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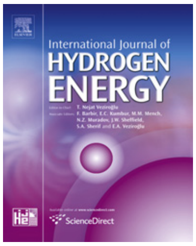

Microbial fuel cell using ferrous ion activated persulfate as a cathodic reactant

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ABSTRACT

In the present paper, the $K_2S_2O_8$–Fe^{2+} system without pH adjustment was used as a cathodic reactant in a two-chamber microbial fuel cell (MFC) for the first time. The performance of the MFC with $K_2S_2O_8$–Fe^{2+} system was discussed and compared with that of $K_2S_2O_8$ and H_2O_2–Fe^{2+} system, respectively. These results demonstrated that the introduced ferrous ion could improve the power generation significantly. The initial $K_2S_2O_8/Fe^{2+}$ molar ratios and the pH, which can affect the performance of MFC, were discussed later. The results revealed that $K_2S_2O_8$–Fe^{2+} system achieved the best performance at $K_2S_2O_8/Fe^{2+}$ molar ratios of 2:1 and the $K_2S_2O_8$-Fe^{2+} system was stable with varying pH value. This study demonstrated that the $K_2S_2O_8$–Fe^{2+} system can be used as an effective cathodic reactant due to its excellent performance.

1. Introduction

Persulfates are mostly used in two industrial applications, polymerization reactions production and printed circuit manufacture. What's more, persulfates are always used as oxidants in cosmetics, textile processing, non-biocidal shock treatment in recreational waters, destructing organic species and contaminants. Spent persulfates are hazardous for human and environment because of their strong oxidizing. Unfortunately, so far there is not an economical and environmental-friendly way to dispose these spent persulfates.

Microbial fuel cell is an emerging technology that can treat wastewater and harvest electricity simultaneously [1–6]. There are several factors that influence the performance of an MFC, including the microbial activity, type of PEM, resistance of the circuit, electrode material and electrolyte solution [7–10]. It is clear that cathode reaction is an important factor to limit the production of electricity in MFC [11–13]. Oxygen is known as the most sustainable cathodic electron acceptor for MFCs, for its availability in the environment and high redox potential [14,15]. However, the slow reduction kinetics on solid electrodes and quite low solubility in water of oxygen limited the performance of MFC [16]. In order to lower the cathodic overpotential for oxygen and improve the efficiency, mediators with high redox potentials were used in MFCs. Ferricyanide ($K_3[Fe(CN)_6]$) as the cathodic electron acceptors has faster reduction kinetics on the cathode and a larger redox potential than that of oxygen [14,17]. The researchers also used permanganate, hydrogen peroxide and hexavalent chromium as the cathodic electron acceptors in MFCs because of their strong oxidizing [18–20]. Nevertheless, they are highly dependent on the initial pH in the cathode chamber.

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Potassium persulfate was an effective cathodic electron acceptor due to its capability of self pH adjustment [21], but the decomposition rate of persulfate was poor. Searching for a novel and efficient electron acceptor is always an issue of great interest.

Aqueous persulfates could decompose in the environment via hydrolysis and metal catalyzed decomposition. The decomposition of persulfate in the presence of ferrous ion can activate persulfate anion to produce the sulfate free radical with a standard redox potential (E^0) of 2.6 V. This potential is higher than the redox potential for sole persulfate ($E^0 = 2.01$ V), hydrogen peroxide ($E^0 = 1.78$ V), permanganate ($E^0 = 1.70$ V), hexavalent chromium ($E^0 = 1.33$ V) and ferricyanide ($E^0 = 0.43$ V). The sulfate free radicals with high oxidation potential can degrade organic contaminants such as lindane and trichloroethylene [22,23] and the high standard redox potential of sulfate free radical will mainly enhance the power generation in MFC. In this sense, the persulfate–ferrous ion system could be used in microbial fuel cell as a cathodic reactant and increase the decomposition rate of spent persulfate.

In the present study, the authors investigated a two-chamber MFC using ferrous ion activated potassium persulfate as the cathode solution. The performance improvement of MFC using $K_2S_2O_8$–Fe^{2+} system compared to other cathodic electron acceptors was described. The authors also evaluated the effects of initial $K_2S_2O_8/Fe^{2+}$ molar ratios and the pH on MFC performance.

