Microbial fuel cell for nutrient recovery and electricity generation from municipal wastewater under different ammonium concentrations

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Abstract

In the present study, a dual-compartment microbial fuel cell (MFC) was constructed and continuously operated under different influent concentrations of ammonium-nitrogen (5 to 40 mg/L). The impacts of ammonium on organics removal, energy output and nutrient recovery were investigated. Experimental results demonstrated that this MFC reactor achieved a CDO removal efficiency of greater than 85%. Moreover, excess ammonium concentration in the feed solution compromises the generation of electricity. Simultaneously, the recovery rate of phosphate achieved in the MFC was insignificantly influenced at the wider influent ammonium concentration. In contrast, a high concentration of ammonium may not be beneficial for its recovery.

Keywords: Microbial fuel cell; Phosphate recovery; Ammonium recovery; Ammonium concentration effect.

1. Introduction

Given the current shortage supply in the phosphorus (P) and high costs associated with the industrial nitrogen (N) production for producing chemical fertilizers (Li et al., 2018; Ye et al., 2018), it is essential to intensively reuse nutrient, in order to ensure food security. Apart from this, nutrient recovery from wastewater could also mitigate the environmental footprint which is resulted from the accumulation of such nutrients (Ye et al., 2017). In recent years, various nutrient recovery systems have been widely studied and developed in terms of technology, source and application (Desloover et al., 2015; Kataki et al., 2016; Pedizzi et al., 2017; Yan et al., 2018). More importantly, these nutrient recovery systems are becoming more competitive when compared to the traditional production technologies of reactive N (Haber-Bosch) and P (phosphate rock mining), especially considering the market value of around € 1.0/kg·N and € 1.9/kg·P (De Vrieze et al., 2016; Desmidt et al., 2015).

Municipal wastewater exists in large quantities and its treatment costs are high with 3% of the world's electricity being consumed (Li et al., 2015). Consequently, it is essential to optimize energy consumption in the urban wastewater treatment whilst it is suggested that recovering valuable resources such as phosphate and ammonium from sewage can increase the economic feasibility of the treatment system. The energy optimization and nutrient recovery from domestic wastewater would make the treatment process more sustainable since this: (1) reduces energy requirements; (2) reduces the nutrient load into the natural environment and helps prevent environmental issues from becoming worse; (3) satisfies the increasingly strict government regulations about discharge responsibilities; and (4) generates valuable products and thereby creates

another revenue stream. Thus, the selection of cost-effective technologies which are applied to make nutrient recovery possible from domestic wastewater is critical.

Microbial fuel cell (MFC) is a possible technology strategy for realizing this goal. MFC is an example of a bioelectrochemical bioreactor system and has the ability to directly convert organics contained in wastewater into electrical energy (Bhande et al., 2019; Pareek et al., 2019; Sravan et al., 2017). A traditional MFC reactor consists of an anode compartment, cathode compartment and separator (often cation-exchange membrane [CEM]). Theoretically, anaerobic microbes in the anode chamber of the MFC could be utilized to catalyze the anode reactions. In this case, the biodegradable chemical oxygen demand (COD) could be transformed into protons and electrons by using electrochemically active bacteria (EAB), which eventually forms an electrical field on the anode (Kim et al., 2015). Moreover, the generated electrons are transported to the cathode electrode and then reduced by electron acceptor (often air) to complete the electrical loop.

In MFC studies, the cathode reaction could lead to a high pH zone in the cathode chamber in double-chamber MFCs. Subsequently, this offers a possibility for MFCs to recover ammonium and phosphate through chemical precipitation in wastewater treatment (Ichihashi and Hirooka, 2012; Yan et al., 2018). Furthermore the electricity being generated in the MFC could offset the energy costs in the nutrient recovery system, while the membrane fouling potential of CEM could be reduced. What makes this possible is the current field between the anode compartment and cathode compartment (Wang et al., 2013).

