Microbial fuel cell performance of multiwall carbon nanotubes on carbon cloth as electrodes

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ABSTRACT

Microbial fuel cell (MFC), which can produce electricity during treatment of wastewater, has become one of the emerging technologies in the field of environmental protection and energy recovery. Of all parts of MFC, the electrode materials play a crucial role in the electricity generation. In this study, we investigate the performance of carbon nanotube (CNT) modified carbon cloth electrodes in single-chamber MFC. The MFC is first inoculated with bacteria in wastewater and then its capability of using acetate sodium as fuel is examined. The results show that the MFC with CNT coated onto carbon cloth electrode improves the power density. In this study, the obtained maximum power density is 65 mW m^{-2}, the highest chemical oxygen demand (COD) removal efficiency is 95%, and the maximum Coulombic efficiency is 67%. Compared with other reported studies, the CNT/carbon cloth composite electrode has demonstrated high potential for the use of MFC.

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1. Introduction

Wastewater treatment processes require considerable operation and capital investment due to its high aeration equipment costs and the large volume of wastewater to be treated. Therefore, the conventional sewage treatment systems requiring high energy need to be replaced with alternative treatment technologies that are low-cost and require less energy in operation. In addition, due to global environmental concerns and energy insecurity, there is increasing interest in finding out sustainable and clean energy sources. Microbial fuel cell (MFC) is an emerging technology that converts the energy, released by breaking chemical bonds of organic compounds, into electrical energy through catalytic reactions of microorganisms under anaerobic conditions. The concept of using microorganisms as catalysts in fuel cells was explored in the 1970s [1–2] and the idea of using microbial fuel cells to treat domestic wastewater was presented in 1991 [3]. The main advantage of using MFC is that, by the elimination of the need of exogenous mediator, the cost is reduced [4]. Furthermore, recently microbial fuel cells with enhanced power output have been developed, increasing the possibilities for practical applications [5].

A typical double-chamber MFC consists of two chambers, anode and cathode, separated by proton exchange membrane (PEM).

Microorganisms oxidize the substrate to produce electrons and protons in the anode chamber. Electrons, collected on the anode, are transported to cathode by external circuit and protons are transferred internally to the cathode chamber through the membrane. Thus, a potential difference between the anode and the cathode chambers is produced due to dissimilar liquid solutions. Electrons and protons are consumed during oxygen reduction in the cathode chamber, in which oxygen molecules are usually dissolved in water.

In the year 2004, Liu et al. have investigated the performance of single-chamber MFC in wastewater treatment [6]. The design of single-chamber MFC has several advantages over the traditional double-chamber MFCs. The structure consists of both cathode and anode electrodes placed in a single reaction chamber with PEM directly fused to the surface of the cathode. This type of MFC design allows continuous flow condition during wastewater treatment. Later in the same year, Liu et al. have examined the performances of air-cathode single-chamber MFC individually in the presence and in the absence of PEM and their study demonstrated the possibility of using MFC systems without the expenditure of PEM materials [7]. Furthermore, the air-cathode design allows oxygen molecules to pass through the cathode layer directly and thus reduces the cost in building air exposure equipments.

The performance of an MFC, other than chamber design, is affected by several factors including the microbial inocula, chemical substrate, presence or absence of proton exchange membrane, equivalent internal and external resistance of the cell, solution ionic strength, electrode materials, and electrode spacing [8–11]. As an

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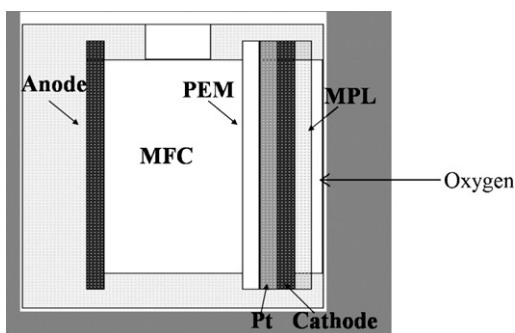*E-mail address:* hytsai@pme.nthu.edu.tw (H.-Y. Tsai).

Fig. 1. Operating principles of a MFC (not to scale).

important part of MFC, the electrode materials play a great role in the electricity generation. To improve the power output of MFC, much research has focused on cathode modification and optimization of bacteria inoculation [11]. However, according to Qiao et al.'s work, modification of anode results in considerable contribution to the overall performance of the MFC [12]. The anode material and its structure can directly affect bacteria attachment, electron transfer and substrate oxidation [13]. To date, porous carbon materials such as carbon cloth, carbon paper and carbon rod are applied in most MFC anodes due to their chemical stability in a microbial inoculum mixture, high conductivity and high specific surface-area. However, they have little electrocatalytic activity for the anode microbial reactions and thus modification on the carbon electrode is the main approach to improve their performance.

