Persulfate: A self-activated cathodic electron acceptor for microbial fuel cells

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ARTICLE INFO

ABSTRACT

Potassium persulfate aqueous solution without pH adjustment is used as the cathodic electron acceptor in a two-chamber microbial fuel cell (MFC) in the present study. The performance of the MFC with $K_2S_2O_8$ solution is evaluated and compared with that of $K_3Fe(CN)_6$ solution. The results show that the maximum power density of the $K_3Fe(CN)_6$ used MFC doubled that with $K_2S_2O_8$ solution when fresh aqueous solution is used. However, a significant increase in electrical power generation is observed in the case of the MFC with $K_2S_2O_8$ solution after 2-day operation under an external resistance of 1000 Ω. This improvement can be attributed mainly to the increased cathode performance as a result of the hydrolysis of the persulfate ion in aqueous solution. It is demonstrated that persulfate can be used as an effective cathodic electron acceptor due to its unique capability of self pH adjustment.
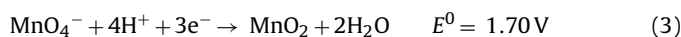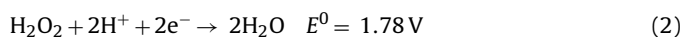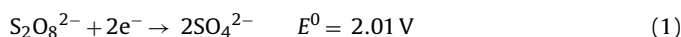
1. Introduction

Persulfates are strong oxidants that have been widely used in many industries for initiating emulsion polymerization reactions, clarifying swimming pools, hair bleaching, micro-etching of copper printed circuit boards, total organic carbon analysis and destructing soil and groundwater contaminants. The persulfate anion is the most powerful oxidant in the peroxygen compounds. Spent persulfates are classified as hazardous waste because they are oxidizers. An acceptable disposal method for spent persulfate solutions is to dilute them with large quantities of water and dispose via a treatment system. However, it is not an economical and environmental-friendly solution and thereby should be replaced by a more sustainable one.

Because of their capability to produce electricity and simultaneously treat organic wastewater, much attention has been paid to microbial fuel cells (MFCs) as a sustainable energy generation devices [1–5]. One of the most common MFCs in the laboratory usually consists of an anode and a cathode separated by a proton exchange membrane (PEM). In the anode compartment, microorganisms oxidize organic wastewater to generate electrons and protons. The protons travel through the membrane, while the electrons travel through an external circuit. They are combined with electron acceptors at the cathode. The net result of these simultaneous reactions is the generation of electrical power. There are many factors that influence the maximum power production in MFCs, such as, proton transport through the membrane, operational

parameters, cell configuration, and electrode surface properties [6–20]. Recently, increasing efforts have been devoted to optimizing the reduction of the electron acceptor on the cathode electrode surface [21].

So far, oxygen is still widely used as the electron acceptor in most MFC applications due to its availability in the environment and its high redox potential. However, the main disadvantages of the poor contact between gaseous oxygen and cathode electrode as well as the slow rate of oxygen reduction on the surface of carbon electrodes hinder the application of MFCs. In order to overcome these problems, the use of catalyst-coated air cathode is a promising approach for the cathode reaction improvement [22–26]. In addition to oxygen, hexacyanoferrate is also a widely used electron acceptor in MFCs because it can considerably improve the cathode performance due to its faster reduction kinetics and higher concentration on the cathode electrode [27–29]. Cathode losses can also be minimized using a class of strong oxidants like permanganate [30], hydrogen peroxide [31] and hexavalent chromium [32]. However, the practical applications of these oxidants are limited because the performance is highly dependent on the initial pH value in the cathode compartment.
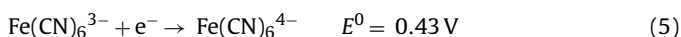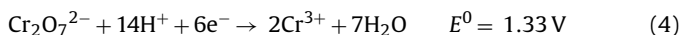
In aqueous solutions, the standard oxidation–reduction potential (E^0) for the half-cell persulfate reaction is ~2.12 V (Eq. (1)). This potential is higher than the redox potential for hydrogen peroxide ($E^0 = 1.78$ V), permanganate ($E^0 = 1.70$ V), hexavalent chromium ($E^0 = 1.33$ V) and ferricyanide (Eqs. (2)–(5)):

$$S_2O_8{}^{2-} + 2e^- \rightarrow 2SO_4{}^{2-} \qquad E^0 = 2.01\,V \tag{1}$$

$$H_2O_2 + 2H^+ + 2e^- \rightarrow 2H_2O \quad E^0 = 1.78\,V \tag{2}$$

$$MnO_4{}^- + 4H^+ + 3e^- \rightarrow MnO_2 + 2H_2O \qquad E^0 = 1.70\,V \tag{3}$$

