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Treatment of oilfield wastewater using a microbial fuel cell integrated with an up-flow anaerobic sludge blanket reactor

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ABSTRACT

In this study, an integrated system of an up-flow anaerobic sludge blanket (UASB) reactor and a microbial fuel cell (MFC) was used to treat oilfield wastewater at laboratory scale. Results showed that the integrated system performed efficiently in treating the wastewater. The removal efficiencies were higher than 90 and 83% for chemical oxygen demand and NH_3–N, respectively, at 26 h hydraulic retention time. The instrumental analysis indicated that most of the hydrocarbons were satisfactorily biodegraded in the integrated system. This work demonstrated that the UASB and sequential MFC reactor coupled system could be applied to achieve electricity production with simultaneous oily wastewater treatment.

Keywords: Petroleum hydrocarbons; Acidification; UASB; Anaerobic

1. Introduction

Oilfield wastewater is generated after separation from crude oil during the primary separation process, which accounts for the majority of the waste derived from oilfield development. Water content is usually low in early stages of oil production, whereas later its proportion may rise to as high as over 90% [1]. Since oilfield wastewater contains high concentrations of petroleum hydrocarbons, oilfield chemicals, salinity, suspended solids, and heavy metals, it can cause considerable environmental impacts if discharged without effective treatment [2]. Nowadays, the petroleum industry faces a huge challenge in meeting increasingly stringent environmental standards.

During the past decades, various technologies have been developed for the treatment of oilfield wastewater such as membrane filtration [3], reverse osmosis [4], oxidation [5], etc. Compared with physical and chemical processes, biological treatment is a cost-effective and environmentally friendly technique and more compatible with existing plant facilities and operation. Thus, more and more attention has been paid in the treatment of oilfield wastewater using a variety of biological methods such as aerated filter [6], anaerobic baffled reactor [7], anaerobic tank [8], and bio-contact oxidation [9]. To improve the biodegradability of oilfield wastewater, chemical oxidation approaches can be combined with the biological treatment units [10,11].

Based on the combination of biological treatment and electrochemical process, a newly developed technology termed microbial fuel cells (MFCs) has recently drawn extensive research interest owing to its high efficiency, low cost, environmental sustainability, ambient operating temperatures with biologically compatible materials, and added value by-products such as electricity, fuels, and chemicals [12]. Recently, MFCs have emerged as a competitive technology for wastewater treatment. Different real wastewaters,

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including domestic and hospital [13], agricultural [14], animal [15], and oil mill [16] wastewaters, have been successfully remediated using MFCs. As oilfield wastewater contains high concentrations of hydrocarbons and various recalcitrant substances, it would be difficult to remediate the wastewater using MFCs alone. Thus, an integrated system implementing other technologies was necessary for efficient treatment of oilfield wastewater.

The anaerobic hydrolysis/acidification process is known as an effective pretreatment for aerobic treatment of refractory wastewater [17]. Recalcitrant organic compounds can be transformed into degradable substances during the process. Simultaneously, a portion of chemical oxygen demand (COD) can be removed.

In this study, an integrated system of up-flow anaerobic sludge blanket (UASB) and MFC was designed for oilfield wastewater treatment. The UASB stage was designed to enhance biodegradability and organic removal efficiency of wastewater. The MFC stage was to remove partial ammonia and major body of organic pollutants companying with simultaneous electricity generation.

2. Materials and methods

2.1. Oilfield wastewater

Raw wastewater used in this study was collected from a settling tank belonging to a conventional oilfield wastewater treatment plant (Henan, China) and kept at 4°C before use. The characteristics of the wastewater are listed in Table 1 and the GC-MS profile is shown in Fig. 1.