Carbon nanotubes (CNTs) have exhibited very promising properties as a catalyst support and they have enhanced catalyst activity in fuel cell applications due to their unique electrical and structural properties [14–16]. CNTs have also served as electrode materials for enzymatic biofuel cells [17–19]. Some works have been done showing the effects of CNTs-modified anode on the MFC [20–21]. Moreover, it has been reported that CNTs made by combustion chemical vapor deposition (CCVD) have no obvious cellular toxicity, which is a desired property for applications with the use of microorganisms [22]. The 3D-MWCNT (multiwall carbon nanotube)-based networks architectures are not only mechanically strong and electrically conductive, but they also contribute considerably to the adhesion and growth of bacterial cells [23].

In this study, we designed a set of mediatorless single-chamber MFCs similar to the design studied by Cheng et al. [24] and examined a new type of electrode architecture by coating high-surface-area CNT onto a carbon cloth, which forms a highly conductive electrode with a high specific surface-area in MFC. Fig. 1 illustrates the MFC system we used in our experiments. In order to evaluate the property and performance of CNT modified MFCs, we have investigated a group of MFC systems with or without the presence of Pt catalyst, PEM or modified anodes.

2. Materials and methods

2.1. CNT purification and preparation of electrodes

Commercial MWCNTs were ultrasonically dispersed in a 3:1 concentrated sulfuric and nitric acid mixture for 3 h. The mixture was set still for 1 h before diluting with de-ionized water. Large particles and agglomerates were removed from the solution by centrifuging.

The centrifuged liquid was carefully decanted, filtered through a polycarbonate membrane (pore diameter 0.8 μm), and washed with de-ionized water until slurry with pH 6–7 was obtained. This slurry was added to a small amount of ethanol, which was sonicated to disperse the CNTs. It resulted in a product which was a stable, dis-

persed solution of MWCNTs ink. A carbon cloth was dipped into the MWCNT ink, removed, and then baked at 150 °C for 1 h to eliminate residual water. This resulted in a thick, black coating of MWCNTs on the carbon cloth's surface.

2.2. Air-cathode MFC construction

The MFCs with different anode and cathode spacing were constructed by using plastic (Plexiglas) cylindrical chambers as its main bodies. The MFC consisted of an anode and a cathode placed on opposite sides of a plastic (Plexiglas) cylindrical chamber with the diameter of 3.5 cm and length of 6 cm. Polytetrafluoroethylene (PTFE) layers were coated on the air-side of the carbon cloth cathode to reduce water loss due to leakage [24].

In this study, ten different types of single-chamber MFCs were constructed to compare the performances of the MFCs. In order to distinguish different types of reactors, we designated the configuration of each reactor using the notation of MFC-X_A, where X is the serial number of cells from 1 to 6 and a presence of additional subscript notation, A, is anode coated with CNT; i.e. MFC-1 is system 1 with regular carbon cloth anode and MFC-1_A is the MFC system with CNT coated anode. Table 1 listed the detailed differences among the MFC system constructions and Fig. 2 illustrated the structures of different types of MFCs used in the experiment.

In the constructed MFC systems, MFC-5 and MFC-6 were used as control sets according to the literature [7]. The cathodes in all the experimental sets (MFC-1–4) were manufactured by coating the CNT directly onto flexible CeTech carbon-cloth electrodes. The MFC-Xs had pure carbon-cloth (without wet proofing) anodes and the MFC-X_As had CNT coated anodes constructed in the same manner as cathodes. In MFC-1s and MFC-2s, 0.5 mg cm^{-2} of Pt catalyst was coated directly onto the CNT coated cathodes. Additionally, MFC-2s and MFC-4s had PEM fused directly to the Pt catalyst layer and CNT layer on the cathode respectively. Copper wire was used to connect the circuit (500 Ω resistor except stated otherwise). MFCs were operated in a temperature-controlled room at 30 °C.