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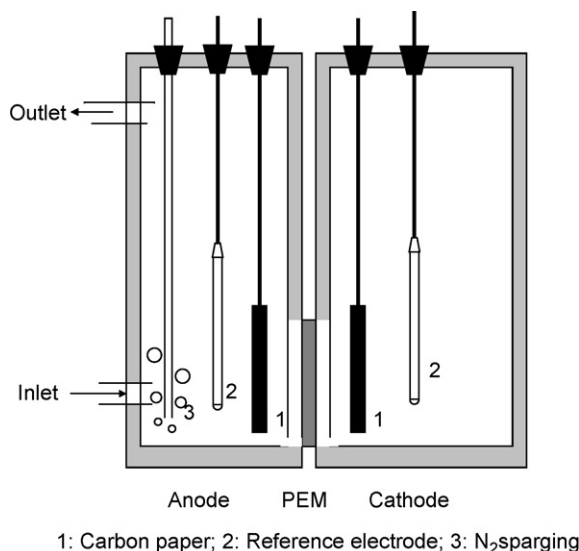
Fig. 1. Schematic of the two-chamber MFC (1: carbon paper; 2: reference electrode; 3: N$_2$ sparging).

$$Cr_2O_7{}^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O \qquad E^0 = 1.33\,V \qquad (4)$$

$$Fe(CN)_6{}^{3-} + e^- \rightarrow Fe(CN)_6{}^{4-} \qquad E^0 = 0.43\,V \qquad (5)$$

In this sense, spent persulfate can also be applied as the cathode electron acceptor in MFCs due to its high oxidation–reduction potential.

In the present study, potassium persulfate aqueous solution at pH 6.1 (without pH adjustment) was used as the electron acceptor in the cathode chamber of a two-chamber MFC. The performance improvement and polarization behaviors of the anode and cathode before and after 2 days operation at a constant external load 1000 Ω are described. The possible mechanism for the performance improvement of the K$_2$S$_2$O$_8$ used MFC is proposed. In addition, the effects of various operating conditions on its performance are also investigated.

2. Materials and methods

2.1. MFC construction

As shown in Fig. 1, a two-chamber MFC was constructed by two acrylic cubic chambers with 500 mL in volume separated by a PEM of 2.2 cm in diameter. Pre-treated PEM was cleaned by successively boiling the membranes in 10% H$_2$O$_2$ solution, followed by deionized (DI) water, 0.5 mol L^{-1} H$_2$SO$_4$ solution, and then DI water again. Each step continued for 1 h. Both anode and cathode electrodes were made of non-wet proof carbon paper (5.0 cm × 5.0 cm). The carbon papers were connected to copper wires and an external resistor box. Two Ag/AgCl reference electrodes were placed in the anode and cathode compartments and connected to the respective electrodes, for independently measuring the individual electrode potentials.

2.2. MFC operation and measurement

The anode compartment was inoculated with effluent from another MFC running on acetate, and then was continuously supplied with medium [9], containing sodium acetate 2.7 g L^{-1}, 6 g L^{-1} Na$_2$HPO$_4$, 3 g L^{-1} KH$_2$PO$_4$, 0.1 g L^{-1} NH$_4$Cl, 0.5 g L^{-1} NaCl, 0.1 g L^{-1} MgSO$_4$·7H$_2$O, 15 mg L^{-1} CaCl$_2$·2H$_2$O and 1.0 mL L^{-1} of a trace elements solution [32]. During the inoculation, the cathode compartment was filled with 10 mM potassium ferricyanide solu-

tion and continuously stirred. A stable current was generated from the reactor after about 2 weeks of continuous feeding at this condition. In order to maintain a mixed condition in the anode chamber and to minimize the influence of the anode potential on the overall cell performance, the anodic medium was continuously supplied with the medium at a rate of 0.4 L day^{-1} during the operation. All MFC tests were conducted in a temperature-controlled room at 25 °C.