2. Materials and methods

2.1. MFC configuration

A two-chamber microbial fuel cell used in the present study was made of polycarbonate and built in a traditional “H” shape. The two compartments were connected by a tube (inner diameter = 2.0 cm) using a proton exchange membrane (PEM, Nafion™ 117, Dupont Co. USA) as a separator, and each chamber has an effective volume of 250 mL. The PEM was pretreated in boiled 10% H_2O_2 solution, deionized water, 0.5 mol L^{-1} H_2SO_4, and deionized water for 1.0 h in turn. Both the anode and the cathode electrodes were consisted of two graphite rods, each rod with a length of 50.0 mm and a diameter of 8.0 mm. The surface area immerged into the solution of each rod was 10.8 cm^2 and the distance between the cathode and anode centers was 80.0 mm. Copper wires were used to connect the electrodes containing a resistance of 1000 Ω (unless stated otherwise).

2.2. Operation

The MFC was conducted in a fed batch mode (unless stated otherwise) at the temperature of 30 ± 1 °C in a water bath. During the experiment, the cathode chamber was abiotic and open to the air, the anaerobic anode chamber was filled with 50 mL excess activated sludge (from the Second Wastewater Treatment of Changsha City in China) and 200 ml synthetic wastewater (glucose 1 g L^{-1}, $NaH_2PO_4 \cdot 2H_2O$ 5.618 g L^{-1}, $Na_2HPO_4 \cdot 12H_2O$ 6.150 g L^{-1}, NH_4Cl 0.31 g L^{-1}, KCl 0.13 g L^{-1}, trace mineral 12.5 ml L^{-1} and vitamin solutions 12.5 ml L^{-1} [24], pH 7.0). All reagents used in this study were of analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). The cathode and anode solutions were stirred continuously. The cathode solutions were out of pH adjustment. During the inoculation, the solution of anode chamber was the same as described above (100 mL excess activated sludge and 150 ml synthetic wastewater), and the cathode compartment was filled with potassium persulfate solution of 20 mmol L^{-1}. After the inoculation, the MFC was refilled with anolyte and catholyte several times. When the maximum voltage was reproducible at least three times, the MFC was considered fully acclimated.

2.3. Measurements and calculations

Voltage (E) was measured at 30 min intervals by a multimeter (UNI-T 803; Uni-Trend Electronics Co., Ltd., Shanghai, China) with a data acquisition system connected to a personal computer. Current density (I_A) and power density (P) were obtained according to $I_A = E/RA$ and $P = EI/A$, where E is the voltage, R is the electrical resistance, I is the current, and A is the surface area of the electrode. In order to obtain the polarization curves based on the current density, external circuit resistances were varied from 9000 Ω to 50 Ω in a decreasing order. The anode and cathode potentials were measured by placing a saturated calomel electrode (SCE, +0.242 V vs. SHE) in each chamber for reference. The internal resistances of the MFCs were calculated through the polarization slope method [25]. The concentration of potassium persulfate was determined using spectrophotometric analysis [26] and analyzed by a spectrophotometer (UV754N UV–Vis Spectrophotometer; Shanghai Jingke Scientific Instrument Co., Ltd., Shanghai, China). The pH was measured with a pH meter (pHS-25; Shanghai Rex Xinjing Instrument Co., Ltd., Shanghai, China).

3. Results and discussion

3.1. The performance in a two-chamber MFC with persulfate-ferrous ion system as the cathodic reagent

The performance of MFC with $K_2S_2O_8$–Fe^{2+} system as the cathodic reagent was compared with that using $K_2S_2O_8$ under the same conditions in two days. The initial concentration of $K_2S_2O_8$ was 20 mmol L^{-1}. The $K_2S_2O_8$–Fe^{2+} solution was composed of $K_2S_2O_8$ (20 mmol L^{-1}) and $FeSO_4 \cdot 7H_2O$ (10 mmol L^{-1}). The MFC using $K_2S_2O_8$–Fe^{2+} solution obtained better performance than MFC using $K_2S_2O_8$ in power density and electrode potential, and the changes were shown in Fig. 1A and B, respectively. Fig. 1A demonstrated that the MFC with $K_2S_2O_8$–Fe^{2+} system acquired a higher power density than the MFC with $K_2S_2O_8$ in two days. Fig. 1B indicated that the anode potentials were nearly identical and stable in different MFCs, whereas the cathode potential of MFC with $K_2S_2O_8$–Fe^{2+} system was obviously higher than that with $K_2S_2O_8$. Fig. 1B implied that the increased cathode potential was the main reason for power generation improvement. The maximum power density was also investigated. And the maximum power density of MFC with $K_2S_2O_8$–Fe^{2+} system was