Recently, many researchers have developed MFC reactors for the recovery of nutrients, including different configurations and sources (Cusick and Logan, 2012;

Hirooka and Ichihashi, 2013; Ichihashi and Hirooka, 2012; Nancharaiah et al., 2016; Ye et al., 2019b; Zang et al., 2012). It was reported that ammonium ions exert cytotoxic impacts on the microbial community (Hansen et al., 1998; Müller et al., 2006). The possible reason for this is two-fold: (a) the activity of cytosolic enzymes could be detrimentally influenced by un-ionized NH_3; and (b) the hydrophobic NH_3 molecules passively diffuse into the cell and are then converted into the $NH4^+$ because of the intracellular pH conditions. Here the ammonium accumulation, in turn, results in the inhibited impacts on the cell by altering intracellular pH conditions (Kadam and Boone, 1996). Apart from this, the bacteria may be dehydrated at high ammonia salt levels due to the changes in the osmic pressure (De Baere et al., 1984). However, only a few relevant studies have been published and evaluated MFCs' ability to inhibit ammonia, including power output and nutrient recovery.

In this paper, a double-compartment MFC was built as described in another recent study (Ye et al., 2019b), and it proved its ability to recover ammonium and phosphate via chemical precipitation from synthetic municipal wastewater. The possible advantages for the double-chamber configuration to recover nutrients include: firstly, the double-chamber MFC could separate the anolyte and catholyte when compared to the air-cathode MFC, which could facilitate the pH elevation of catholyte and the later nutrient recovery by chemical precipitation; and secondly, multi-compartment MFCs demonstrated greater operational complexity than the double-chamber MFC. The present study aimed to explore the influence of influent ammonium concentrations (5-40 mg·NH_4^+-N/L) on electricity generation, COD removal and recovery of nutrients in the MFC. This investigation was conducted over a long-term operation, in order to verify the resistance of microbes to the presence of ammonium.

2. Materials and methods

2.1 MFC design and setup

The MFC design in the present study was assembled according to a previously studied MFC (Ye et al., 2019b). In this work, the MFC consisted of an anode chamber and cathode chamber with 305 mL-working volume of each chamber. The compartments are separated by a CEM membrane (CMI7000, Membranes International Inc., USA). The chambers and CEM were sandwiched together with silicon gaskets to prevent leakage of anolyte and catholyte. A cylinder-shaped graphite felt served as the anode electrode (Sanye Carbon Co. Ltd., Beijing, China) with a thickness of 6 mm and diameter of 30 mm. A carbon-fiber brush (length 30 mm, diameter 30 mm) acted as the cathode electrode.

2.2 MFC operation

The MFC's anode chamber was inoculated via anaerobic sludge from the Cronulla wastewater treatment plant (Greenhills Beach, New South Wales, Australia) which has a treatment capacity of 5.3×10^4 m^3/d. In order to separate the liquid and solids, the anaerobic sludge was moved to the laboratory and then left for 24 h as is. After that, the anaerobic sludge was transferred to the anode chamber as the inoculum in the MFC. As for the remaining sludge, it was stored in a fridge at −5 °C to avoid changes in the biological community. In the present study, the double-compartment MFC was operated under continuous mode at a fixed temperature of 22±2 °C using an external resistance of 1000 Ω throughout these experiments. It should be noted here that the experiments did not commence until the saturated current generation of the MFC was confirmed. Furthermore, the anode compartment and cathode compartment were hydraulically connected, in which the anode effluent served as the influent of the cathode chamber.

Fresh anolyte (i.e., synthetic domestic wastewater) was constantly supplied to the anode chamber at 0.35 mL/min through a peristaltic pump (Model 77202-60, Masterflex, Illinois, United States). The composition of artificial municipal wastewater used in the present study included: 300 ± 15 mg/L of COD, 4.6 ± 0.5 mg/L of KH_2PO_4, 5.4 ± 0.5 mg/L of $MgSO_4 \cdot 7H_2O$, 0.4 ± 0.01 mg/L of $CaCl_2 \cdot 2H_2O$, 32 ± 1.0 mg/L of Yeast and 0.61 mL of trace nutrients per litre of distilled (DI) water (Ye et al., 2019a). The pH of feed solution was maintained at 7.00 ± 0.02 through using NaOH and HCl solutions. In addition, the stepwise increasing concentration of NH_4^+-N from 5 to 40 mg/L in the anolyte was obtained by adding ammonium chloride (NH_4Cl). On the other hand, the distilled (DI) water was utilized as the catholyte with air supply throughout the study, in order to focus solely on anode performance. Further details about the MFC operation are summarized in Table 1. Each continuous experiment was conducted in duplicate using parallel columns.