Voltage (U) and current (I) between two electrodes were measured across a fixed load every 10 or 30 min, and the data were collected automatically by an Agilent 34970A data acquisition unit. The external resistor was varied in a range of 10–100,000 Ω to determine the maximum power density of the MFC. Power density (P) was calculated according to $P = UI/A$, where A is the surface area of the electrode. The potential of each electrode was obtained by immersing an Ag/AgCl reference electrode into solutions, and the pH of the cathodic compartment was measured by a pH meter (S20K, Mettler-Toledo). Persulfate concentrations were determined by spectrophotometric analysis (Lengguang 756mc spectrophotometer) using iodine oxidation according to the method from [33].

3. Results and discussion

3.1. Cell performance

In the present study, K$_2$S$_2$O$_8$ is attempted to be used as the cathode electron acceptor of the MFC and its electricity production is compared with that using K$_3$Fe(CN)$_6$ solution under the same conditions. The corresponding polarization curves of the MFC, as shown in Fig. 2, are obtained by varying the external resistance from 10 Ω to 100 kΩ. It can be seen in Fig. 2a that the measured voltages of the MFC with K$_2$S$_2$O$_8$ are higher than that with K$_3$Fe(CN)$_6$ at low current densities (<20 mA m^{-2}) including the open-circuit condition. This is due to the higher open-circuit potential (OCP) of the persulfate cathode. However, this is not the case at medium to high current densities (>20 mA m^{-2}), where the cell performance of the K$_2$S$_2$O$_8$ used MFC is considerably lower than that with K$_3$Fe(CN)$_6$ solution. This can be attributed to the faster elelctro-reduction kinetics of ferricyanide solution on the surface of the carbon electrode than that of K$_2$S$_2$O$_8$. The maximum power densities of the MFC using fresh K$_3$Fe(CN)$_6$ as the cathode electron acceptor is 166.7 mW m^{-2}, which doubled that with K$_2$S$_2$O$_8$ solution (83.9 mW m^{-2}). However, the cell performance for the case of K$_2$S$_2$O$_8$ solution increases significantly after 2 days operation under an external resistance of 1000 Ω (Fig. 2b). The measured polarization curves show that the OCP of the MFC using K$_3$Fe(CN)$_6$ solution remains unchanged, while the OCP of the K$_2$S$_2$O$_8$ used MFC increases from 1256.3 to 1447.8 mV. Furthermore, the K$_2$S$_2$O$_8$ used MFC shows higher voltages over the whole spectrum of the current density than that of the MFC using K$_3$Fe(CN)$_6$ and the maximum power density reached 211.9 mW m^{-2} after 2-day operation, whereas that of the MFC using K$_3$Fe(CN)$_6$ solution drops to 154.3 mW m^{-2}. The performance decline of the K$_3$Fe(CN)$_6$ used MFC is can be attributed mainly to the decrease of the K$_3$Fe(CN)$_6$ concentration resulting from the consumption of K$_3$Fe(CN)$_6$ during the electrochemical reaction at the cathode.
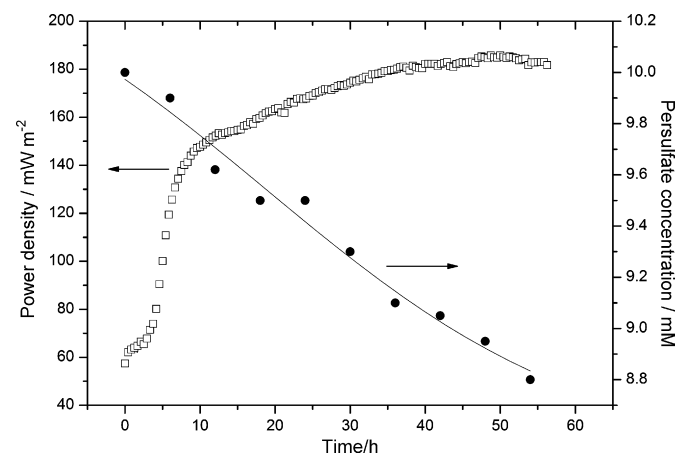
3.2. Anode and cathode potentials