2.2. Experimental equipment

The proposed UASB–MFC system in the present study was composed of one UASB reactor and one MFC unit (Fig. 2) Both the UASB reactor and MFC unit were made of polymethyl methacrylate, in cylindrical shape, with a total volume of 1.35 L (7 cm internal diameter and 35 cm effective height) and an effective volume of 1.15 L. The anode compartment of MFC was filled with 3–5 mm diameter graphite granules as the anode and a copper wire as the current collector. To decrease the metal impurities prior to use, the graphite granule was submerged overnight in 1 M HCl, washed with deionized water, then submerged overnight in 1 mol/L NaOH, and finally washed several times with deionized water [18]. The carbon cloth cathode (30 wt.% wet-proofed) was coated by platinum (0.5 mg/cm^2 Pt) and six diffusion layers [19]. External connection was guaranteed by placing a graphite rod through the anode and cathode compartments. The entire cathode was covered with a thick plexiglass with holes to allow oxygen to reach the cathode. The anode and cathode electrodes were

Table 1
Characteristics of raw oilfield wastewater used in this study

pH	7.3
COD, mg/L	376–425
BOD_5, mg/L	132–168
NH_3–N, mg/L	67–84
TPH, mg/L	32–38
Total phosphorus, mg/L	0.10–0.13
Total nitrogen, mg/L	146–215
Total dissolved solids, mg/L	2,350–2,430

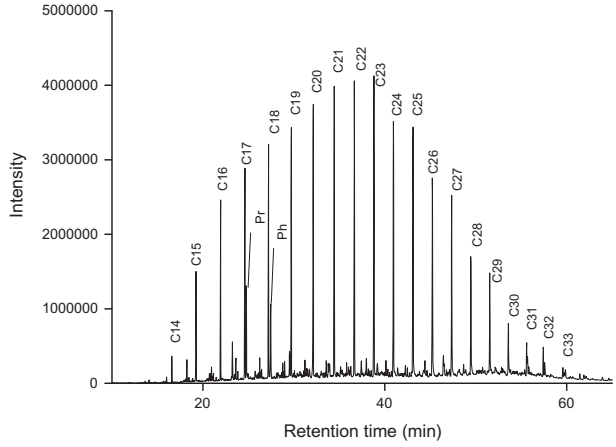

Fig. 1. Total ion chromatogram of the raw oilfield wastewater. Pr, pristane and Ph, phytane.

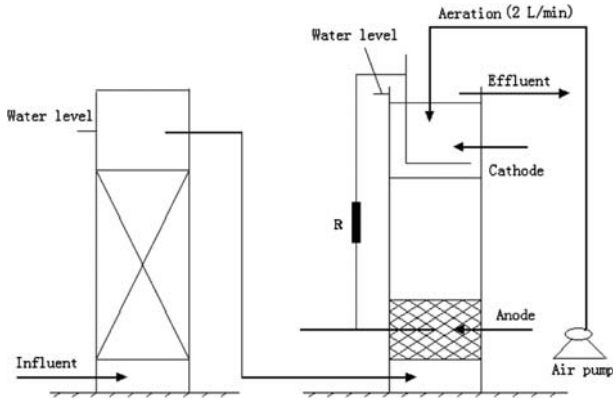

Fig. 2. Schematic diagram of experimental apparatus. *R*: resistance. The network section was the space that the anode was placed.

connected via the copper wire to form a circuit with an external resistor. The aeration rate of the cathode compartment was controlled using aquarium air pumps.

2.3. Inoculum, medium, and operation

The UASB reactor was inoculated with 0.25 L well developed anaerobic granular sludge (MLTSS = 32.8 g/L and MLVSS = 18.6 g/L) obtained from a refinery wastewater treatment plant located in Jiangxi Province, China. The anode of MFC was inoculated with 100 mL of the mixture of the above anaerobic sludge and an aerobic activated sludge at a volume ratio of 2:1. The oilfield wastewater was supplemented with $(NH_4)_2SO_4$ and K_2HPO_4 to give a final COD:N:P ratio of 100:10:1 [20], and then used to feed the integrated system.

The UASB and MFC reactors were both operated in a continuous flow mode for 60 days. To investigate shocking resistance, the system was operated at varied hydraulic retention time (HRT). Three total HRTs (40, 26, and 12 h) were applied to the integrated system. The respective HRT was identical for each reactor (20, 13, and 6 h), since the two reactor has the same effective volume. As the operating temperature has a relatively small effect on the performance of MFCs [21], the UASB–MFC was operated at 35 ± 2°C. All experiments were carried out in triplicate.

