JOURNAL OF
POWER
SOURCES

Short communication

A microbial fuel cell using permanganate as the cathodic electron acceptor

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Abstract

The microbial fuel cell (MFC) was proved to be a novel bioprocess capable of recovering electrical energy from organic matter. In this study, we report that by using permanganate as the cathodic electron acceptor for a MFC we were actually able to recover much more electrical power than using other existing types of electron acceptors, e.g. using permanganate as the cathodic electron acceptor for a two-chamber MFC generated a maximum power density of 115.60 mW m^{-2} which was, respectively, 4.5- and 11.3-fold higher than that produced by using hexacynoferrate (25.62 mW m^{-2}) and oxygen (10.2 mW m^{-2}) as the cathodic electron acceptor. This could be attributed to the higher open circuit potential (OCP) provided by permanganate in the MFC. Besides, pH, unlike permanganate concentration, was further found to have a major impact on the OCP and the cathode potential. SEM and XPS analysis demonstrated that manganese dioxide (MnO_2) was in fact the main reduced product of the permanganate at pH 3.6. Moreover, as compared to a two-chamber MFC, a bushing MFC using permanganate as the cathodic electron acceptor achieved an unprecedented maximum power-output of 3986.72 mW m^{-2}. This study for the first time showed that permanganate could be used as an effective cathodic electron acceptor for a MFC.
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Keywords: Microbial fuel cell; Permanganate; Open circuit potential; Internal resistance; Electron recovery

1. Introduction

Recently, microbial fuel cells (MFCs) are drawing increasing attention as an effective means for electricity energy recycling from not only carbohydrates [1–3] but also from complex organic wastewater [4,5]. Microorganisms such as members of the Geobacter family [6], *Shewanella putrefaciens* [7], *Rhodoferax ferrireducens* [2], *Clostridium butyricum* [8] and *Aeromonas hydrophila* [9], are found able to oxidize organic matter by accepting electrons via the electrode to obtain energy for their own growth, and this process gives rise to electricity generation.
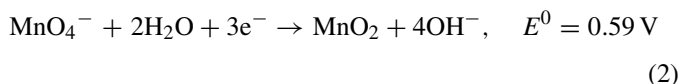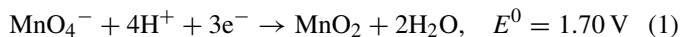
One of the most commonly used MFCs in the laboratory consists of an anode chamber and a cathode chamber being separated by a proton exchange membrane (PEM) [1,10–13].

However, power generation in this system is always found constrained by its high internal resistance (up to 1000 Ω) [10]. Consequently, most research work attempting to enhance MFCs power generation have focused on minimizing the internal resistance including omitting the PEM [14], reducing the distance between the two electrodes [15], and even adding sodium chloride into a single chamber MFC so as to increase its ion strength [15]. On the other hand, the maximum OCP of the MFC is reported ranging from 0.5 to 0.8 V [11,16]. Although a variety of modified MFCs have been designed in order to enhance the electricity recovery, such as a flat plate MFC [4], single chamber MFC [14,17], air-cathode MFC [14,15], UASB MFC [18] and tubular MFC [19], most of those systems fail to achieve cell voltage above 0.8 V due to the fact that almost all of them are using either hexacynoferrate [11,20,21] or oxygen [1,2,4–6,13–15] as cathodic electron acceptor whose maximum theoretical redox potential does not exceed 1.0 V. Moreover, the presence of an overpotential between both electrodes may further reduce cell voltage to a lower level. Therefore, cathodic oxidants with

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high redox potentials are highly desirable to improve MFC efficiency.

In fact, a lot of specialized oxidants have been widely used in industry, among which permanganate is the most commonly used [22–24] by virtue of its high oxidization capacity as well as its environmental safety. In both acidic and alkaline conditions, permanganate accepts three electrons and thus is reduced to manganese dioxide [25,26] as illustrated in Eqs. (1) and (2):

$$MnO_4^- + 4H^+ + 3e^- \rightarrow MnO_2 + 2H_2O, \quad E^0 = 1.70\,V \quad (1)$$

$$MnO_4^- + 2H_2O + 3e^- \rightarrow MnO_2 + 4OH^-, \quad E^0 = 0.59\,V \quad (2)$$

Eqs. (1) and (2) point to a fact that in the acidic condition, permanganate actually has a much higher oxidation potential than in alkaline conditions. Therefore, an acid condition is indeed expected when using permanganate as the cathodic electron acceptor in a MFC. It should be realized that until now permanganate has not been used as the cathodic electron acceptor in MFCs.