As described in Section 3.1, the performance of the MFC using K$_2$S$_2$O$_8$ as the cathodic electron acceptor increases with the operation time. The performance increase of the K$_2$S$_2$O$_8$ using MFC during the operation time is quite inconsistent with our knowledge that the consumption of cathodic electron acceptors generally leads to a lower performance due to a deteriorated oxidant supply to the cathode (c.f. Fig. 3). In order to understand this phenomenon, the
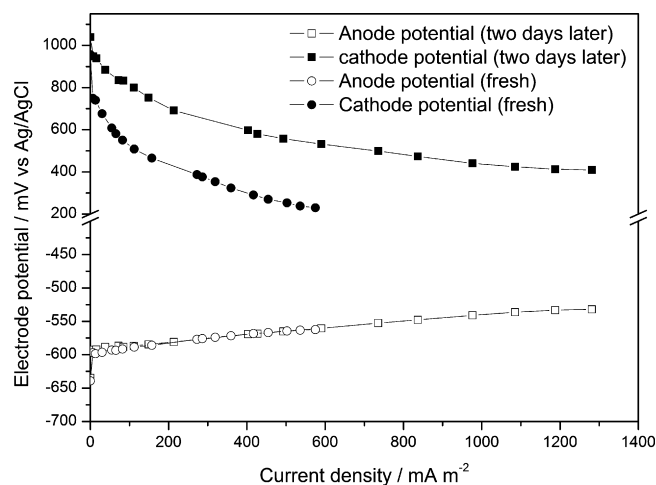
(a)

(b)
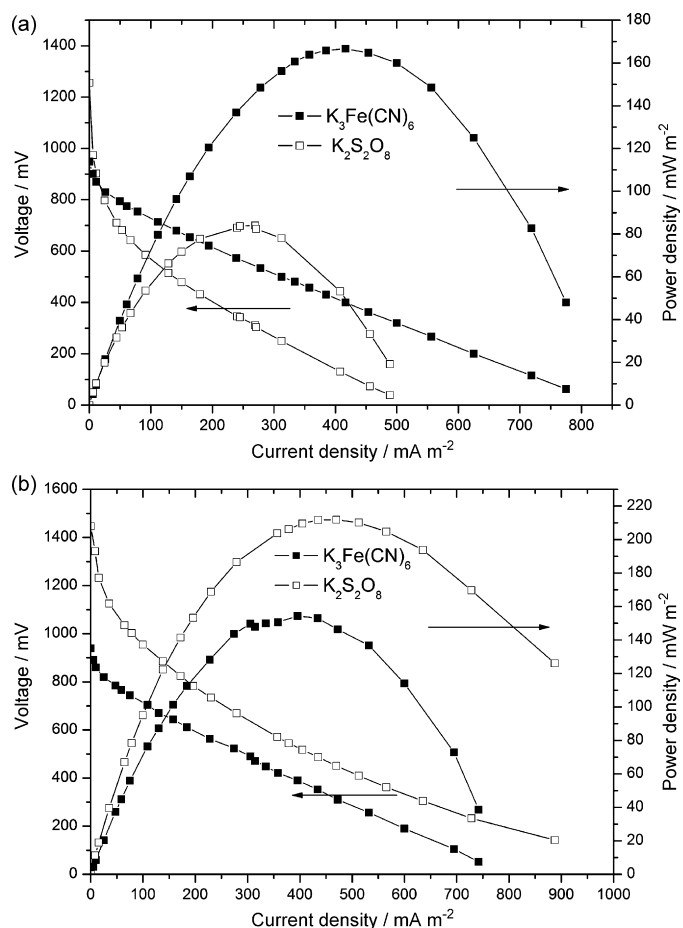
Fig. 2. Polarization curves of the MFC using $K_3Fe(CN)_6$ and $K_2S_2O_8$ solution as the cathodic electron acceptor. (a) Fresh solution, (b) after 2-day operation under an external load of $1000\,\Omega$.

Fig. 4. Effect of operation time on the cathode and anode polarization characteristics of the $K_2S_2O_8$ used MFC (initial pH in the cathode chamber: 6.1; external resistance: $1000\,\Omega$).