2.3. Microorganisms and substrate

Bacteria presented in wastewater were used to inoculate the MFC for they have been shown to be suitable biocatalysts for electricity production in preliminary experiments. Domestic wastewater (primary clarifier effluent) was obtained from the Bisha Fishing Harbour Wastewater Treatment Plant (Taiwan, ROC) and used as the inoculum. The wastewater had a chemical oxygen demand (COD) of 100–200 mg L^{-1}. Acetate mediums including inorganic nutrient salts (100–500 mg L^{-1}) were also made as the fuel to test the effects of different electrode modifications. The artificial fuel solution contained the following (per liter): 1.5 g NH$_4$Cl, 0.1 g KCl, 4.97 g NaH$_2$PO$_4$·H$_2$O, 2.5 g NaHCO$_3$, 0.1 g CaCl$_2$·2H$_2$O, 0.1 g NaCl, 0.1 g MgCl$_2$·6H$_2$O, 0.1 g MgSO$_4$·7H$_2$O, 0.005 g MnCl$_2$·4H$_2$O, and 0.001 g NaMnO$_4$·2H$_2$O.

2.4. Calculations

Voltage was measured by using a multimeter with a data acquisition system (CHY-48R) and converted to power density, P (W m^{-2}), according to the equation $P = IV/A$, where I (A) is the current, V (V) is the voltage, and A (m^2) is the projected cross-sectional area of the anode. Polarization curve experiments were performed to determine the maximum electricity production generated from sodium acetate. When the voltage output became stable, data were collected to determine the voltage and power generation sustained across a range of current densities obtained by varying the resistance between the electrodes. To obtain the polarization curve and

Table 1
Detailed list of the design of electrodes in each MFC system.

	MFC-1	MFC-1$_A$	MFC-2	MFC-2$_A$	MFC-3	MFC-3$_A$	MFC-4	MFC-4$_A$	MFC-5[a]	MFC-6[a]
Cathode CNT	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	No
Anode CNT	No	Yes	No	Yes	No	Yes	No	Yes	No	No
Pt catalyst	Yes	Yes	Yes	Yes	No	No	No	No	Yes	Yes
PEM	No	No	Yes	Yes	No	No	Yes	Yes	No	Yes

[a] MFC controls constructed according to literature [24].

power density curve as a function of current, external circuit resistances were varied from 10 to 3000 KΩ.

The concentration of organic matter in the MFC was measured as COD by using standard methods. All samples were filtered through a 0.2 μm (pore diameter) membrane filter prior to COD measurements. COD removal was calculated as:

$$E_{COD} = \frac{COD_{in} - COD_{out}}{COD_{in}} \times 100\%, \tag{1}$$

where COD_{in} is the influent COD and COD_{out} is the effluent COD.

The Coulombic efficiency (CE), ε_{cb} (%), is generally defined as the ratio of total coulombs of produced electricity, actually transferred to the anode during the substrate removal process to the maximum possible coulombs, if all substrate removal processes produce current. The total coulombs obtained was determined by integrating the current over time and the Coulombic efficiency for an MFC run in fed-batch mode, ε_{cb} (%) evaluated over a period of time t_b, was calculated as:

$$\varepsilon_{cb}(\%) \frac{M \int_0^{t_b} I \, dt}{FbV\Delta\,COD} \times 100\% \tag{2}$$

where $M = 32$, the molecular weight of oxygen, F is Faraday's constant, $b = 4$ is the number of electrons exchanged per mole of oxygen, V is the volume (in liters) of liquid in the anode compartment, and ΔCOD is the change in COD over time t_b. Detailed information can be found in a previous report [25].

The internal resistance of MFCs operated in batch mode, R_{int}, was calculated from:

$$R_{int} = \frac{OCV - E_{cell}}{I_L} \tag{3}$$

where OCV is the open-circuit voltage of the MFC, I_L is the current under a load and E_{cell} is the electromotive force of the cell. The equation was derived from applying Kirchoff's voltage law to a circuit where a power source was connected to a known load.
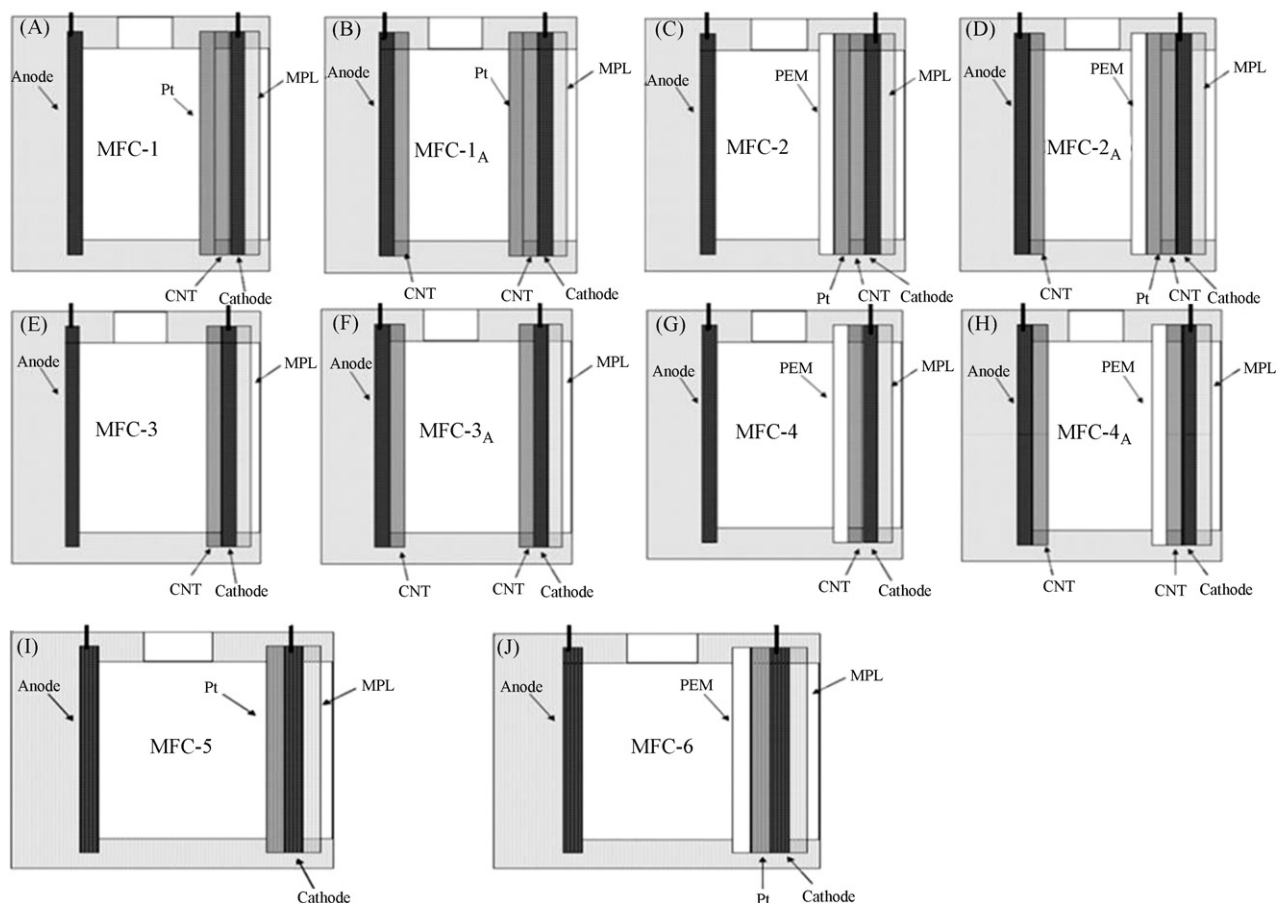
Fig. 2. Schematic diagrams of single-chamber MFCs (A) anode without CNT; cathode with Pt-CNT, (B) anode with CNT; cathode with Pt-CNT, (C) anode without CNT; cathode with Pt-CNT-PEM, (D) anode with CNT; cathode with Pt-CNT-PEM, (E) anode without CNT; cathode with CNT, (F) anode with CNT; cathode with CNT, (G) anode without CNT; cathode with CNT-PEM, (H) anode with CNT; cathode with CNT-PEM, (I) anode without CNT; cathode with Pt, and (J) anode without CNT; cathode with Pt-PEM.
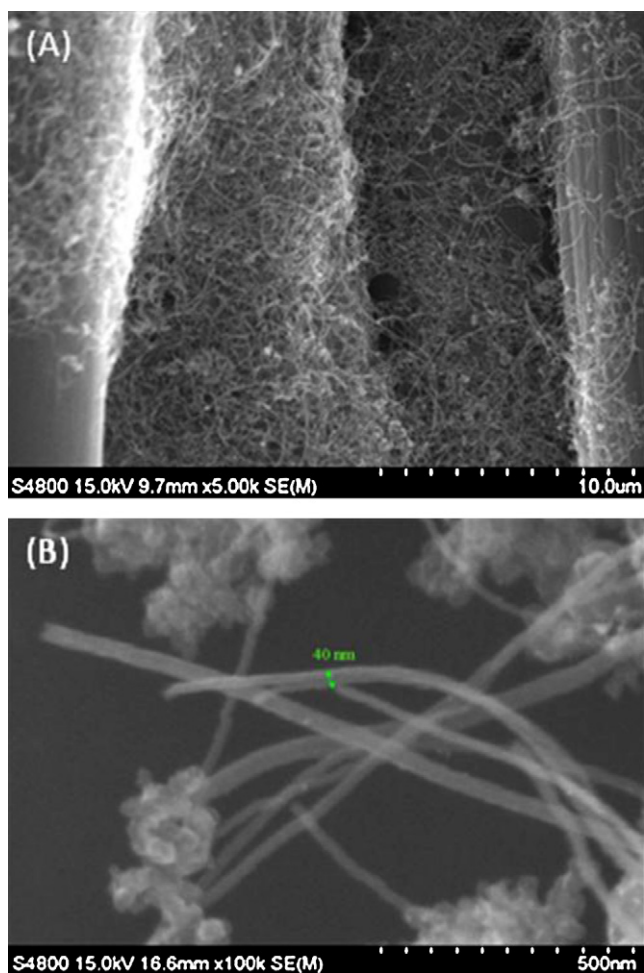
Fig. 3. SEM images of MWCNTs (A) on carbon cloth, and (B) at higher magnification.