This study comprises four phases, i.e. (i) using permanganate as the cathodic electron acceptor for electricity generation in a two-chamber MFC within which effects of pH and initial permanganate concentration were examined; (ii) comparison of permanganate to the other cathodic electron acceptors such as hexacynoferrate and oxygen (with Pt and without Pt) in two-chamber MFCs in terms of power generation and Coulomb efficiency (CE); (iii) analyses of the chemical form of the cathodic reduced products by using scanning electron microscope (SEM) and X-ray photoelectron spectrometer (XPS); (iv) comparison of power output between permanganate and hexacynoferrate as cathodic electron acceptors in a bushing MFC (BMFC) at lower internal resistances.

2. Materials and methods

2.1. Experimental set-up

The two-chamber MFC was set-up by connecting two plastic bottles (10.0 cm in height and 3.5 cm in diameter) by a tube

(4.0 cm in length and 2.0 cm in diameter) which contained a PEM (E-TEK) inside. Preparations of PEM and electrodes were done as described by Bond and Lovely [6]. In order to facilitate the growth of biofilm, carbon paper (2.0 cm × 5.0 cm, E-TEK) with rough surface and porosity was placed in the anode chamber to accumulate the electron-transferring bacteria. A carbon cloth (2.0 cm × 5.0 cm, E-TEK) was inserted into the cathode chamber to act as a cathode. In tests with oxygen in the cathode, a carbon cloth loaded with Pt catalysts (5% content and 0.4 mg cm^{-2}, E-TEK) was used. The schematic of this two-chamber MFC can be referred to Fig. 1A.

The BMFC had a different design for the anode and cathode as compared to that of a two-chamber MFC. Its anode chamber was made with a plastic bottle (10 cm in height and 3.5 cm in diameter) which has four arc-shape holes separated by four thin bars (8.0 cm in height and 0.5 cm in width). While the cathode chamber was designed to enclose the anode chamber inside with an annular interspace of 100 mL. The PEM (84.5 cm^2 in total area) was hot-pressed onto the bars to separate the anode and the cathode chambers. Carbon cloth (E-TEK) was inserted into a column (8.0 cm in height and 5.0 cm in diameter) and then placed in the arc zone of the cathode chamber. The schematic of this BMFC set-up is illustrated in Fig. 1B.

2.2. Inoculation

Anaerobic sludge was cultivated by incubating aerobic activated sludge (3500 mg MLSS L^{-1}), collected from the secondary clarifier of Wenchang Wastewater Treatment Plant, Harbin, PR China, in a 1000 mL anaerobic bottle. 1.0 g L^{-1} glucose was used as carbon source. For innoculation, diluted anaerobic sludge (500 mg MLSS L^{-1}) was mixed with 600 mg chemical oxygen demand (COD) L^{-1} glucose, and injected into the anode chamber of two-chamber MFC reactor. After successful startup, medium containing glucose (600 mg COD L^{-1}) as the sole electron donor, together with the other nutrients such as NaHCO$_3$ (3.13 g L^{-1}), NH$_4$Cl (0.31 g L^{-1}), KCl (0.13 g L^{-1}), NaH$_2$PO$_4$ (4.22 g L^{-1}), Na$_2$HPO$_4$ (2.75 g L^{-1}), (NH$_4$)$_2$SO$_4$ (0.56 g L^{-1}), MgSO$_4$·7H$_2$O (0.2 g L^{-1}), CaCl$_2$ (15 mg L^{-1}), FeCl$_3$·6H$_2$O (1 mg L^{-1}), MnSO$_4$·H$_2$O (20 mg L^{-1}) and neces-
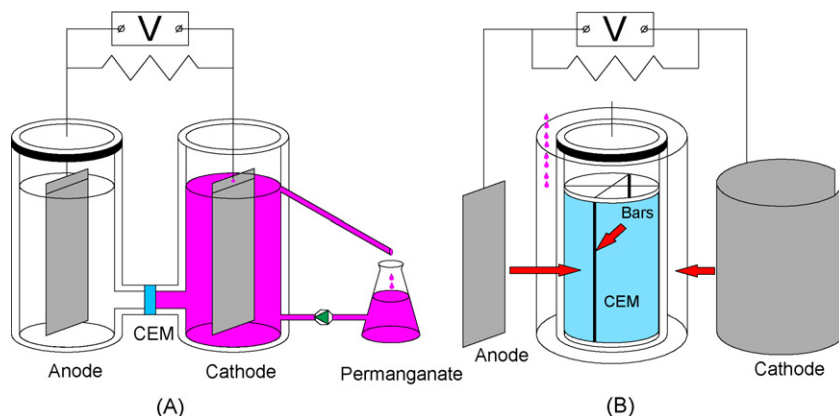

Fig. 1. Schematic of (A) two-chamber MFC and (B) bushing MFC (BMFC). The two MFCs are different in the area of the PEM and cathode configurations.

sary trace elements, was employed to sustain the continuous electricity production in two-chamber MFC.