2.4. Analytic methods

Measurement of COD was based on digestion with potassium dichromate in concentrated sulfuric acid for 2 h at 150°C [9] and the interference by chloride ions was eliminated by precipitating the chloride ions using $HgSO_4$ reagents. BOD_5 was determined after five days at 20°C in the dark in a thermostated incubator, by measuring the oxygen concentration. Ammonia nitrogen was measured by Nessler's reagent colorimetry [22]. The volatile fatty acids (VFA) concentration was analyzed by bicarbonate alkalinity and the acidification efficiency was calculated according to Zhang et al. [23]:

$$\text{Acidification efficiency} = [(\text{VFA}_e - \text{VFA}_i)/\text{COD}_i] \times 100 \quad (1)$$

where VFA_e and VFA_i are the VFA concentration present in the effluent and influent of the UASB reactor, respectively. COD_i is the COD concentration in the influent of the UASB reactor.

The wastewater sample was extracted by liquid–liquid technique with dichloromethane three times. The three extracts were combined and condensed to 1 mL in a rotary evaporator and then fractionated by silica-gel column chromatography to separate saturate and aromatic fractions according to Bastow et al. [24]. The measurements of *n*-alkanes were performed on an Agilent7890-5975c gas chromatograph (GC) equipped with a model 5975 mass selective detector (MSD; SIM mode) and a DB5-MS (60 m × 0.25 mm × 0.25 μm) capillary. The following temperature program was used for *n*-alkane measurements: initial temperature of 40°C for 2 min; and then heating to 300°C at 3°C/min and hold 55 min. The carrier gas was helium at 37 kPa (1.5 mL/min). The 16 priority polycyclic aromatic hydrocarbons (PAHs) were analyzed also using the same GC-MSD equipped with Thermol Thermo Scientific TRACE TR-5MS GC Column (30 m × 0.25 mm × 0.25 μm) with helium as carrier gas (1 mL/min). The analytical conditions were: initial temperature of 70°C for 1 min; and heating to 260°C at 10°C/min and hold 4 min; and then heating to 300°C at 5 °C/min and hold 4 min. Hydrocarbon Window Defining Standard C_8-C_{40} was purchased from AccuStandards Inc. (New Haven, CT, USA) and PAH Mix was from Sigma-Aldrich Co. Ltd (St. Louis, MO, USA). The concentrations of the following 16 priority PAHs were determined: naphthalene (Nap), acenaphthene (Ane), acenaphthylene (Any), fluorene (Fle), phenanthrene (Phe), anthracene (Ant), pyrene (Pyr), fluoranthrene (Fla), benzo[ghi]perylene (Bpe), benz[a]anthracene (Baa), chrysene (Chr), benzo [a]pyrene (Bap), benzo[b]fluoranthene (Bbf), benzo[k] fluoranthene (Bkf), indeno[1,2,3-cd]pyrene (I1p), and dibenz[ah]anthracene (Daa).

2.5. Electrochemical monitoring

The voltage of the MFC was measured using a personal measurement device (PMD-1208LS, Measurement Computing Corporation, USA), and the data were recorded by a personal computer every 5 min. Current density (mA/m^2) was calculated as $i = V/(RA)$, where V (mV) is the voltage, R (Ω) is the external resistance, and A (m^2) is the projected surface area of the cathode electrode. Power density (mW/m^2) was obtained according to $p = 10\ iV$ (in which 10 is used for units conversion). Polarization data were collected by changing the external resistance (varied from 10 Ω to 10 kΩ) by means of a variable resistor box during the stable power production stage of the experiment [25]. The maximum power density was determined using the polarization curve obtained by altering the external resistance from 10 kΩ to 10 Ω and then back again to 10 kΩ.

2.6. Data analysis

All analyses or measurements, unless otherwise explained, were repeated three times. Data reported were taken from three repetitions of each experiment ($n = 3$).