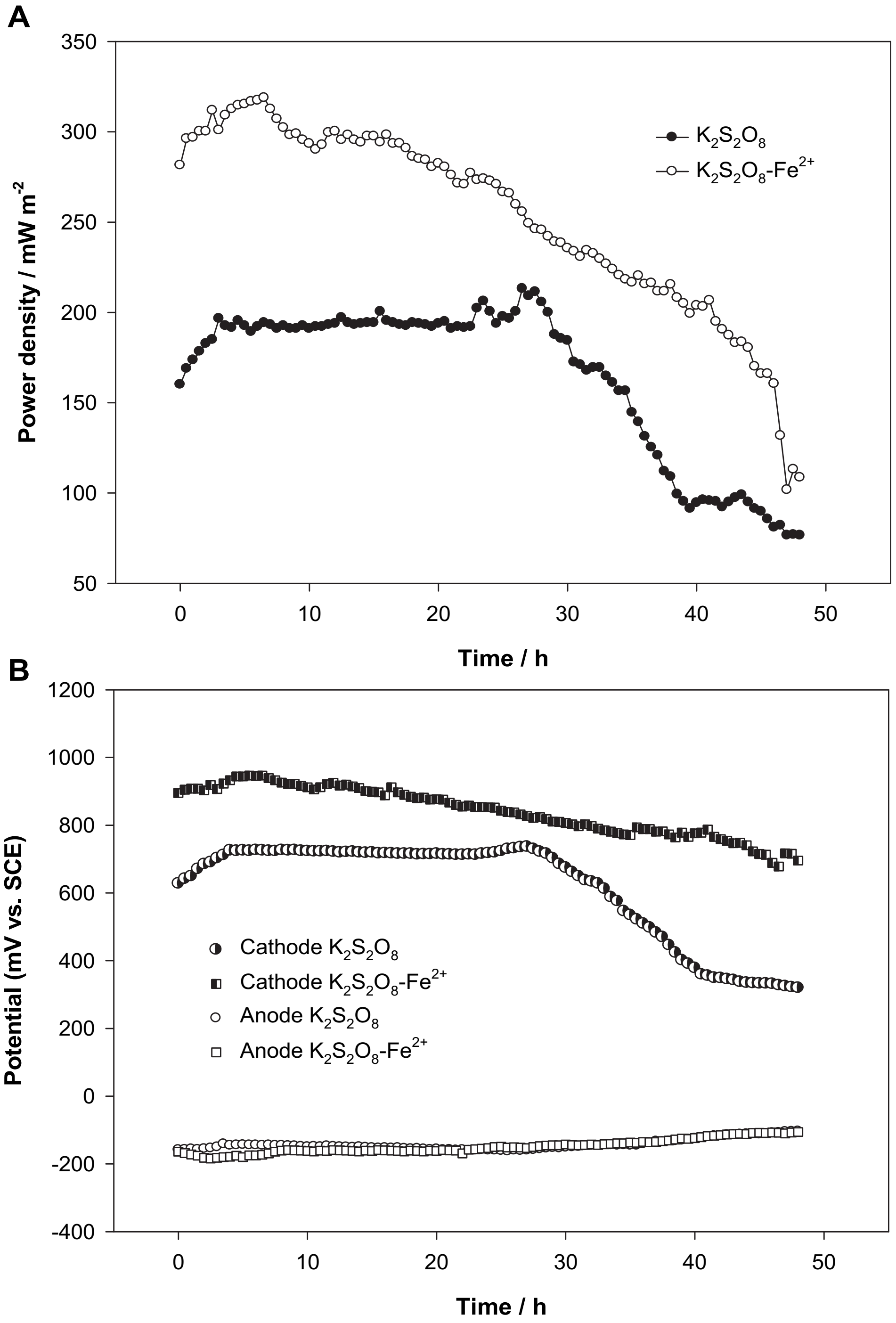

Fig. 1 – Variation of power density (A) and electrode potential (B) as a function of time for the MFCs using $K_2S_2O_8$–Fe^{2+} and $K_2S_2O_8$ as the cathodic reactants.

401 mW m^{-2}, which was nearly 1.7 times higher than the MFC with $K_2S_2O_8$ (234 mW m^{-2}) (Fig. 2).

In addition, ferrous ion can activate persulfate anion to produce the sulfate free radical. According to Eqs. (1) and (2), sulfate free radical with a standard redox potential of 2.6 V which is higher than that of $S_2O_8^{2-}$ (2.01 V).