After finishing this two-chamber MFC test, its anodic electrode with biofilm was taken out and inserted into the anode of BMFC. The action of disassembling and assembling of the anode was carried out in an anaerobic glovebox. Cathodic solutions with pre-determined concentration were prepared with analytical-grade potassium permanganate ($KMnO_4$) or potassium hexacynoferrate [$K_3Fe(CN)_6$] chemicals. A peristaltic pump (type BT100-1Z, China) was used to circulate the cathodic solution between the cathode chamber and a conical flask (1000 mL). Copper wires were used to connect the anode and the cathode of both MFCs. Different external resistance (50–50,000 Ω) was applied in order to obtain polarization curve for determination of the maximum power generation.

2.3. Calculations

Voltages were recorded every 60 s directly from the potentiostat via a dual-channel voltage collection instrument (12 bit A/D conversion chips, US) connecting to a computer by universal serial bus (USB, Intel) interface. Calibration was done with a digital multimeter (Agilent HP 34970, US) before each test. Measurements of the working potential of the cathode were carried out by a reference electrode (Ag/AgCl, 195 mV versus standard hydrogen electrode, SHE) connecting to a multimeter (Agilent HP 34970, US). Voltage obtained was converted to power density P (mW m^{-2}) according to $P = UI/A$ in which U (V) denotes the voltage, I (mA) the current, and A (m^2) the area of the anode.

Coulombic efficiency (CE) was calculated according to

$$CE = \frac{C_{EX}}{C_{TH}} \times 100\% \tag{3}$$

in which C_{EX} is the total coulombs by integrating currents measured at each time interval over time, i.e.

$$C_{EX} = \frac{\int U(t)\,dt}{R} = \frac{\sum_{i=1}^{T} U_i t_i}{R} \tag{4}$$

where R (Ω) is the external resistance and T (s) is the total reaction time once voltage output below 0.05 V. And

$$C_{TH} = \frac{bCVF}{M} \tag{5}$$

where M is relative molecular mass (32 g mol^{-1} by oxygen), b the number of moles of electrons produced per mole of substrate (4 mol e$^-$ mol^{-1} based on oxygen), C (mg L^{-1}) the overall removal of substrate in term of COD measured according to the standard methods [27], V (96 mL) the anode liquid volume and F the Faraday's constant (96,485 C (mol e$^-$)$^{-1}$).

2.4. Analyses

The morphologies of the anode and cathode surfaces were studied by using scanning electron microscope (SEM) (Hitachi, S570; Japan). The anodic samples were collected and fixed

overnight with 2.5% paraformaldehyde and 1.5% glutaraldehyde in a buffer solution (0.1 M cacodylate, pH 7.5) at 4 °C, and then washed twice followed by stepwise dehydration in a gradient series of water/ethanol solutions (25, 50, 70, 85, 95, 100%), and then was critical-point dried (carbon dioxide). Samples were finally coated with Au/Pt before SEM observation. Cathodic samples were fixed and dried overnight in a vacuum oven (25 °C), followed by a direct examination. Determination of the chemical composition of cathodic reduced products was done by using an X-ray photoelectron spectrometer (XPS) (PH1 5700 ESCA system, US). This XPS study was conducted by using a hemispherical analyzer and an aluminum anode (monochromatic Al Kα 1486.6 eV) as source (at 12–14 kV and 10–20 mA). The system was operated under retarding model with a binding energy of 630–680 eV. The peaks for manganese element were calibrated with reference to C1s (284.6 eV). pH was adjusted by using sulfuric acid (1 mol L^{-1}) or sodium hydroxide (2 mol L^{-1}), and measured by pH meter (type pHs-3c, Shanghai, China).