3. Results and discussion

3.1. Performance of UASB

In this study, the UASB reactor acted as a hydrolysis/acidification system. It has been reported that anaerobic hydrolysis/acidification process is an effective pretreatment for aerobic treatment of refractory wastewater [17,26]. Changes in the influent and effluent COD from the UASB reactor are shown in Fig. 3 (a). At day three, the effluent COD concentrations were low to the level of 352 mg/L, which only resulted in 16.1% of COD removal as shown in Fig. 3 (b). Then the effluent COD concentrations decreased gradually. The effluent COD concentrations were kept at <250 mg/L when HRT decreased from 20 to 13 h. When HRT decreased further from 13 to 6 h, the COD removal decreased significantly, indicating that high organic loading results in lower organic removal percentage. At HRT 20, 13, and 6 h, the average COD removal efficiencies in the UASB reactor were 33.8, 35.5, and 19.1%, respectively.

Fig. 3(b) shows the relationship between acidification efficiency and HRT during hydrolysis/acidification treatment. In general, greater acidification efficiency corresponded to higher COD removal efficiency. The maximum acidification efficiency was obtained at HRT 13 h. This trend was similar with that of the ratio of BOD_5/COD as shown in Fig. 3(c). A short HRT (6 h) would result in a reduction in the acidification efficiency and BOD_5/COD ratio, due to the incomplete hydrolysis of the refractory organics at shorter HRT [27]. The BOD_5/COD ratio is an extensively used biochemical index for quantifying biodegradability, and the wastewater with the ratio >0.4 can be considered with high biodegradability [11].

Recently, various novel biological approaches were applied in treating oilfield wastewater. Lu and Wei [17] used a zerovalent iron/EDTA/air system to pretreat oilfield wastewater containing polyacrylamide, followed by activated sludge treatment. Total removal efficiencies of 97 and 92% were obtained for TPH and COD, respectively. Dong et al. [28] prepared a suspended ceramic carrier, which was used to feed moving bed biofilm reactors to remediate oilfield produced water. Ji et al. [7] used an anaerobic baffled reactor to treat heavy oil produced water, achieving average COD and oil removals of 65 and 88%, respectively, which was higher than that in our study. This may be ascribed to that longer HRTs (144–60 h) were applied by Ji et al. [7] than in our study (6–20 h).