Ag/AgCl reference electrodes are adopted to separate the anode and cathode performance, respectively. Fig. 4 shows the cathode and the anode polarization curves of the $K_2S_2O_8$ used MFC before and after 2 day operation at a constant external load of $1000\,\Omega$. It can be clearly seen that the polarization curves of the anode are nearly identical before and after the operation, whereas the cathode performance is significantly improved. These results imply that the increase in the cathode potential is the main reason for the performance improve-

ment of the $K_2S_2O_8$ used MFC during the operation. In addition, it is also worth to note, as it can be derived from Fig. 4, that the difference between the cathode and anode potentials does not correspond to the cell voltage in Fig. 2. This can be mainly attributed to the presence of ohmic losses in the electrodes and electrolytes [21].

Furthermore, the anode and cathode potentials of the $K_2S_2O_8$ used MFC at a constant external load $1000\,\Omega$ are recorded as functions of time and shown in Fig. 5. The cathode potential monotonically increases to 466.3 mV during the first 10 h, and then it increases more slowly with time reaching a maximum of 497.7 mV after 40 h of operation. However, the anode performance degrades over time, which can be attributed to the increased overpotential at the anode as a consequence of the enhanced current generation from the MFC at a fixed resistor. Nevertheless, the increase of the anode potential is relatively small compared with that of the cathode. The MFC performance losses resulted from the slightly deteriorated anode performance are compensated by the greatly enhanced MFC cathode performance, leading to a steady net increase in power density from 61.1 to 183.3 mW m^{-2}, as indicated in Fig. 3.
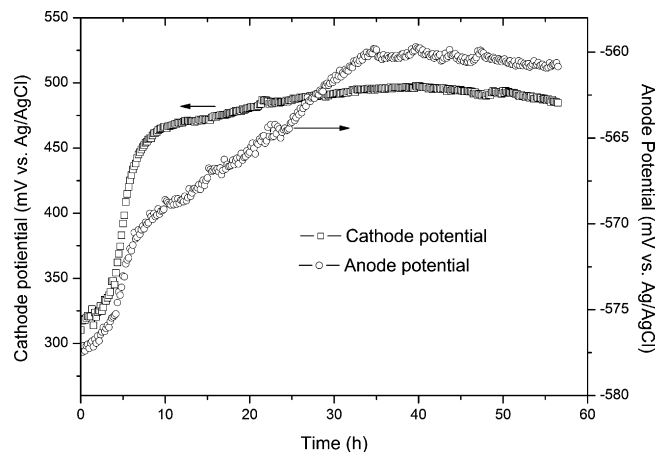

Fig. 3. Variation of power density and persulfate concentration with time for the $K_2S_2O_8$ used MFC during the operation under an external load of $1000\,\Omega$.

Fig. 5. Variation of anode and cathode potentials with time for the $K_2S_2O_8$ used MFC during the operation under an external load of $1000\,\Omega$ (initial pH in the cathode chamber: 6.1).
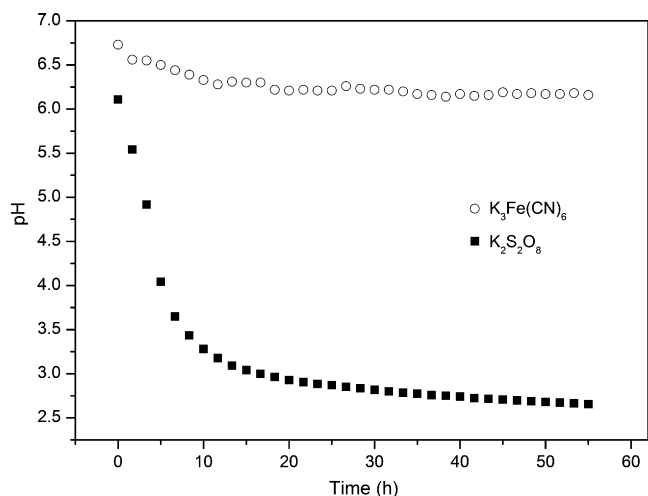
Fig. 6. Variation of the cathode pH with time for the $K_3Fe(CN)_6$ and $K_2S_2O_8$ used MFCs during the operation under an external load of 1000 Ω.
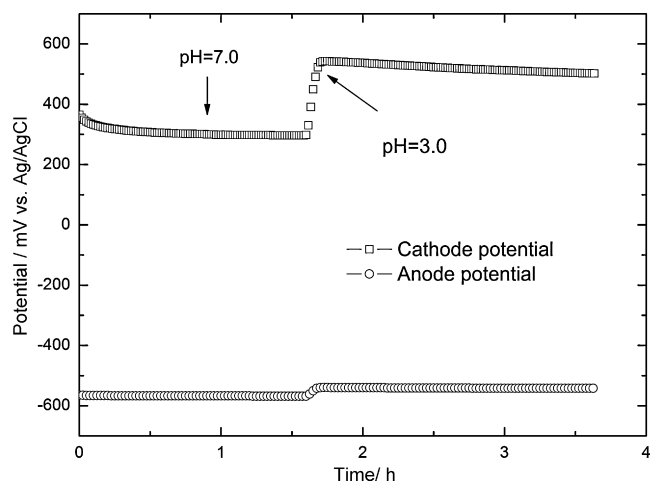