3. Results

3.1. Electricity generation in a two-chamber MFC with permanganate as the cathodic electron acceptor

The two-chamber MFC was operated in batch mode by adding glucose as substrate into anode and permanganate solution (0.1 g L^{-1}, pH 7.0) into the cathode. Fig. 2 shows measured OCP and current after 240 h operation. OCP steadily increased from 0.75 V at 240 h to 0.9 V at 246 h (Fig. 2), after which an artificial decrease of pH to 3.5 was done in the cathode chamber and this led to a substantial increase of OCP to 1.382 V. Such a sharp OCP increase is most likely due to the high redox potential (1.70 V) of permanganate in acidic conditions as illustrated in Eq. (1) [25]. In a control MFC with no bacteria growth on anode,
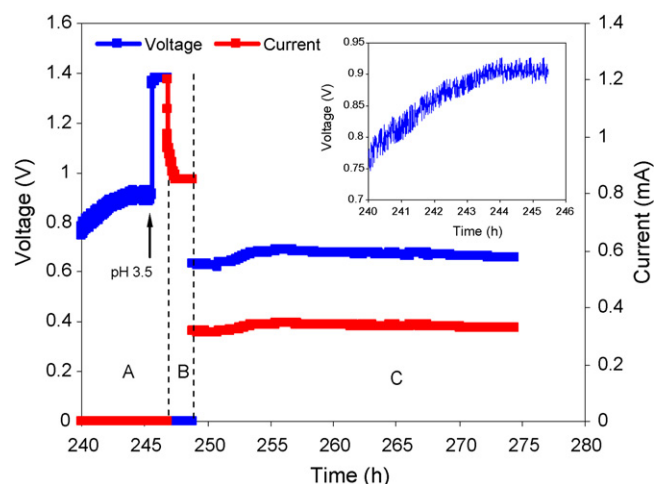

Fig. 2. Voltage and current generation in a two-chamber MFC using permanganate at different operation modes, in which (A) represents the disconnection condition, (B) the short-circuit condition and (C) the connection of the external resistance of 2000 Ω within the circuit. The inserted figure stands for the initial OCP (from the values above 0.75 V).

Fig. 3. SEM image of electron-transfer bacteria attached to the surface of the anode.

Fig. 4. Effect of (A) pH (permanganate of $100\,\mathrm{mg\,L^{-1}}$) and (B) initial permanganate concentration (at pH 3.5) on the potential of two-chamber MFC.

no voltage was observed, indicating the absence of biotic reaction in the anode chamber. After an external resistance ($2000\,\Omega$) was inserted into the circuit from 250 to 275 h, the OCP quickly fell down to 0.68 V (Fig. 2). The internal resistance of the system was determined as $1623\,\Omega$ which was comparable to that observed in two-chamber membrane-MFC ($1286\,\Omega$) by Min et al. [10]. The SEM observation (Fig. 3) shows that anodic electrode surface was covered by bacilliform bacteria which were responsible for electron transfer and thus current generation in the MFC.

3.2. Effects of pH and initial permanganate concentration on OCP and cathode potential in the two-chamber MFC

A pH-dependent OCP and cathode potential change was observed in Fig. 4A. For example, OCP and cathode potential decreased from 1.38 to 0.432 V and 1.18 to 0.352 V, respectively, as the pH increased from 3.6 to 9.5. In another words, high pH played a negative effect on the OCP and cathode potential when using permanganate in this MFC. This observation is actually in line with theoretical predictions in Eqs. (1) and (2). On the other hand, the OCP and cathode potential appeared to be insensitive to the initial permanganate concentration, e.g. OCP and cathode potential increased only 9.2 and 9%, respectively, when the permanganate concentration increased 10-fold from 20 to $200\,\mathrm{mg\,L^{-1}}$ (Fig. 4B).