Fig. 4. Voltage generation in the (A) MFC-Xs, and (B) MFC-X_As with sodium acetate.

3. Results and discussion

3.1. Morphology of carbon nanotube on carbon cloth

Fig. 3(A) shows the SEM images of long MWCNTs coated on carbon cloth followed by the image of MWCNTs analyzed under higher magnification in Fig. 3(B). When investigating the MFCs with Pt catalyst coated electrodes, it was found that since the dispersion of the MWCNTs cannot yet be precisely controlled, the sprayed Pt particle may just be deposited on the top surface of the high density MWCNTs cluster. Therefore, the property of MWCNTs having high specific surface-area still cannot be totally investigated and utilized until Pt is deposited uniformly on well dispersed MWCNTs. However, compared to the ones without MWCNT functioning as support and carrier for catalyst, the raw surface of carbon cloth is relatively inert and difficult to support particles homogeneously, which often results in the agglomeration of nanoparticles. Thus, although this is beyond the scope of our study, it is important to improve the adhesion through better surface chemical modification and CNT dispersion techniques if Pt catalyst is to be used.

3.2. Electricity generation using sodium acetate

Our preliminary experiments conducted in MFC system demonstrated that electricity could be generated by using wastewater, and the results proved that the needed bacteria had already been present in the wastewater (data not shown). Afterwards, sodium
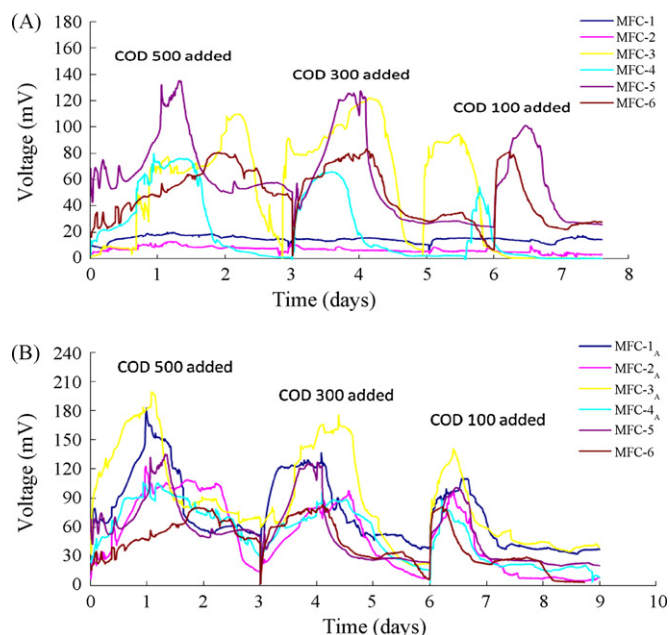
acetate was used as fuel in order to initiate bacteria activity on carbon cloth electrodes. Electricity was immediately generated when sodium acetate (500, 300 and $100\,\mathrm{mg\,L^{-1}}$) was added into the MFC. Here, acetate was provided as the electron donor, and no electron acceptors other than the anode were intentionally provided.