2.3 Calculation

The current density was obtained according to Equation (1) through the combination of Ohm's law and Joule's law.

$$P_A = \frac{U^2}{RA} \quad (1)$$

where P_A (mV/m^2) is the current density; U (mV) is the cell voltage; R (Ω) is the resistance; and A (m^2) is the surface area of anode electrode (in the present case on both sides).

Another important parameter is coulombic efficiency (CE) which could represent the efficiency of the recovery of electrons (see Eq. [2]) (Logan, 2008). As shown in Equation (2), Logan et al. (2006) believed that CE is a ratio representing the measured

current generation and the theoretically possible electron generation potential based on substrate oxidation:

$$C_E = \frac{8\int_0^t Idt}{F\Delta CODV} \times 100\% \quad (2)$$

where C_E (%) is the coulombic efficiency; t (d) is the operation time of MFC; I (mA) is the current produced by the MFC over a certain period of time; F (96485 C/mol) is a constant of the Faraday; ΔCOD (mg/L) is the COD reduction through the MFC at a certain period of t; and V (mL) represents the anode compartment's working volume of the MFC.

2.4 Chemical analysis

During the experiments, daily samples were collected from the anode and cathode compartments of the MFC and then employed to identify the changes in the solution pH, COD, NH_4^+-N and PO_4^{3-}-P. Before being utilized to measure the solution pH, the pH meter (HI9025, Hanna Instruments, Limena, Italy) was firstly standardized using buffer solutions of pH 4.0 and pH 7.0 (Fisher Scientific, USA). Furthermore, the concentrations of COD were tested through a test kit HI93754B-25 (Hanna Instruments Australia, Melbourne, Australia) whilst the NH_4^+-N and PO_4^{3-}-P contents were determined using test kits 100683 and114848 (Merck Millipore, Burlington, USA), respectively. Notably, the liquid samples for determining anion and cation concentrations were filtered using 0.20 μm filters (Merck Millipore, Burlington, USA) prior to analysis. The cell potential was recorded three times per day via a universal digital meter (VC86E, Shenzhen City Station Win Technology Co. Ltd., Shenzhen, China). To increase the accuracy of experimental results, all samples were analyzed in duplicate.

3. Results and discussion

3.1 Electricity generation

The variations in the electricity generation of the dual-compartment MFC were investigated while increasing the influent concentration of NH_4^+-N from 5 to 40 mg/L (see Fig. 1). Reportedly, the ammonium could be directly oxidized in the anode compartment of the closed-circuit MFC when the ammonium was the sole substrate (Hussain et al., 2016; Zhan et al., 2012). Electrons could be generated as a result of this. In the present study, however, no evidence was found that electricity generation was enhanced due to the presence of ammonium ions which may act as the electron donor. Certainly, it is possible to operate the MFC in a situation where the ammonium ions served as the only energy source in a closed-circuit, which could identify the potential of direct ammonium oxidization and ammonium acting as the electron donor.

However, this method does not help to conserve methanogenic bacteria and EAB. Also, a certain increase in the amount of ammonium in the anode chamber of MFC could enhance the anolyte's ionic concentration and conductivity, which improved the electron transport and thereby benefited electricity generation in the MFC reactor. However, the conductivity of anolytes does not have a direct relationship with the MFC reactor's generation of electricity (Liu et al., 2017) since increasing the conductivity of feed water may result in simultaneously elevating diffusion resistance and reduction in ohmic resistance (Nam et al., 2010a). On the other hand, ammonium could also directly serve as a building block for anode-attached microorganisms (Nam et al., 2010b). Based on these methods, the outcomes of ammonium on the power output of MFC are complicated and difficult to quantify.