3.3. Effects of initial permanganate concentration and external resistance on the current density

The effect of permanganate concentration on current density with an external resistance (R_{ex}) of 200 and $2000\,\Omega$ is shown in Fig. 5. It can be seen that when the MFC was operated at low R_{ex}, e.g. $200\,\Omega$, the current density increased at a slope of 0.152 ($R^2 = 0.991$) with permanganate concentration from 0.02 to $0.2\,\mathrm{g\,L^{-1}}$. Once switched to a high R_{ex}, e.g. $2000\,\Omega$, this slope dropped 19-fold to 0.008 ($R^2 = 0.981$). This seems to point to the
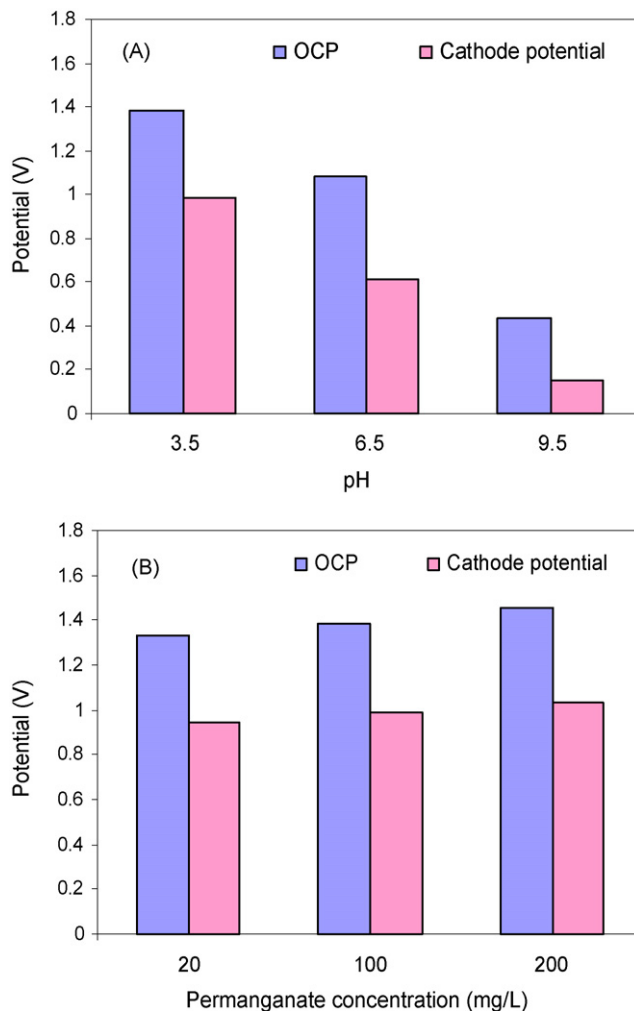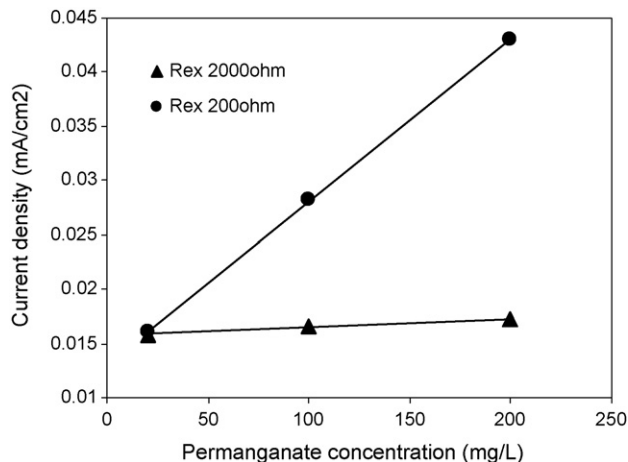

Fig. 5. Current density as a function of the permanganate concentration in a two-chamber MFC.
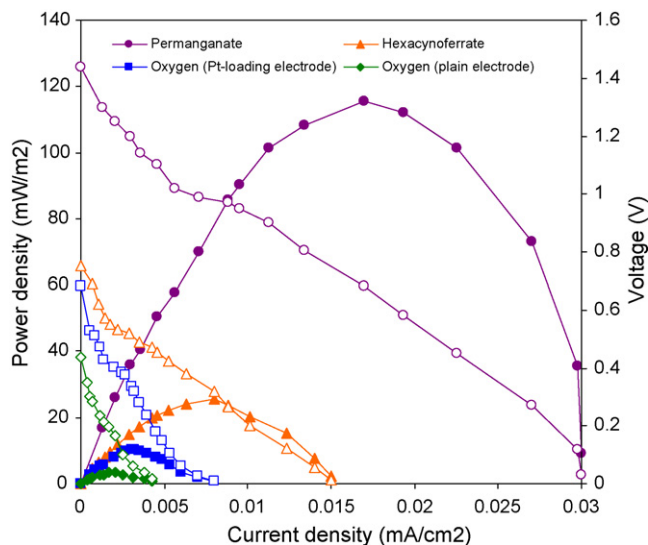
Fig. 6. Power generation as a function of the current density using permanganate (10 mM), hexacyanoferrate (10 mM) and oxygen (Pt-loading electrode and plain electrode) in the cathode of a two-chamber MFC (open symbols indicated voltage and filled symbols indicated power).

fact that a high permanganate concentration is required to ensure efficient electricity production while the external resistance has to be taken into consideration as it is apparently a limiting factor to current density.