Fig. 4 shows the results of a cycle of voltage generation for reactors fed with different initial sodium acetate concentrations (500, 300 and $100\,\mathrm{mg\,L^{-1}}$). In the cases of anodes without CNT (MFC-Xs), a maximum of $136 \pm 0.5\,\mathrm{mV}$ was obtained from the control set-up (MFC-5) as shown in Fig. 4(A). In the cases of anodes with CNT, MFC-X_As, a maximum of $200 \pm 0.5\,\mathrm{mV}$ was obtained from MFC-3$_A$ (Fig. 4B). Higher concentration resulted in a greater voltage output. The voltage decreased from peak after approximately 2 days due to exhaustion of the sodium acetate.

When the sodium acetate in the reactor was consumed, the voltage and generated current began to decrease. However, the voltage as well as generated current was restored to the maximum level, similar to previous levels, as soon as fresh anaerobic sodium acetate solution was added. These results showed that sodium acetate consumed in the MFC system was coupled to current generation. After the replacement of fresh sodium acetate, the voltage returned to peak voltage. This pattern of change in voltage output was also reproducible in our preliminary experiments for electricity generation using wastewater. When fresh wastewater was added to the fuel cell, the dropped voltage was regained even though no precautions were taken to maintain anaerobic conditions during liquid replacement.

Considering that the suspended bacteria cells in the solution have been removed during the solution replacement, its quick recharge (spans typically 1–3 h in Fig. 4) to its original state after the addition of sufficient substrates could be attributed to the attachment of bacteria cells to the anode. However, a gradual decrease in maximum voltage value was observed during the sequential-batch mode operation of the microbial fuel cell. This might be due to nutrient limitation since sodium acetate was the only substrate that was used. Other possibilities for the decrease were that sodium acetate might be utilized by other contaminant bacteria species from the replacement solution or the attached electrici-
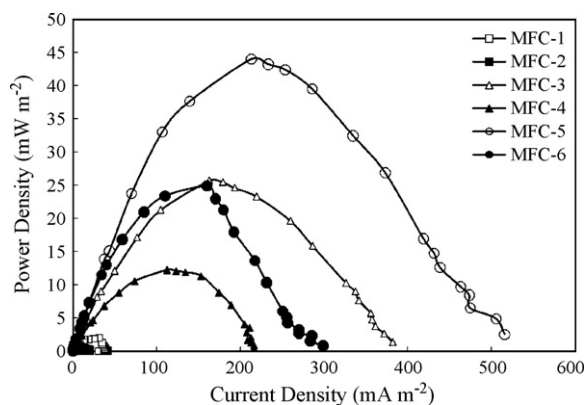
Fig. 5. Power density as a function of current density for acetate (500 mM) in MFC-Xs and controls.
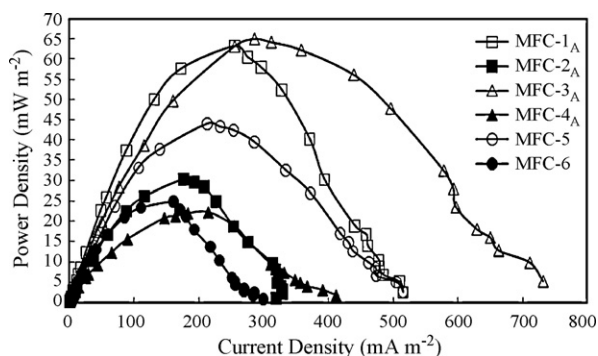

Fig. 6. Power density as a function of current density for acetate (500 mM) in MFC-X_As and controls.

gens on the anode were dying and the total number of electricigens decreased.

3.3. Power density and internal resistance

As shown in Fig. 5, the power density increased with the increase in current density. After reaching a maximum value, it then sharply fell with further increase in current density. This was a typical relationship of output power density against the current density.

In the MFC-X systems, in which anodes had no CNT, the maximum power density generated was 44 mW m^{-2} at current density of 213 mA m^{-2} by MFC-5, whereas the power reached a maximum of 26 mW m^{-2} at a current of 163 mA m^{-2} by MFC-3. In the MFC-X_A systems, in which anodes were CNT coated, the maximum power density generated was 65 mW m^{-2} by MFC-3$_A$ as shown in Fig. 6. In this study, the lowest internal resistance was 884 Ω in MFC-3$_A$ and the values for the other reactor conditions were summarized in Table 2. The open circuit voltage (OCV) was 507 ± 0.5 mV when the CNT was present on the anode. Without the CNT, the OCV

Table 2
Power production and internal resistance of MFCs containing various components (500 mM acetate).