As shown in Figure 1, the power output exhibited a decline as the feeding ammonium increased from 5 to 40 mg·NH_4^+-N/L, in which the maximum values of 598.9 mV (voltage generation) were obtained at the influent ammonium concentration of 5mg·NH_4^+-N/L. This may be attributed to the ammonium inhibition on the bioactivities of EAB, which detrimentally influenced the electricity generation of the MFC system. Ammonium inhibition on the power production has also been observed by other researchers in laboratory-scale MFC reactors (Hiegemann et al., 2016; Hiegemann et al., 2018; Kim et al., 2011; Nam et al., 2010b; Tice and Kim, 2014). As an example, the inhibited impacts of ammonium on electricity generation were detected in single-compartment MFCs while increasing the NH_4^+-N concentration over 500 mg/L (Nam et al., 2010b) or 3500 mg/L (Kim et al., 2011). By contrast, Tice and Kim (2014) and Kuntke et al. (2011) did not report the toxic impacts of ammonium on electricity generation even when the NH_4^+-N concentrations were larger than 2500 or 4000 mg/L, respectively. The different conclusions in these publications may be ascribed to the different wastewater sources and inoculum sources.

Interestingly, the inhibited effects of ammonium on the electricity generation gradually weakened with increasing ammonium concentrations. The possible reason for this is that the EAB utilized in the present anode chamber could be generally resistant against high ammonium concentration (Kim et al., 2011; Müller et al., 2006; Nam et al., 2010b). Consequently, the toxic impacts resulted from higher ammonium concentrations on the energy output of the MFC system would be generally decreased. In this scenario, decline in electricity generation could be eased. The low COD concentration of the domestic wastewater leading to maximum electricity generation in the present study was low compared with the results reported in other publications (Xia

et al., 2019). However, it suggested the potential for the dual-compartment MFC to produce electricity.

In addition, the power density and coulombic efficiency were analyzed in the present study. As illustrated in Fig. 2, the highest point of average power density was 230.17 mW/m^2 at the influent NH_4^+-N concentration of 5 mg/L. It could be seen that the power density decreased by approximately 50% when the NH_4^+-N concentration increased eight-fold to 40 mg /L. This relationship suggests an optimum ammonium nitrogen concentration of 5 mg/L. Besides, the optimum value varied from different studies; for instance, Kim et al. (2011) reported the value was approximately 4000 mg NH_4^+-N/L. Similarly, the coulombic efficiency fell from 25% to 15% while increasing the ammonium nitrogen concentration in the influent from 5 to 40 mg/L in the feed solution. It should be noted here that the reduction in power density and coulombic efficiency of the dual-compartment MFC was weakened at a higher concentration of ammonium nitrogen. This may be attributed to the EAB's ability to generally adapt to the environment with high ammonium concentrations.

3.2 COD removal

In the experiment, the carbon source of the double-chamber MFC was fixed at 300·CODmg/L. The COD removal efficiency of the MFC as per various feeding ammonium concentrations (5-40 NH_4^+-N·mg/L) was also studied (Fig. 3). In this continuous study, the COD removal efficiency in the MFC was calculated based on reactor influent and effluent COD concentrations. As shown in Figure 3, it could be evidently observed that the variations of influent NH_4^+-N concentration made little or insignificant impact on COD reduction, in which the removal efficiencies were almost over 90% during the MFC operation lasting for 150 days. Generally, the COD removal

rates in the MFC are influenced by the reactor operation, microbial source and growth at the anode compartment, MFC configurations, wastewater sources and many other factors affecting the COD removal. Interestingly, the microbial communities are also influenced by operational conditions (Zhang et al., 2011).

In the present study, the electrogenic bacterial and non-electrogenic bacterial in the anode chamber may determine the amount of COD removed (Logan, 2008). Moreover, the surface area of the anode electrode could contribute to a reduction in COD since the anode-attached biofilm could simultaneously adsorb and degrade the organics (Tamilarasan et al., 2017). Even though the EAB's activity was detrimentally influenced by increasing the concentration of ammonium nitrogen, it was reported that methanogens that are responsible for the organic removal are more tolerant of elevated ammonia concentrations than EAB (Nam et al., 2010b), which could nonetheless result in effective COD removal. Some authors reported that methanogenesis is negatively influenced by a high ammonium concentration (Chen et al., 2014; Nettmann et al., 2010; Wilson et al., 2012). Since the ammonium concentrations in the synthetic municipal wastewater were not too high, there was no observation of ammonium inhibition on methanogens.