3.4. Comparison of permanganate, hexacynoferrate and oxygen as electron acceptors in the two-chamber MFCs

Comparative studies using permanganate (10 mM, pH 3.6), hexacynoferrate (10 mM) and oxygen as electron acceptors were performed in the two-chamber MFCs. Corresponding polarization curve was obtained by varying external resistance from 50 to 50,000 Ω. As can be seen from Fig. 6, the highest maximum power density (115.60 mW m^{-2}) was achieved in MFC using permanganate as electron acceptor at a cur-
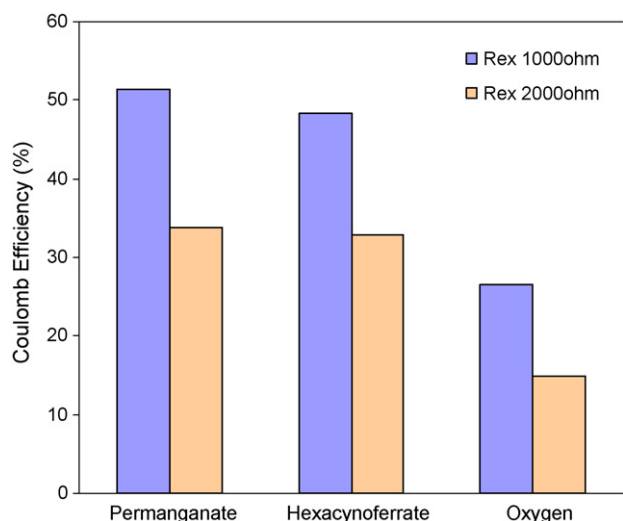
rent density of 0.017 mA cm^{-2}. While, hexacynoferrate and oxygen used MFCs only produced maximum power densities of 25.62 mW m^{-2} (at 0.008 mA cm^{-2}), 10.2 mW m^{-2} (at 0.003 mA cm^{-2} with Pt) and 3.40 mW m^{-2} (at 0.002 mA cm^{-2} without Pt), respectively, which are all even less then one fifth of that in permanganate used MFC (Fig. 6).

At $R_{ex} = 1000 \, \Omega$, CE was determined as 51.43, 48.29 and 26.55% in MFCs using permanganate, hexacynoferrate and oxygen (with Pt) as electron acceptors, respectively (Fig. 7). When R_{ex} was increased from 1000 to 2000 Ω, a general CE decrease was observed in Fig. 7. The similar phenomenon was also observed by Rabaey et al. [19].

3.5. Characteristics of the Cathode Surface

SEM photos clearly showed deposits was formed on the surface of the cathode (Fig. 8). To identify the chemical form of
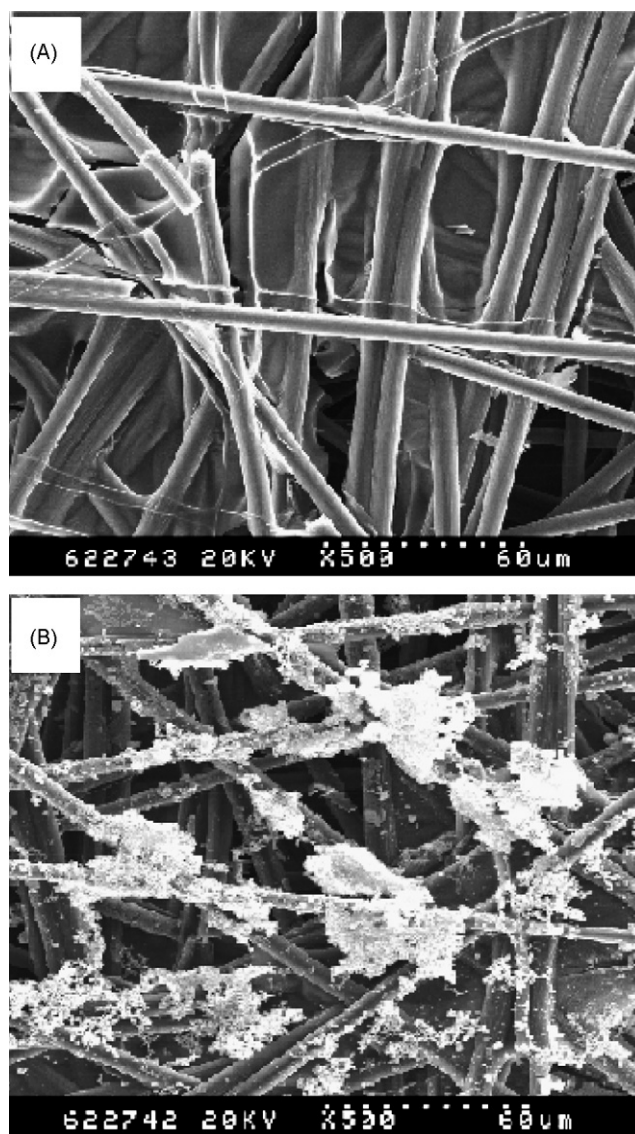