Reactor type	Maximum power (mW m^{-2})		Internal resistance (kΩ)	
	Anode without CNT	Anode with CNT	Anode without CNT	Anode with CNT
MFC-1	2	63	5.2	1.2
MFC-2	0.7	30	9	1.3
MFC-3	26	65	1.2	0.9
MFC-4	13	22	1.6	1.3
MFC-5	44		1.4	
MFC-6	25		2.3	

was 422 ± 0.5 mV. The observed phenomenon that power density was much larger when using the MFC without a PEM than when using the MFC with a PEM was consistent with the previous study [7].

3.4. Effect on the performances of MFCs by CNTs

When examining the effect of pure CNT-coated electrode, MFC-3 and MFC-3$_A$ were compared with MFC-5 (control set) since other sets were not the focus of this study. Furthermore, the performances of voltage output, maximum power density produced and internal resistance obtained from MFC-3 and MFC-3$_A$ systems were much better than those achieved by using other test conditions.

It was observed that the output voltage and power density obtained from MFC-5 were much greater than that obtained from MFC-3. However, the results still indicated that the modification on carbon cloth electrode with CNT was excellent and promising for MFC applications because it provided a satisfactory performance even without the presence of Pt catalyst. Additionally, when examining the performance of the CNT-coated anode in reactors for optimized power generation, it was found that MFCs with CNT-coated anode had voltage output with values approximating 62% higher than those obtained from untreated anode. Also, results showed that maximum power density produced by a MFC-3$_A$ was 65 mW m^{-2} which was an improvement of 250% over the non-CNT-coated anode (26 mW m^{-2} by MFC-3) and 148% over the control set MFC-5 (44 mW m^{-2}).

MFC performance was substantially increased by using new reactor conditions with CNT-coated carbon cloth electrodes. These results of output voltage and the maximum power density demonstrated the effect of CNT. The power density produced by MFC-3$_A$ was significantly higher than that produced by MFC-5 with sodium acetate as the substrate. The cause of differences in maximum power density in these MFC systems was related to the different internal resistances of the systems. In MFC-X_As, it was evident, from the data shown in Table 2, that in the CNT modified air-cathode MFC systems, the internal resistance was reduced and resulted in the observed increase in power density. These results suggested that the specific surface-area of the electrode have been increased by the addition of CNT to carbon cloth. Such increased area was available for increased reduction activity on the cathode surface and increased biofilm formation on the anode surface. Consequently, the combined effect on the electrodes enhanced the electrochemical activity in a MFC. This superior performance of low-cost CNT materials in this study showed that CNTs can be effectively used in MFC systems without the use of Pt catalyst and mediator.

3.5. COD removal and Coulombic efficiency

Both COD removal and power generation were considered in the evaluation of the performance of the MFC. Percentage COD removals at the end of 8 days operation were almost within the range of 75–97% in the MFC-X systems as shown in Fig. 7(A). Variations in percentage COD removal after 9 days of operation for different initial COD levels in MFC-X_A systems were depicted in Fig. 7(B). Percentage COD removal in the MFC-X_As was nearly constant within the range of 85–95% for all initial COD concentrations. Initial power density generated by the MFCs increased with increasing initial COD content as depicted in Fig. 8. Increases in initial power density with increased initial COD were rather low at COD levels below 300 mg L^{-1}, but the power density increased considerably for initial COD levels above 300 mg L^{-1}. These results showed that the MFC could be used in the wastewater treatment process. In the perspective of wastewater treatment, as long as COD removal was accomplished by either electricity generation or other methods, the goal of wastewater treatment is achieved. However,
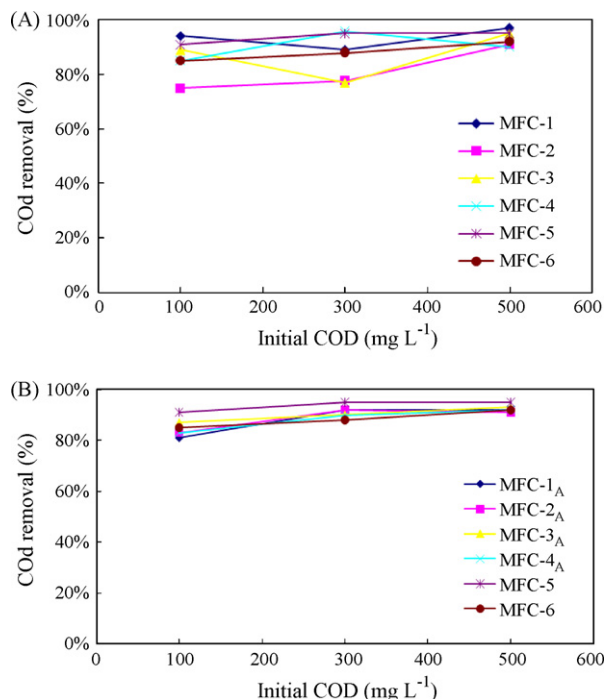
Fig. 9. The Coulombic efficiency of MFC-3 and MFC-3$_A$ as a function of initial acetate concentration (100–500 mM).