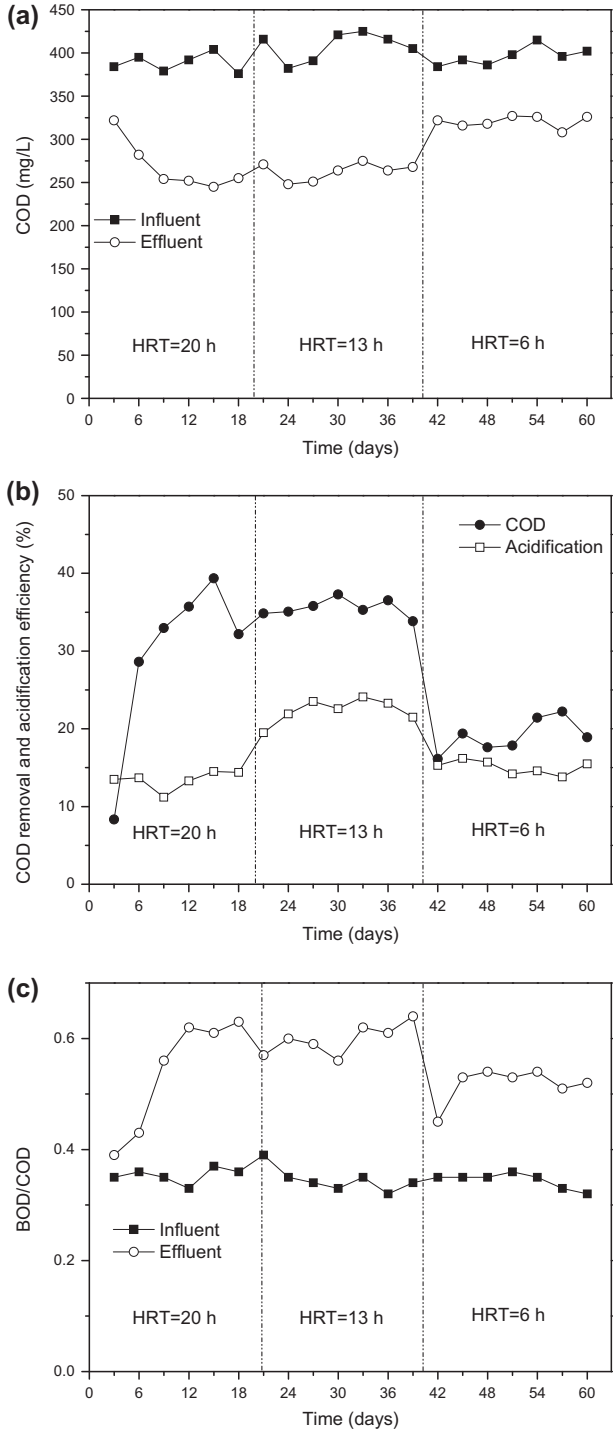

Fig. 3. (a) Time course of COD changes in the influent and effluent from the UASB reactor; (b) time course of COD removal and acidification efficiency in the UASB reactor; and (c) the ratio of BOD_5/COD in the UASB reactor.

Liu et al. [8] used an anaerobic tank to treat oilfield produced water, during which approximately 80 mg/L COD (30%) and 7 mg/L TPH (25%) were removed at initial concentrations of 270 and 28 mg/L for COD and TPH, respectively. In general, a very high HRT is needed for obtaining acceptable treatment efficiency when anaerobic approaches are used for the treatment of oilfield produced water. Actually, anaerobic process is usually suitable for a pretreatment stage prior to aerobic treatment.

3.2. Performance of MFC

3.2.1. COD removal efficiency

The COD removal efficiency of the MFC stage and the integrated system during the 60-day operation is shown in Fig. 4. When respective HRT was controlled at 20 h, the COD removal efficiencies were 88.5–93.5% and 53.2–60.4% for the UASB-MFC and MFC system, respectively, except for the low values at day three. Compared with Fig. 3(b), it can be found that the COD removal efficiency of MFC was obviously higher than that of the acidogenic UASB reactor. With HRT decreasing from 20 to 13 h, the efficiency of the MFC reactor was not influenced significantly. Even at HRT 6 h, the COD removal efficiencies of 42.6–48.0% and 58.7–67.2% were obtained for the MFC reactor and the UASB–MFC system, respectively. It indicated that the MFC reactor had a greater ability than the UASB unit to resist organic shock loading. It has been reported that MFC may maintain its performance in the aspect of COD removal without any process inhibition [29].

3.2.2. NH_3–N removal efficiency

High removal of ammonia–nitrogen (NH_3–N) was obtained in MFCs by some researchers [30,31]. In this study, the MFC stage was found to be effective at removing ammonia. As can be seen from Fig. 5, the maximum total ammonia removal efficiency by the integrated system reached 83.4% at day nine (Fig. 5). However, the UASB unit had no effect on ammonia removal. The NH_3–N concentration in the UASB effluent was even higher than that of the influent. This is attributed to the ammonification reactions under anaerobic conditions in the UASB reactor.

Changes in ammonia content depend on the available microbial biomass and the transformation of nitrate, nitrite, and organic nitrogen into ammonia. On the one hand, nitrogen was consumed by the microorganisms for self-sustenance. On the other hand, part of organic nitrogen was transformed to inorganic nitrogen, meanwhile nitrate and nitrite could be transformed to ammonia under ammonification conditions, causing an increase of ammonia in the effluent. Some of bacteria like *Escherichia coli*, *Citrobacter sp.*, *Klebsiella sp.*, and the group *Enterobacteriaceae* are capable of dissimilating nitrate or nitrite to ammonia [32]. Denitrification may also occur in the UASB reactor. The above results suggested that the ammonification community had advantages over the denitrification community. In the MFC stage, ammonia losses during electricity generation were partly due to ammonia volatilization with conversion of ammonium ion to the more volatile ammonia species as a result of an elevated pH near the cathode (where protons are consumed). In addition, biological nitrification could occur in the cathode compartment of the MFC, resulting in the removal of ammonia.