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

$$SO_4^{\bullet-} + e^- \rightarrow SO_4^{2-} \quad E^0 = 2.60\ \text{V} \tag{2}$$

The increased oxidation–reduction potential of cathodic reagent was responsible for power generation improvement. The stoichiometric relationship between persulfate and ferrous ion was shown in Eqs. (3)–(5) [27]:

$$2Fe^{2+} + S_2O_8^{2-} \rightarrow 2Fe^{3+} + 2SO_4^{2-} \tag{3}$$

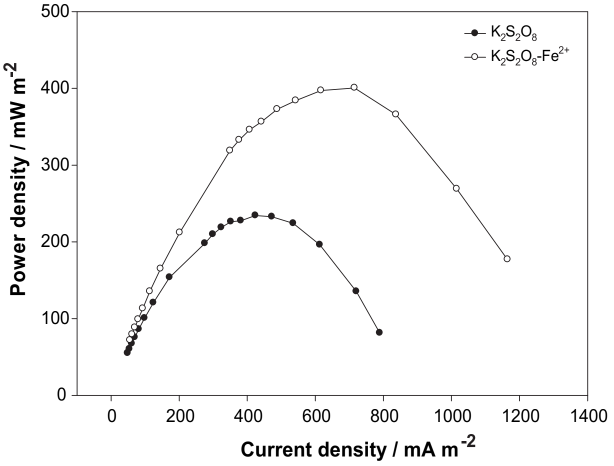

Fig. 2 – Power density curves of MFCs using $K_2S_2O_8$ and $K_2S_2O_8$–Fe^{2+} solutions as the cathodic reagents.

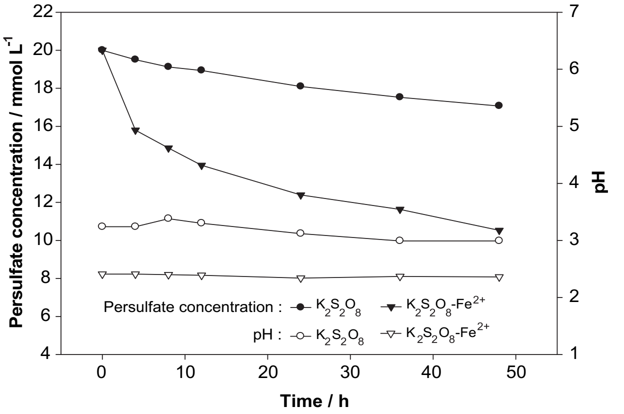

Fig. 3 – The concentration and pH of $K_2S_2O_8$ as a function of reaction time for MFCs using $K_2S_2O_8$–Fe^{2+} and $K_2S_2O_8$ as the cathodic reactants.

Through the steps:

$$Fe^{2+} + S_2O_8^{2-} \rightarrow Fe^{3+} + SO_4^{-\bullet} + SO_4^{2-} \quad (4)$$

$$SO_4^{-\bullet} + Fe^{2+} \rightarrow Fe^{3+} + SO_4^{2-} \quad (5)$$

Sulfate free radicals with high oxidation–reduction potentials could enhance power generation in MFC. The hydrolysis of the persulfate can also produce sulfate free radicals (Eq. (6)).

$$S_2O_8^{2-} \rightarrow 2SO_4^{-\bullet} \quad (6)$$

The activation energy for decomposition of persulfate is 140.2 kJ mol^{-1}, and the activation energy for the reaction between persulfate and ferrous is 50.7 kJ mol^{-1}. Ferrous activated persulfate got lower activation energy than persulfate anion itself. It is easy for the persulfate–ferrous ion reaction to produce sulfate free radical, therefore, the presented MFC system obtained better performance than previous studies with persulfate [21]. A comparison of results with and without ferrous ion revealed that the addition of ferrous ion enhanced the power generation in MFC. Furthermore, the change of $K_2S_2O_8$ concentration and pH was monitored during the operation. The results in Fig. 3 confirmed that the ferrous ion can increase the decomposition of persulfate, and the pH in the cathode solutions exhibited insignificant difference in two days. The pH of the catholyte with $K_2S_2O_8$–Fe^{2+} system was about 2.4.