Fig. 7. Effect of pH adjustment by HCl in the cathode chamber on the performance of the $K_2S_2O_8$ used MFC (initial pH of the cathode chamber: 7.0; external resistance: 1000 Ω).

3.3. Possible mechanism for cathode performance improvement of the $K_2S_2O_8$ used MFC

Fig. 6 illustrates the pH change of the cathode chamber in $K_2S_2O_8$ used MFC during the operation under an external resistance of 1000 Ω. It is seen that the cathode pH value of the $K_2S_2O_8$ used MFC decreased sharply from 6.1 to 3.3 within first 10 h and then gradually decreased to 2.65 in the following 40 h. The decrease of pH during the operation might be explained not only by the hydrolysis of the persulfate ion in aqueous solution (Eq. (6)), but also by the migration of protons from anode to cathode resulted from the continuous production of proton in the anode chamber during the operation.

$$2S_2O_8^{2-} + 2H_2O \rightarrow O_2 + 4SO_4^{2-} + 4H^+ \tag{6}$$
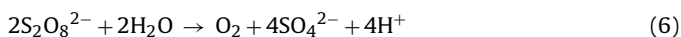
To address the possible effect of proton migration from anode to cathode on the cathode pH, the pH changes of the cathode chamber in $K_3Fe(CN)_6$ used MFC are also recorded (Fig. 6). It can be seen in Fig. 6 that the cathode pH value of the $K_3Fe(CN)_6$ used MFC exhibits a slight decrease, suggesting that the amount of protons migrated from anode to cathode during the operation is relatively much smaller than that of produced from the hydrolysis of the persulfate ion in aqueous solution.

In addition, it is interesting to note that the variation trend of the catholyte pH is nearly opposite to that of the cathode potential during the operation time as it appears in Fig. 5. This result implies that the decrease of the catholyte pH plays an important role in the improvement of the power generation during the operation. To determine the effect of pH value in the cathode chamber on the performance of the $K_2S_2O_8$ used MFC, HCl is added to the cathode chamber with $K_2S_2O_8$ solution (pH 7.0) to obtain the desired pH value during the operation (at 1000 Ω). It can be found from Fig. 7 that the cathode potential increases sharply from 297 to 530 mV within several minutes, whereas the anode potential increases from −567 to −542 mV when the cathode chamber pH decreases from 7.0 to 3.0. This confirms that the pH value of the catholyte has a significant influence on the performance of the $K_2S_2O_8$ used MFC.

It is generally accepted that the hydrolysis of the persulfate ions involves the formation of reactive sulfate and hydroxyl radicals (Eqs. (7)–(8)), which possess stronger oxidization and are more active than $S_2O_8^{2-}$ ($E^0_{SO_4^-} = 2.6$ V; $E^0_{OH^-} = 2.7$ V) [34].