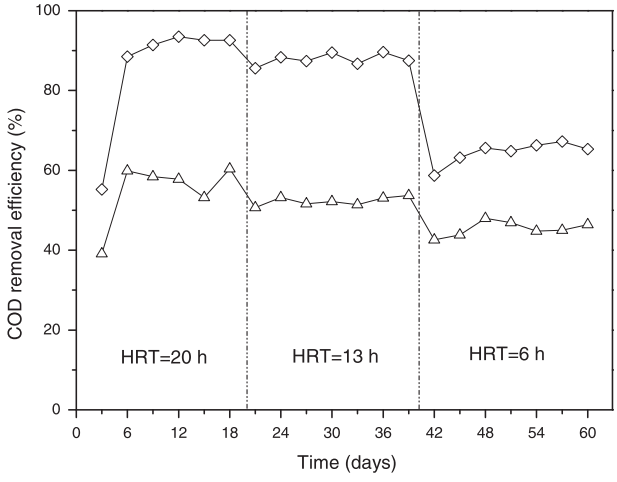

Fig. 4. COD removal efficiencies by UASB–MFC (◇) and MFC alone (Δ).

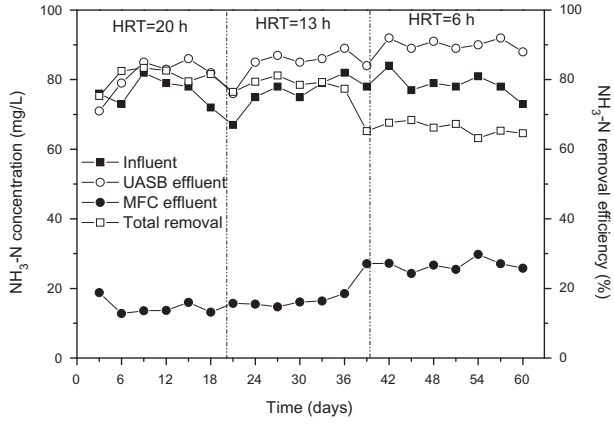

Fig. 5. Time course of ammonia removal during the 60 days operation.

In this study, pH was continuously monitored. We found that the influent pH was stable at around 7.3 and the effluent pH was kept at 6.5–6.7. The pH was within the range required for good biodegradation. Hence, no pH adjustment was conducted in this study.

An integrated system of two-stage MFCs and immobilized biological aerated filters was used by Cheng et al. [16] to treat palm oil mill wastewater at laboratory scale. The results showed that the final effluents COD and NH_3–N could be reduced to below 350 and 8 mg/L, with removal efficiencies higher than 96.5 and 93.6%, respectively. Hence, the integration of MFC and other treatment technologies for wastewater treatment promises a good application prospect.

3.3. Electricity generation

HRT is an important parameter in wastewater treatment, determining the content of effluent substrate and dissolved oxygen in the MFC. Fig. 6 displays the polarization curves obtained for the MFC stage at different HRTs. As shown, the maximum power density reached 93 mW/m^2 when HRT was 13 h. Increasing HRT from 13 to 20 h decreased the substrate concentration in the MFC, which decreased the voltage output from 383 to 254 mV. Decreasing HRT to 6 h increased the substrate concentration in the MFC, which caused a decrease in power density from 93 to 76 mW/m^2. Hence, the maximum electricity generation was obtained at HRT 13 h and a higher or lower HRT would result in decreased power output. In general, the voltage output of MFC could be increased under higher substrate load [29]. With the increase in HRT, more substrate could be utilized by the micro-organisms, resulting in the enhancement of electricity output. After treated in the UASB reactor, the organic molecule was broken to smaller ones, which became better fuels for the MFC. However, once HRT was too high, acidification efficiency would decrease and resulted in a reduction in the amount of easily utilized substrates, thus the electricity generation decreased. This trend was similar with that of the COD and ammonia removal. Relatively high COD and ammonia removal was achieved at HRT 13 h. Based on the result of the effect of HRT on power generation and removal of COD and ammonia, 13 h was chosen as the optimal respective HRT.