In batch MFCs, the substrate will be depleted with respect to time, and results in a decrease of bioelectricity production. For this purpose, one approach is to use a continuous MFC to make the nutrients available all the time. And, the continuous MFCs are more suitable than bath system for practical applications [28]. The performance of MFC with a continuous flow MFC in anode chamber was also investigated in this paper. The anode chamber was continuously supplied with the synthetic wastewater in an up-flow mode at a rate of 0.2 L day^{-1}, the cathode chamber was the same as the batch mode (fed with 20 mmol L^{-1} $K_2S_2O_8$ and 10 mmol L^{-1} $FeSO_4 \cdot 7H_2O$). Overall, the operation of the MFC in continuous flow mode resulted in more stable power generation compared to reactors operated in batch mode (Fig. 4). The removal rates of persulfate could reach 90% after 7 days.

3.2. *Comparison of persulfate-ferrous ion system and hydrogen peroxide-ferrous ion system as reagent in the two-chamber MFCs*

$K_2S_2O_8$ and H_2O_2 are inorganic peroxide, and transition metal activators such as ferrous ion (Fe^{2+}) can activate their decomposition and then facilitate the formation of free radicals. Hydroxyl radical with a standard redox potential of 2.8 V was generated from the reaction between hydrogen peroxide and ferrous ion Eqs. (7) and (8).

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + {}^{\bullet}OH + HO^- \quad (7)$$

$$^{\bullet}OH + H^+ + e^- \rightarrow H_2O \quad E^0 = 2.80\ V \quad (8)$$

In this test, the performance of MFC with $K_2S_2O_8$–Fe^{2+} system as the cathodic reagent was compared with the one

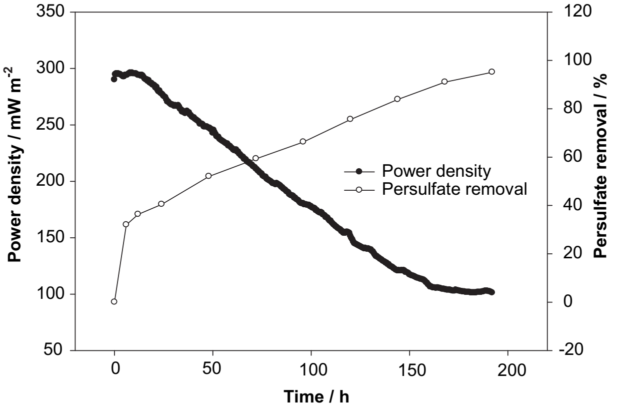

Fig. 4 – Power production and persulfate removal rates using $K_2S_2O_8$–Fe^{2+} solution as the cathodic reagent in a continuous flow MFC.

using H_2O_2–Fe^{2+} under the same conditions in two days. The initial concentration of $K_2S_2O_8$–Fe^{2+} system was the same as Section 3.1. H_2O_2–Fe^{2+} solution was composed of H_2O_2 (initial concentration 20 mmol L^{-1}) and $FeSO_4 \cdot 7H_2O$ (initial concentration 10 mmol L^{-1}). Polarization curves and power density curves for the whole MFC were shown in Fig. 5. Compared to the MFC with H_2O_2–Fe^{2+} system as the cathodic reagent, the maximum power density of the MFC with $K_2S_2O_8$–Fe^{2+} (401 mW m^{-2}) was nearly 2.1 times higher than the MFC with H_2O_2–Fe^{2+} system (187 mW m^{-2}). MFC performance is restricted by internal resistance, since MFC conductors have an intrinsic resistance to charge flow [14,29]. The internal resistance of MFC with $K_2S_2O_8$–Fe^{2+} was 398.5 Ω, much lower than that of MFC with H_2O_2–Fe^{2+} (524.5 Ω). As shown in Fig. 6, the anode potentials obtained from the two different systems were nearly the same, while the cathode potential of MFC with $K_2S_2O_8$–Fe^{2+} was higher than that with H_2O_2–Fe^{2+}. Due to the cathode reaction plays a key role in power generation, the variation of cathode potential was in accordance with the variation of cell voltage. In general, the higher redox potential resulted in better power generation in MFC. But, compared to H_2O_2–Fe^{2+} system, the MFC with $K_2S_2O_8$–Fe^{2+} system exhibited a higher cathode potential and more power generation despite the standard redox potential of hydroxyl radical (2.8 V) was higher than sulfate free radical (2.6 V). This unnatural result between redox and power generation potentially related to the internal resistance, which the $K_2S_2O_8$–Fe^{2+} system is lower than H_2O_2–Fe^{2+} system under the same condition. In addition, the redox potential of $K_2S_2O_8$ in cathode solution ($K_2S_2O_8$–Fe^{2+} system) is higher than H_2O_2 (H_2O_2–Fe^{2+} system), therefore the cathode potential of MFC using $K_2S_2O_8$–Fe^{2+} system displayed better than H_2O_2–Fe^{2+} system overall (Fig. 6). Based on what has been mentioned above, we can safely draw the conclusion that the MFC with $K_2S_2O_8$–Fe^{2+} system performed better than that of H_2O_2–Fe^{2+} system.