Fig. 7. Percentage COD removal for different initial COD levels in the (A) MFC-Xs, and (B) MFC-X_As.

it was found that a large percentage of the organic matter in the wastewater was removed by processes without generating electricity. It is thus important to find methods to increase the fraction of the organic matter that is converted into electricity in order to increase the power density of the system for electricity generation purpose.

Substrate removal was nearly complete (93% COD removal for MFC-3$_A$, 95% for MFC-3) when the voltage of the batch experiments (with 500 Ω resistor) dropped to less than 10 mV for all tests at different initial CODs. The overall Coulombic efficiency was a function of initial COD. CE decreased from 39% to 15% for the MFC-3 and from 67% to 46% for the MFC-3$_A$ when the initial COD was increased from 100 to 500 mg L^{-1} respectively as shown in Fig. 9. A decrease of the Coulombic efficiency with the increase of initial COD was observed. This relationship between the Coulombic efficiency and the substrate concentration was likely a consequence of an increase in the operation time. Slower substrate utilization due to higher COD concentration resulted in more oxygen transferred into the chamber via air-cathode before exhaustion of the substrate. The higher the initial COD, the longer the period of time was needed to fully degrade the substrate. As the time increased, more oxygen could leak into the
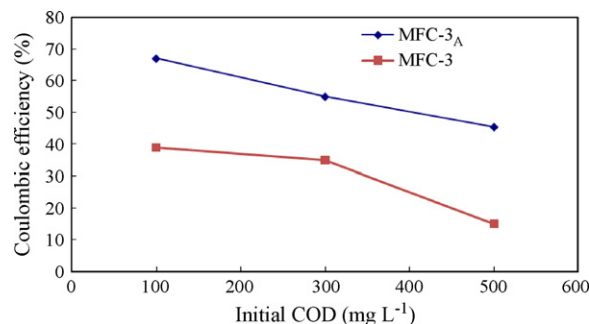
system causing aerobic removal of the substrate, lowering the overall the Coulombic efficiencies. The Coulombic efficiencies obtained in this study were relatively high compared to 81–83% obtained from Chaudhuri and Lovley's work using mixed cultures and glucose [25]. However, the Coulombic efficiencies in other studies have varied widely with the values of 0.04% [10], 3–12% [6], 12% [26], 89% [13]. Possible reasons for the lower the Coulombic efficiencies in other studies include loss of substrate due to diffusion of oxygen through membrane, presence of other electron acceptors (such as NO$_3^-$ or SO$_4^{2-}$), and differences in bacterial yield [7].

4. Conclusion

In this study, we investigated the performance of single-chamber MFC with carbon nanotube/carbon cloth composite electrodes. The MFC system designed for wastewater treatment showed the promising properties of CNT that addition of CNT to carbon cloth electrode greatly enhanced the voltage output, maximum power density and the Coulombic efficiency. Such improvements in the performance allowed the elimination of expenditure on high-cost Pt catalyst and PEM material. Hence, the commercialization of large scale wastewater treating MFC systems can be realized.

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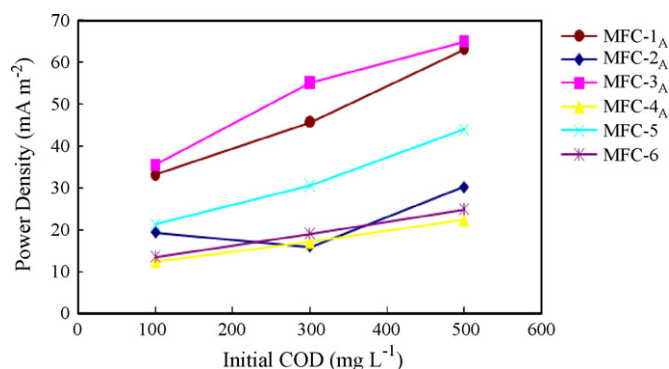[20] T. Sharma, A.L.M. Reddy, T.S. Chandra, S. Ramaprabhu, Int. J. Hydrogen Energy 33 (2008) 6749–6754.

Fig. 8. Variation of power density with different initial CODs.

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