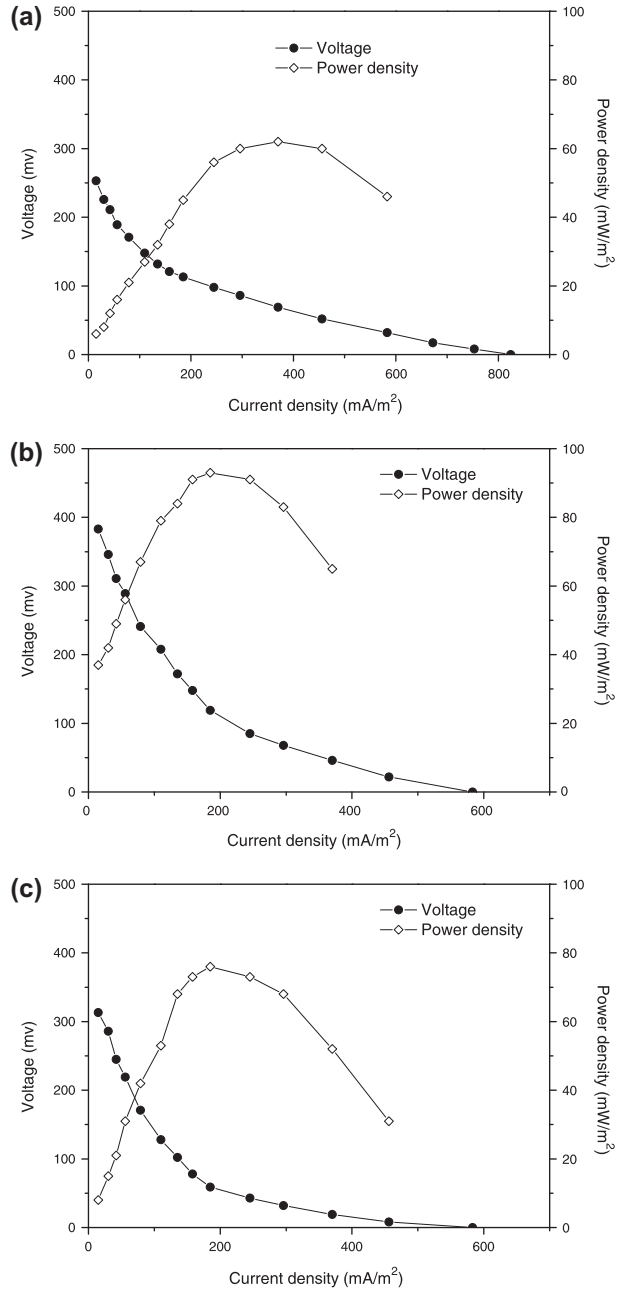

Fig. 6. Polarization curves of MFC (a) at HRT 20 h, day 15; (b) at HRT 13 h, day 33; and (c) at HRT 6 h, day 51.

3.4. Degradation of petroleum hydrocarbons

The *n*-alkanes were detected from C_8 to C_{40} with main contents of C_{13}–C_{32} (Table 2). Approximately 43.5% of total *n*-alkanes were biodegraded in the UASB stage. Biodegradation of hydrocarbons under anaerobic conditions is drawing more and more researching interests. Under anaerobic conditions, electron acceptors, such as nitrates, are utilized for microbial respiration and during this process, hydrocarbons are oxidized to intermediate molecules and eventually CO_2, while terminal electron acceptors are reduced.

Table 2
Concentrations (ng/mL) of individual hydrocarbon at day 33, HRT = 13 h

Hydrocarbon		Influent	UASB effluent	MFC effluent
n-alkanes	$C_{8–12}$	116	76	8
	$C_{13–16}$	2,853	1,420	182
	$C_{17–20}$	7,620	5,360	435
	$C_{21–24}$	7,860	4,310	464
	$C_{25–28}$	4,740	2,163	297
	$C_{29–32}$	3,630	1856	314
	$C_{33–40}$	2,450	1,352	134
	Sum	29,270	16,540	1,834
PAHs	Nap	86.5	55.3	11.4
	Any	11.2	6.3	0.6
	Ane	14.5	8.3	1.2
	Fle	126.6	65.3	12.5
	Phe	486.5	342.7	48.3
	Ant	19.3	8.2	3.7
	Fla	25.7	11.6	5.3
	Pyr	42.8	16.5	6.2
	Baa	12.5	3.2	1.4
	Chr	135.8	62.6	8.7
	Bbf	42.4	16.8	3.6
	Bkf	5.2	2.4	0.8
	Bap	21.6	5.7	2.1
	I1p	1.2	0.6	0.2
	Daa	3.4	1.6	0.6
	Bpe	6.5	2.4	1.3
	Sum	1,042	610	118