Fig. 8. SEM image of (A) plain cathode in a control set-up without current and (B) surface of cathode at pH 3.6 in two-chamber MFC (both permanganate concentration of 100 mg L^{-1}; reacting time of 15 h).
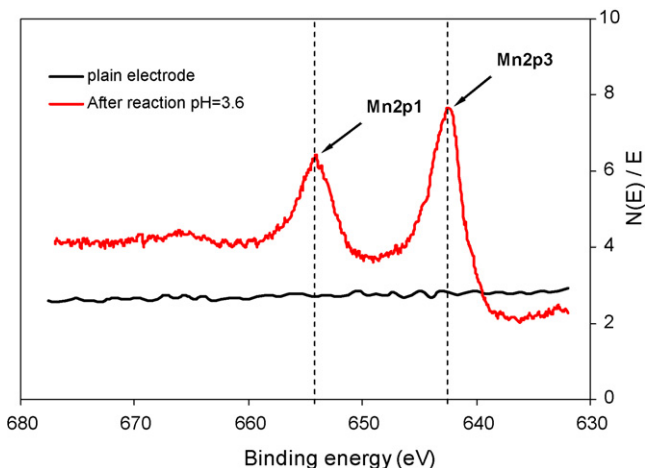

Fig. 7. Coulombic efficiency (CE) using different cathodic electron acceptors in two-chamber MFC.

Fig. 9. XPS analysis for the surface of the cathode (permanganate concentration of $100\,mg\,L^{-1}$; reacting time of 15 h).
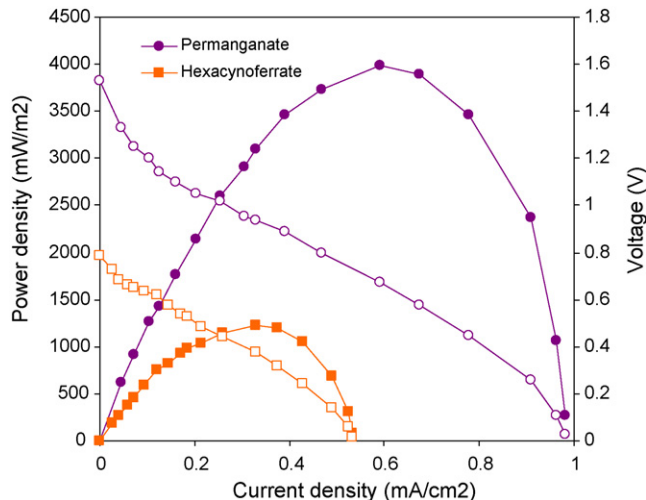

Fig. 10. Power generation as a function of the current density in a BMFC using permanganate (10 mM) and hexacyanoferrate (10 mM), respectively (open symbols indicated voltage and filled symbols indicated power density).

those nubbly or netty deposits on the carbon fiber, XPS analysis was applied. As shown in Fig. 9, binding energies peaks corresponded to Mn 2p1 (654.03 eV) and Mn 2p3 (642.6 eV) (Fig. 9), pointing to a fact that manganese dioxide (MnO_2) was the main cathodic products [28].

3.6. Electricity generation in BMFCs using permanganate and hexacynoferrate as electron acceptors

BMFC is considered to have less ohmic and cathodic limitation than others due to its larger PEM and cathode area. Hence, BMFCs using permanganate (10 mM and pH 3.6 ± 0.1) and hexacynoferrate (10 mM) as respective electron acceptors were studied and compared. The results in Fig. 10 revealed that the maximum power density in used BMFC was $3986.72\,mW\,m^{-2}$ (at $0.59\,mA\,cm^{-2}$) which accounted for more than three times of that in hexacynoferrate used BMFC ($1231\,mW\,m^{-2}$ at $0.33\,mA\,cm^{-2}$). The OCP of permanganate (1.532 V) used BMFC were also two times of that in hexacynoferrate used BMFC (0.788 V). While the internal resistance in permanganate used BMFC (51.2 Ω) accounted for only 1.43-fold of that in hexacynoferrate used (BMFC) (72.7 Ω). CEs also appeared high when using permanganate (78.49%) in comparison with using hexacynoferrate (76.65%). All those information point to one fact that using permanganate as electron acceptor in BMFC has many advantages over using hexacynoferrate such as its high power density, high OCP, low internal resistance as well as high CE.