$$S_2O_8^{2-} \rightarrow 2SO_4^{-\bullet} \tag{7}$$

$$SO_4^{-\bullet} + H_2O \rightarrow H^+ + SO_4^{2-} + HO^\bullet \tag{8}$$

Therefore, it is sure that these intermediates participate in the cathode reaction as well. From the above discussion, it can be deduced that the cathode performance would depend not only on the reduction of $S_2O_8^{2-}$, but also on the yield of $SO_4^{-\bullet}$, and OH^-.
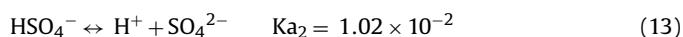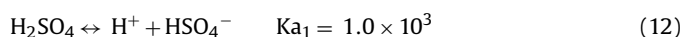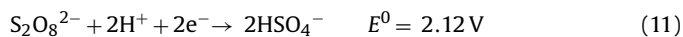
The decrease of pH in the cathode chamber during the operation has the following two beneficial effects on the yield of $SO_4^{-\bullet}$, and OH^-: (1) under acidic conditions the breakdown of persulfate into sulfate free radicals can be further acid catalyzed as follows [35]:

$$S_2O_8^{2-} + H^+ \rightarrow HS_2O_8^- \tag{9}$$

$$HS_2O_8^- \rightarrow SO_4^{2-} + H^+ + SO_4^{-\bullet} \tag{10}$$

(2) the life-time of these radicals decreases with the increase of the pH value since sulfate and hydroxyl radicals decay rapidly due to the competitive reaction with hydroxyl ions [36,37]. Therefore, the pH decrease in the cathode compartment due to the hydrolysis of the persulfate ions leads to a higher radical yield and thereby an enhanced cell performance.

3.4. Effect of initial cathode pH on cell performance

To further investigate the above hypothesis, we evaluated the cell performances using the catholyte with the same initial $K_2S_2O_8$ concentration (10 mM) and different initial pH values. The half-cell persulfate reaction can be represented either as Eq. (1), excluding protons in the equation, or as Eq. (11), which includes protons. These are linked by the prototropic equilibria (Eqs. (12) and (13)).

$$S_2O_8^{2-} + 2H^+ + 2e^- \rightarrow 2HSO_4^- \qquad E^0 = 2.12 \text{ V} \tag{11}$$

$$H_2SO_4 \leftrightarrow H^+ + HSO_4^- \qquad Ka_1 = 1.0 \times 10^3 \tag{12}$$

$$HSO_4^- \leftrightarrow H^+ + SO_4^{2-} \qquad Ka_2 = 1.02 \times 10^{-2} \tag{13}$$

On the basis of Eqs. (11), (12) and (13), the theoretical cathode potential, $E_{cathode}$, can be thermodynamically predicted by the Nernst equation as,

$$E_{cathode} = E^0(S_2O_8^{2-}, H^+/HSO_4^-)$$
$$+ \frac{RT}{2F} \ln \frac{[S_2O_8^{2-}]}{C^2} + \frac{RT}{F} \ln \left(\frac{[H^+]^2}{Ka_1} + [H^+] + Ka_2 \right) \tag{14}$$

where E^0 is the standard cathode potential; $[S_2O_8^{2-}]$ and $[H^+]$ are the concentrations (mol L^{-1}) of the two types of ions in solution; R is the ideal gas constant (8.314 J mol^{-1} K^{-1}); T is the temperature (K); F is the Faraday's constant (96,485 C mol^{-1}); C

Table 1
Effect of pH on the OCP, maximum power density, anodic and cathodic potential of the $K_2S_2O_8$ used MFC.

pH value	OCP (mV)	Anodic potential (mV vs. Ag/AgCl)	Cathodic potential (mV vs. Ag/AgCl)	Maximum power density (mW m^{-2})
1.0	1532	−578	1178	198.7
2.0	1592	−614	1160	215.8
3.0	1558	−614	1105	151.4
4.0	1494	−614	1040	95.7
5.0	1415	−615	961	48.5
7.0	1284	−614	846	24.5
10.0	1086	−614	594	23.6

Table 2
The maximum power densities of the MFCs with different persulfate concentrations.

