oxygen or minute EET to the anode, and their electricity generation capability is negligible.

The effective area of biofilm was 2.5 times the anode area of the concentric ring-shape UMEs, whereas that of the plain gold disk electrode remained almost constant. This was because the area of the disk electrode, 82.6 mm\(^{2}\), was significantly larger than the increased biofilm area. Taking into consideration of the increase of the effective area of biofilm, the areal power density normalized to the biofilm area was 3.09 \( \pm \) 0.04 W m\(^{-2}\) and 2.70 \( \pm \) 0.05 W m\(^{-2}\) for 100 \( \mu \)m and 220 \( \mu \)m gap UMEs, resulting in a power density of 56% and 36% improvement compared with that of the control (plain-disk electrode). This power density enhancement was due to the mass transfer improvement induced by the edge effect of the UME anode.

Significant power enhancement was expected on MFCs equipped with UME anodes over the plain gold disk electrode. However, the power densities of these two MFCs with UMEs demonstrated rather moderate improvement, less than expected. Two possible reasons accounted for the rather incremental power enhancement: (1) although the mass transfer at the vicinity of biofilm on the UME was enhanced, the mass transfer inside the biofilm was still limited. Effective diffusion coefficient of acetate inside biofilm is one fourth of that in aqueous solution, due to the presence of microbial cells, extracellular polymers, etc [29]. Consequently mass transfer of acetate and H\(^{+}\) carrying buffer was lower in the biofilm than in the anolyte, which resulted in insufficient acetate and finite acidification inside biofilm, limiting the current and power enhancement; (2) the electrons generated by Geobacter far from the anode had a lower transfer rate into the anode, resulting in the
The micro-scale MFCs in this work may find applications in powering ultra-low power electronics, such as low-power wireless sensor network and passive RFID tags, especially in long-term environmentally unfriendly conditions, where it is difficult to approach for regular maintenance [1, 2, 35–38]. The wireless sensor network and RFID can be integrated on MFC by microfabrication techniques [39–41]. The power density presented in this work is comparable to, or slightly higher, than conventional energy conversion techniques (table 3) [1, 42, 43].

5. Conclusion

This study presents carbon-neutral renewable micro-scale MFCs with high power density and efficiency by implementing a concentric ring-shape UME. The successful implementation delivers high power densities of 7.72 W m\(^{-2}\) (per original anode surface area), 3.09 W m\(^{-2}\) (per effective surface area), as well as a high CE and EE of approximately 70% and 17%, respectively. The high power density and efficiency makes the micro-scale MFCs useful for powering ultra-low power electronics, especially in long-term environmentally unfriendly conditions.

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