4. Discussion

Permanganate has been used as an environment-friendly oxidant in industries for many years [11]. Its high redox potential offers the possibility of its application in a fuel cell system to establish a high potential difference between the anode and the cathode [29]. In this study, we for the first time showed that permanganate also can be used as an effective cathodic electron acceptor in MFC, e.g. five-fold more power density can be achieved in a permanganate two-chamber MFC than with other electron acceptors such as hexacynoferrate and oxygen; In a BMFC, also a three-fold maximum power density can be produced when using permanganate as the electron acceptor as compared to using hexacynoferrate.

The OCP indicates the potential difference between the anode and the cathode, in which the anode potential is generally determined by factors such as substrate conversion rate, electrons from microorganisms to anode transfer rate and surface characteristic of anode; while cathode potential depends on the types of cathodic electron acceptor used as well as the mass and electrochemical reaction rates. Assuming the redox potential of $NAD^+/NADH$ in the anode is a constant of -0.32 V [30], OCP would be only dependent on the performance of cathode. Although the OCP in a MFC is usually reported in the range of 0.50–0.80 V when oxygen or hexacynoferrate was used as

Table 1
Redox reactions and potentials for different electron acceptors used in the cathode of a MFC (standard condition, temperature 273 K and pressure 1 atm; vs. SHE)

Oxidant	Theoretical potential (V)	Experimental potential (V)	Experimental OCP (V)	References
Fe^{3+}	+0.771	0.332	0.772	[11]
Oxygen	+0.804[a]	0.268	0.751	[11]
H_2O_2	+1.270	–	0.620	[31]
MnO_4^-	+1.700[b]	1.284	1.532	This study

[a] Based on partial pressure of oxygen of 0.2 and pH 7.0.
[b] At permanganate concentration of $1\,mol\,L^{-1}$ and pH 1.0.

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the cathode [16], it should be realized that a different electron acceptor actually gives a different cathode potential on the basis of the redox potential (Table 1), and thus the OCP value actually depends on the type of electron acceptor used. As introduced previously, permanganate gives a higher OCP in acidic conditions than in alkaline conditions (Eqs. (1) and (2)). Since manganese dioxide has been identified to be the main cathodic product in this study (Fig. 8), and the cathode potential as a function of pH and permanganate concentration can be described by using Nernst equation based on Eq. (1), i.e.

$$E = 1.70 + 0.0197 \log[MnO_4^-] - 0.0787pH \quad (6)$$

If we assume $[MnO_4^-] = 0.1\,g\,L^{-1}$ $(6.33 \times 10^{-4}\,mol\,L^{-1})$ and pH 3.6 based on the results from this study, the cathode potential (E) could be calculated as 1.342 V from Eq. (6). When $[MnO_4^-]$ is increased 400% from 0.1 to 0.5 g L^{-1}, the cathode potential would only increase 2.4% to 0.033 V. It means the E value is insensitive to $[MnO_4^-]$. This indicates that MFC should better off be operated at relative low permanganate concentration.

Power density can be calculated by using

$$P = \frac{OCP^2}{(R_{ex} + r)\,A} \quad (7)$$

where r denotes the internal resistance and A denotes the anode area which is 20 cm^2 in this study.

It can be seen from Eq. (7) that power density would increase with OCP but decrease with other variables. This explains the reason why using permanganate is able to produce a much higher maximum power density than using hexacynoferrate or oxygen. In another work, it is the outstanding redox potential of the permanganate that enhanced the power output of a MFC. The similar mechanism also applies to the other high redox potential electron acceptors such as hexacynoferrate which generates higher power by higher redox potentials than dissolved oxygen as reported by Oh et al. [11].

The internal resistance (r) is a limiting factor in power generation in a MFC as indicated in Eq. (7). BMFC is known to have a low r in comparison with a two-chamber MFC mainly as a result of it's large area of the PEM. This is in line with the results in this study, i.e. r in BMFC is only 3% of that in two-chamber MFC. For that reason, BMFC produced 34.5 times more power density than the two-chamber MFC (Figs. 6 and 10), implying that reducing the internal resistance is indeed a necessary means to enhance power generation in a MFC.

Based on information obtained from this study, it is reasonable to conclude that permanganate is an effective electron acceptor. It is able to enhance the maximum power density of a MFC up to an unprecedentedly high level of 3986.72 mW m^{-2} in contrast to other studies, e.g. 1330 mW m^{-2} by Liu et al. [15], 1580 mW m^{-2} by Cheng at al. [19] and 3600 mW m^{-2} by Rabaey et al. [21]. Moreover, it is worth pointing out that this permanganate method has no need for a catalyst, which makes this process simple and economical. But on the other hand, it should be noted that like the other liquid-state electron acceptors this permanganate MFC also requires liquid replacements to compensate its depletion. Therefore, this technique may be only applied to small-scale power supplies.

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