It is worth noting here that the average removal efficiency of COD increased from 85.56% to 93.70% as the influent ammonium nitrogen concentration varied from 5 to 40 mg/L. Similarly, Tice and Kim (2014) also found that COD removal was still high (> 90%) until the ammonium concentration reached 1000 mg·N/L. Overall, the present dual-chamber MFC reactor shows superior efficiency of electricity generation and organic reduction (maximum power density of 230.17 mW/m^2 with 93.70% COD

removal) when compared to other MFC studies (e Silva et al., 2019; Hiegemann et al., 2016; Ichihashi and Hirooka, 2012).

3.3 Nutrient recovery

The concentrations of NH_4^+-N and PO_4^{3-}-P in the anode effluent and cathode effluent were examined, respectively. For this part of the experiment, the average removal rates of nutrients in the anode chamber and recovery rates of nutrients in the cathode chamber are depicted in Figure 4.

At the anode chamber of the dual-chamber MFC, the ammonium ions are always removed by the microbial growth and their transport across the CEM to the cathode compartment. The latter pathway offers an opportunity for the continual removal/recovery of ammonium in the double-chamber MFC. It should be noted here that the ammonium transfer to the cathode chamber includes current-driven migration and diffusion caused by the concentration gradient, in which the ammonium migration goes against the concentration gradient. From Fig. 4, the average amount of ammonium removed at the anode chamber increased from 0.7 to 5.78 mg/L as the NH_4^+-N concentration varied from 5 to 40 mg/L. Since the electricity generation was detrimentally influenced by higher feeding ammonium concentrations as well as the current-driven migration of ammonium, the possible reason for the enhanced ammonium removal at the anode chamber is that the anaerobic microorganisms gradually adapted to the high ammonium concentrations and took up more ammonium ions for their growth.

At the cathode compartment of MFC, on the other hand, several cations such as K^+, Mg^{2+} and Ca^{2+} existing in the feed solution would undoubtedly transfer to the cathode with the ammonium ions. Compared to other coexisting cations, K^+ ion has the highest

mobility, followed by NH_4^+, Ca^{2+}, and Mg^{2+} ions (Faulkner and Bard, 2002). The move rate of NH_4^+ is around 2 times smaller than for H^+. As a result of this, the transport of cations such as H^+ and K^+ against a concentration gradient to the cathode chamber could contribute to the generation of OH^- ions localized in the cathode electrode due to maintaining the charge neutrality of the MFC system (Rozendal et al., 2006). Also, the cathode reaction ($2H_2O + O_2 + 4e^- \rightarrow 4OH^-$) could result in the enhanced concentration of OH^- ions near the cathode electrode. Therefore, H_2O formation and a pH increase at the cathode occurs prior to NH_4^+ transport from the anode to the cathode (Faulkner and Bard, 2002; Kim et al., 2015). An outcome of this is that it is possible for ammonium ions to be removed by air stripping and/or recovered by chemical precipitation at the cathode compartment. According to another study (Ye et al., 2019b), these two pathways made a combined contribution to the ammonium removal at the cathode chamber. It could be seen from the figure that the average recovery rate of ammonium in the present MFC reactor declined from 85.11% to 15.33% when ammonium concentrations increased. This confirms that the double-compartment MFC could not effectively recover ammonium from ammonium-rich sewage. In contrast, Kuntke et al. (2012), for example, achieved a maximum ammonium recovery rate of 3.29 $gN/d \cdot m^2$ (*vs*. membrane surface area) with around 57% of ammonium being recovered from urine at the influent ammonium concentration of 4gN/L while using an air-cathode MFC. The difference is mainly attributed to different concentrations of mixed liquor suspended solids (MLSS) in anode chamber of MFC, influent COD concentrations, wastewater sources, MFC configurations, species of anaerobic microorganisms, DO concentrations in the cathode chamber and other factors influencing the ammonium recovery.