3.3. *Effect of initial $K_2S_2O_8$/Fe^{2+} molar ratios on power generation*

Optimization of cathodic reagent composition and concentration can enhance the performance of MFCs [15]. In this section, the $K_2S_2O_8$–Fe^{2+} cathode solutions with five molar ratios of 10:1, 5:1, 2:1, 1:1 and 1:2 were employed. They had the same amounts of $K_2S_2O_8$ (20 mmol L^{-1}) but different Fe^{2+} concentrations. Meanwhile, each cathode solution was without pH adjustment.

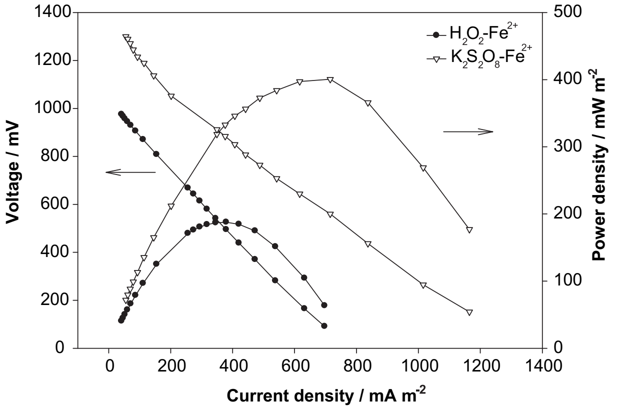

Fig. 5 – Polarization curves and power density curves of MFCs using $K_2S_2O_8$–Fe^{2+} and H_2O_2–Fe^{2+} solutions as the cathodic reagents.

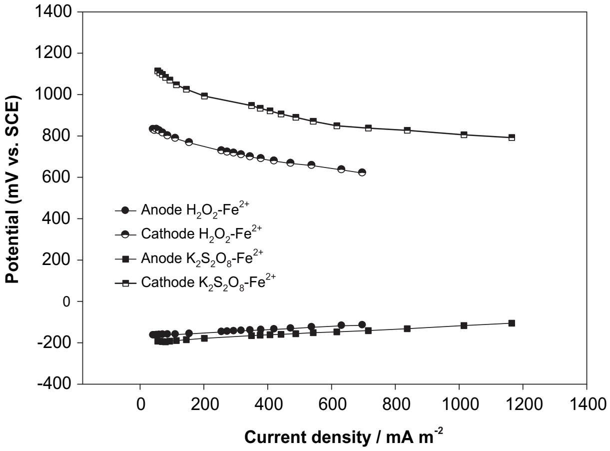

Fig. 6 – The anode and cathode polarization curves of MFCs using $K_2S_2O_8$–Fe^{2+} and H_2O_2–Fe^{2+} solutions as the cathodic reagents.

Power density curves of the five MFCs using different $K_2S_2O_8$–Fe^{2+} molar ratios were shown in Fig. 7A. The cathode solution with $K_2S_2O_8$–Fe^{2+} (2:1) achieved the best performance, a maximum power density of 401 mW m^{-2} at a current density of 715 mA m^{-2}. The maximum power density and cathode potential were increased while the ferrous ion proportion changed from 1:10 to 1:2, and went down at the proportion of 1:1 (Fig. 7B and C). Increasing Fe^{2+} concentration can produce more sulfate free radicals that would increase power output. However, excess Fe^{2+} could possibly lead to the destruction of sulfate free radical (Eq. (5)) [22], and the reduction of sulfate free radical made the power generation decreased in MFC using $K_2S_2O_8$–Fe^{2+} (1:1) system. The internal resistances of MFCs with different $K_2S_2O_8$–Fe^{2+} molar ratios of 10:1, 5:1, 2:1, 1:1 and 1:2, which were calculated from the polarization curve (Fig. 7A), were 512.5 Ω, 447.2 Ω, 398.5 Ω, 383.7 Ω and 352.6 Ω, respectively. The internal resistances decreased with the increasing of the initial concentration of Fe^{2+}, which was also one of the reasons that the ferrous ion enhanced the power generation in MFC systems. Similar to the results of Section 3.1 and 3.2, the variation of cathode polarization curves with different molar ratios was in accordance with the variation of cell polarization curves (Fig. 7C). Cathode reaction was responsible for power generation. The MFC using $K_2S_2O_8$/Fe^{2+} molar ratios 2:1 obtained the highest cathode potential.