Persulfate concentration (mM)	Maximum power density before operation (mW m^{-2})	Maximum power density after operation (mW m^{-2})	Increase in maximum power density (mW m^{-2})
5	52.9	178.7	125.8
10	84.0	211.6	127.6
20	135.5	266.2	130.7
30	183.0	317.5	134.5

is defined as: $C = [H_2SO_4] + [SO_4^{2-}] + [HSO_4^-]$, in which $[H_2SO_4]$, $[SO_4^{2-}]$, $[HSO_4^-]$ represent the concentrations of undissociated sulfuric acid, sulfate and hydrogenosulfate (mol L^{-1}), respectively. It can be easily deduced from Eq. (14) that $E_{cathode}$ should increase with the decrease of the pH in the cathode chamber. However, the values of $E_{cathode}$ calculated from Eq. (14) in a pH range of 2–10 may differ by negligibly small amounts, e.g., by <40 mV when the pH value decreases from 10.0 to 2.0. Therefore, if the above hypothesis is right, the cathode potentials would significantly increase with the decrease of the initial catholyte pH since a lower pH induces a higher radical yield. The initial maximum power density, the OCP value and the anode and cathode potentials of the cell operated at the same initial $K_2S_2O_8$ concentration (10 mM) and different initial catholyte pH values are listed in Table 1. It is clear that the cathode potentials indeed show the anticipated trend. These experiments confirm the importance of the sulfate and hydroxyl radicals on the cell performance.

It is also worth of noting, as it can be seen from Table 1, that the anodic potential shows practically little variation until the initial catholyte pH reaches 2; for a pH value of 1.0, there is a noticeable increase of 40 mV in the anodic potential. It was known that the sulfonate groups of Nafion are mainly occupied by cations in the anolyte [38,7]. This prior occupation results in the hindrance of proton migration between the anode and cathode chamber. The small variation of the anodic potentials in the pH range of 2–10 might be explained by the combined effects of the small proton gradient between both chamber, the capability of buffer solution to stabilize the anolyte pH and the prior occupation of sulfonate groups of Nafion by the cations in the anolyte. However, in the case of the MFC using the catholyte at pH 1.0, the proton migration from cathode to anode is facilitated due to the increased proton concentration gradient, causing the inhibition of bacterial activity and generation of toxic conditions in anode chamber, and consequently the increase of the anodic potential. These results imply that the persulfate solution for practical MFC applications should be at a pH higher than 2.0.

3.5. Effect of initial persulfate concentration on cell performance

The maximum power densities of the MFC before and after 2-day operation at an external load 1000 Ω with different persulfate concentrations are listed in Table 2. It can be seen that the maximum power density of the MFC using fresh persulfate solution increases from 52.9 to 183.0 mW m^{-2} with the increase in the persulfate concentration from 5 to 30 mM. This observation is consistent with the findings reported by You et al. [30] and Wang et al. [39]. After 2

days operation at an external load 1000 Ω, the maximum power densities of the MFC using 5, 10, 20, 30 mM persulfate increase to 178.7, 211.9, 266.2 and 317.5 mW m^{-2}, respectively. However, comparing the cell performances of the $K_2S_2O_8$ used MFC before and after operation, it is interesting to note that the performance gains of the MFC with different persulfate concentrations show tiny differences. This is understandable because the contribution of sulfate and hydroxyl radicals to the MFC performance improvement is limited since these intermediate species are very unstable and could be quickly consumed before contacting the electrode.

4. Conclusions

In the present study, potassium persulfate aqueous solution without pH adjustment was used as the electron acceptor in the cathode of a two-chamber MFC. The performance of the $K_2S_2O_8$ used MFC was evaluated and compared with that of $K_3Fe(CN)_6$ solution under the same condition. The results show that the maximum power densities with fresh $K_3Fe(CN)_6$ and $K_2S_2O_8$ solution as the cathode electron acceptor are 166.7 and 83.9 mW m^{-2}, respectively. After 2 days operation under an external resistance 1000 Ω, the maximum power density of the MFC using $K_3Fe(CN)_6$ solution as the cathodic acceptor drops slightly, while almost two times increase in the maximum power density is observed in the case of the $K_2S_2O_8$ used MFC. By comparing the results of the cathode and anode polarization curves before and after operation, it is found that the overall cell performance improvement is resulted from the increased cathode performance. It is also demonstrated that the enhanced cathode performance can be mainly attributed to the decrease of the pH value in the cathode chamber as a consequence of the hydrolysis of the persulfate ion in aqueous solution. This study demonstrates that persulfate can be used as an effective cathodic electron acceptor without pH adjustment.

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