Table 2 also lists the concentrations of 16 priority PAHs in the influent and effluent streams. The Nap, Fle, Phe, and Chr were the four dominant constituents, representing 8.3, 12.1, 46.7, and 13.0% of the total 16 PAHs detected in the influent oilfield wastewater. Their respective removal efficiencies were 36.1, 48.4, 29.6, and 53.9% for the UASB reactor and 50.8, 41.7, 60.5, and 39.7% for the MFC reactor. The removal efficiency of the 16 PAHs was 41.5 and 40.6% for the UASB and MFC reactors, respectively. This indicated that both reactors removed PAHs effectively. The higher removal efficiency of PAHs in the UASB than that in the MFC may be attributed to that a main portion of easily biodegradable compounds had been transformed in the UASB reactor, thus the remaining substances entering into the MFC would be more refractory.

It has been demonstrated that a number of PAHs containing four and more rings can be degraded anaerobically [33]. Additionally, it was found that biodegradation of hydrocarbons can be enhanced in MFCs. Morris and Jin [34] successfully used a single-cell MFC to enhance biodegradation of petroleum hydrocarbons in groundwater under anaerobic conditions. Morris et al. [35] demonstrated that anaerobic biodegradation of diesel range hydrocarbons was significantly enhanced in an MFC (82% removal) as compared to an anaerobically incubated control cell (31% removal) over 21 days at 30°C.

The results obtained in this study demonstrated that MFC technology may be useful for bioremediation of petroleum-contaminated wastewater. An MFC design would basically cause an indirect oxidation of petroleum compounds by transferring electrons from anaerobic degradation in the anode to a cathode exposed to oxygen. Additionally, a variety of anaerobic bacteria are capable of passing electrons onto a solid-state electrode and, therefore, generating power in an MFC during regular cellular metabolism. Various electron transport mechanisms have been identified, including direct transport, transport via chemical mediators (produced by microbes or added artificially), and through a cellular nanowire structure formed in certain microbial species [34].

4. Conclusions

In this study, an integrated system of UASB and MFC has been found to perform efficiently in treating oilfield wastewater. Results showed that the optimal HRT for both wastewater treatment and electricity production was 26 h for the integrated system. In the integrated system, COD and NH_3–N removal efficiencies were higher than 90 and 83%, respectively, at 26 h HRT. Compared with the professional emission standard of petrochemical industry of PR China (GB4287-92), the effluent concentrations of NH_3–N and COD in the integrated system could satisfy grade one (NH_3–N < 20 mg/L, COD_{Cr} < 100 mg/L) at HRT of 40 and 26 h, and grade two (NH_3–N < 30 mg/L, COD_{Cr} < 120 mg/L) at HRT of 12 h.

Results from GC-MS analysis demonstrated that *n*-alkanes and PAHs could be biodegraded under both aerobic and anaerobic conditions. The results of this study indicated that the integrated system could be of potential in practical application for oilfield wastewater remediation, since electricity could be generated from MFCs during treatment of wastewater.

Acknowledgments

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Yangtze University, for the assistance in the instrumental analysis.

Abbreviations

MFCs	—	microbial fuel cells
COD	—	chemical oxygen demand
BOD_5	—	five-day biochemical oxygen demand
TPHs	—	total petroleum hydrocarbons
UASB	—	up-flow anaerobic sludge blanket
PAHs	—	polycyclic aromatic hydrocarbons
VFA	—	volatile fatty acids
MLTSS	—	mixed liquor total suspended solids
MLVSS	—	mixed liquor volatile suspended solids

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