3.4. *Effect of pH on power generation in cathode chamber*

The electrolyte pH serves an important function in the MFCs power output [30]. The authors investigated the effect of cathode solution pH on voltage output of the MFCs with $K_2S_2O_8$–Fe^{2+} at the molar ratios of 2:1. The pH of cathodic

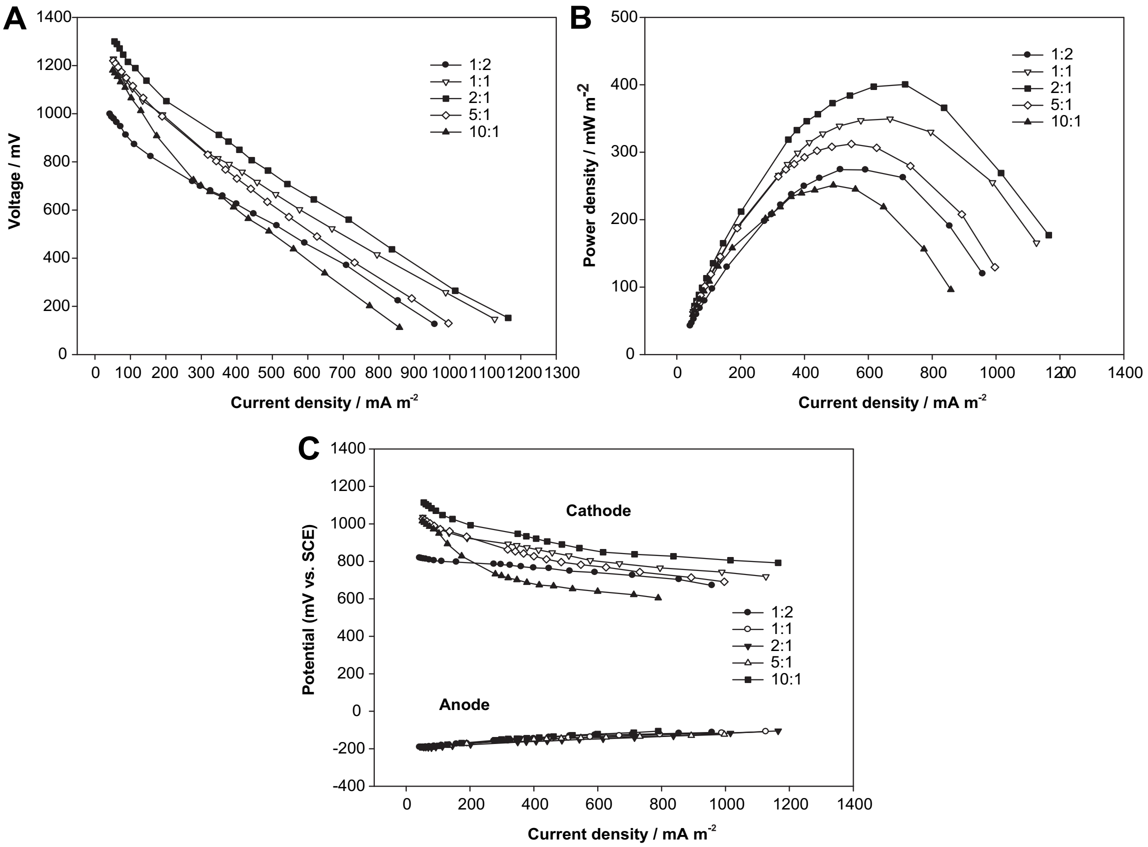

Fig. 7 – The performance of MFCs using $K_2S_2O_8$–Fe^{2+} at different molar ratios as the cathodic reagents. The molar ratios of $K_2S_2O_8/Fe^{2+}$ were 1:2, 1:1, 2:1, 5:1 and 10:1. (A) Polarization curves, (B) Power density curves for the whole MFCs, (C) Polarization curves for the anode and the cathode.

reagent was adjusted by sulfuric acid or sodium hydroxide to the designed values (2.0, 3.0, 4.0, 5.0, 6.0) when the MFCs voltage achieved a steady-state stage. Under acidic conditions the breakdown of persulfate was shown as follow:

$$S_2O_8^{2-} + 2H^+ + 2e^- \rightarrow 2HSO_4^- \quad E^0 = 2.12\ \mathrm{V} \tag{9}$$

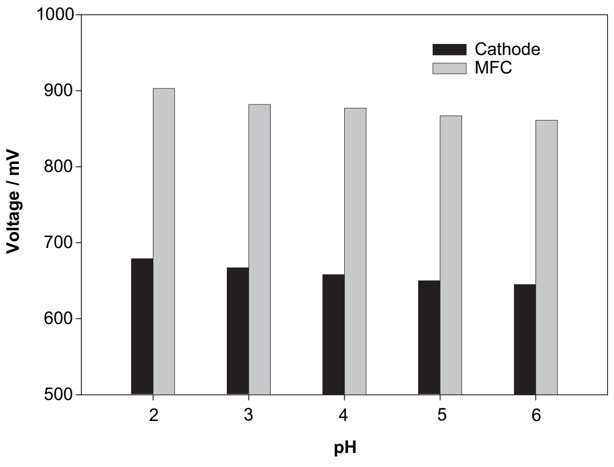

Fig. 8 – The effect of cathode solution pH on voltage output of the MFC with $K_2S_2O_8$–Fe^{2+} system at the molar ratios 2:1.

As mentioned above, cathode potential was a principal factor for increasing MFC performance. The theoretical cathode potential can be described by using Nernst equation based on Eq. (9), where E is the theoretical cathode potential; 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}).

$$E = 2.12\ \mathrm{V} - \frac{RT}{F}\ln\left[HSO_4^-\right] + \frac{RT}{2F}\ln\left[S_2O_8^{2-}\right] - \frac{2\times 2.303\times RT}{F}pH \tag{10}$$

The Nernst equation indicated that the cathode potential would decrease with the increasing of pH. The results in Fig. 8 demonstrated this conclusion, the cathode potential decreased from 679 mV to 645 mV when pH was increased from 2.0 to 6.0. In an oxidizing system, iron activators precipitate to iron hydroxide ($Fe(OH)_3$) or hydrous ferric oxide ($Fe_2O_3\cdot nH_2O$) when pH was above 3.5. Iron hydroxide and hydrous ferric oxide are poor activators for persulfate [31], which would decrease the power generation in MFC. From Fig. 8, we can also see that the MFC using $K_2S_2O_8$–Fe^{2+} was less influenced by pH. As pH was increased from 2.0 to 6.0, voltage output decreased from 903 mV to 861 mV.

4. Conclusions

In the present study, the authors proposed a novel cathodic reagent of $K_2S_2O_8$–Fe^{2+} system in a two-chamber MFC. The

$K_2S_2O_8$–Fe^{2+} system obtained the best performance compared to $K_2S_2O_8$ and H_2O_2–Fe^{2+} system under the same condition. The results stated that the $K_2S_2O_8$–Fe^{2+} system acquired a maximum power density of 401 mW m^{-2}, which was nearly 1.7 times and 2.1 times higher than the MFC with $K_2S_2O_8$ (234 mW m^{-2}) and H_2O_2–Fe^{2+} system (187 mW m^{-2}), respectively. And the cathode potential from $K_2S_2O_8$–Fe^{2+} system was obviously higher than that with $K_2S_2O_8$. The performance improvement can be mainly attributed to the increased oxidation–reduction potentials of $K_2S_2O_8$–Fe^{2+} system, the addition of ferrous ion can enhance the power generation in MFC. By comparing the maximum power density of different $K_2S_2O_8/Fe^{2+}$ molar ratios, it is indicated that MFC with $K_2S_2O_8/Fe^{2+}$ molar ratios 2:1 achieved the best performance. Also, the $K_2S_2O_8$–Fe^{2+} system exhibited excellent stability in cathode solution of different pH values. The voltage output of MFC using $K_2S_2O_8$–Fe^{2+} system was just slightly influenced (from 903 mV to 861 mV) by pH (increased from 2.0 to 6.0). Taken together, the authors believe that the proposed $K_2S_2O_8$–Fe^{2+} system can provide an alternative cathodic reagent for application.

Acknowledgments

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