As for phosphate ions, the concentration of PO_4^{3-}-P in the solution feeding to the anode chamber of the MFC was 1 mg/L. Either the phosphate removal in the anode chamber or phosphate recovery in the cathode chamber was insignificantly affected by the variations in ammonium concentrations. Specifically, the efficiency in removing phosphate ranged from 11.37% to 13.33% whilst its recovery rate was in the 76.03-83.23% range. The performance of the double-compartment MFC reactor with reference to the phosphate recovery is superior to results published in several MFC studies (Fischer et al., 2011; Ichihashi and Hirooka, 2012). In the study of Fischer et al. (2011), for instance, 48% of phosphate was recovered in a dual-chamber MFC in the sewage treatment, but additional $MgCl_2$ and NH_4OH was needed for the struvite formation, which may reduce the system's sustainability. By contrast, a multi-chamber MFC proposed by Sun et al. (2018) could recover more than 89% of P from municipal wastewater because this configuration could enhance the phosphate concentration, which facilitates the further phosphate recovery. However, the operation in the multi-compartment MFC is more complicated compared to the dual-chamber MFC in terms of recovering nutrients.

4. Conclusion

In the present study, the impacts of ammonium loading rates on the dual-compartment MFC system were explored. This analysis demonstrated that COD removal was negligibly influenced by the feeding ammonium concentration. The MFC system obtained the maximum voltage generation of 598.9 mV and the ammonium inhibition on the electricity generation decreased at higher ammonium concentrations. Furthermore, the influent concentration of NH_4^+-N wielded great and negligible impacts on the recovery of ammonium and phosphate ions, respectively. Conclusively, the

double-compartment MFC was useful for recovering nutrients and energy from domestic wastewater under this premise of the acceptable range of NH_4^+-N concentration.

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Figure captions

Figure 1. Energy output of the MFC system as per different influent NH_4^+-N concentration (5-40 mg/L).

Figure 2. Coulombic efficiency and average powder density as per different influent NH_4^+-N concentration (5-40 mg/L).

Figure 3. COD removal efficiency of the MFC system as per different influent NH_4^+-N concentration (5-40 mg/L).

Figure 4. Average removal rate in the anode chamber and recovery rate of the double-chamber MFC: (a) NH_4^+-N and (b) PO_4^{3-}-P as per different influent NH_4^+-N concentration (5-40 mg/L).

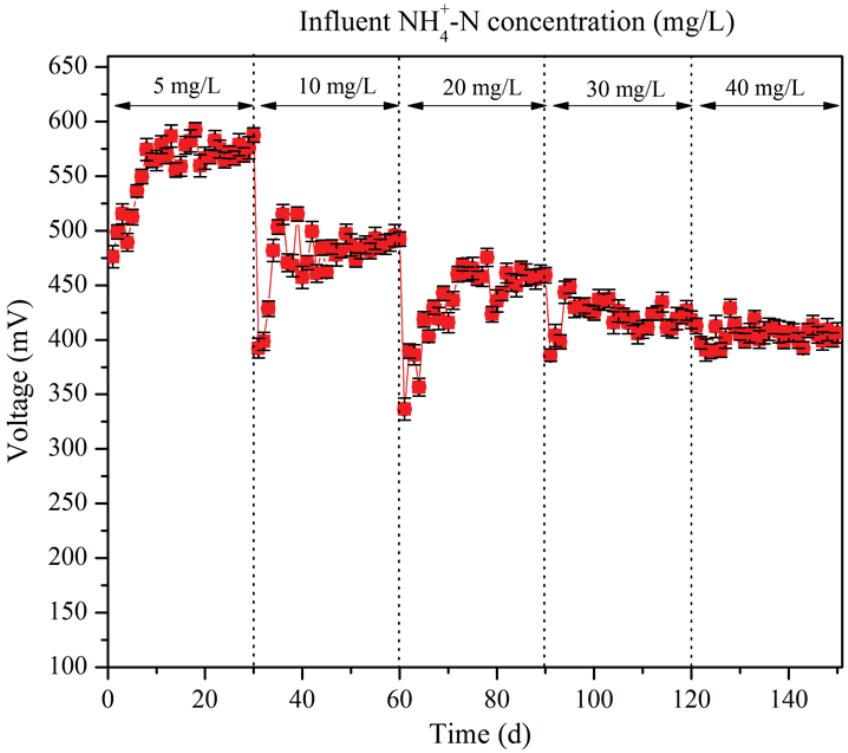

Fig. 1

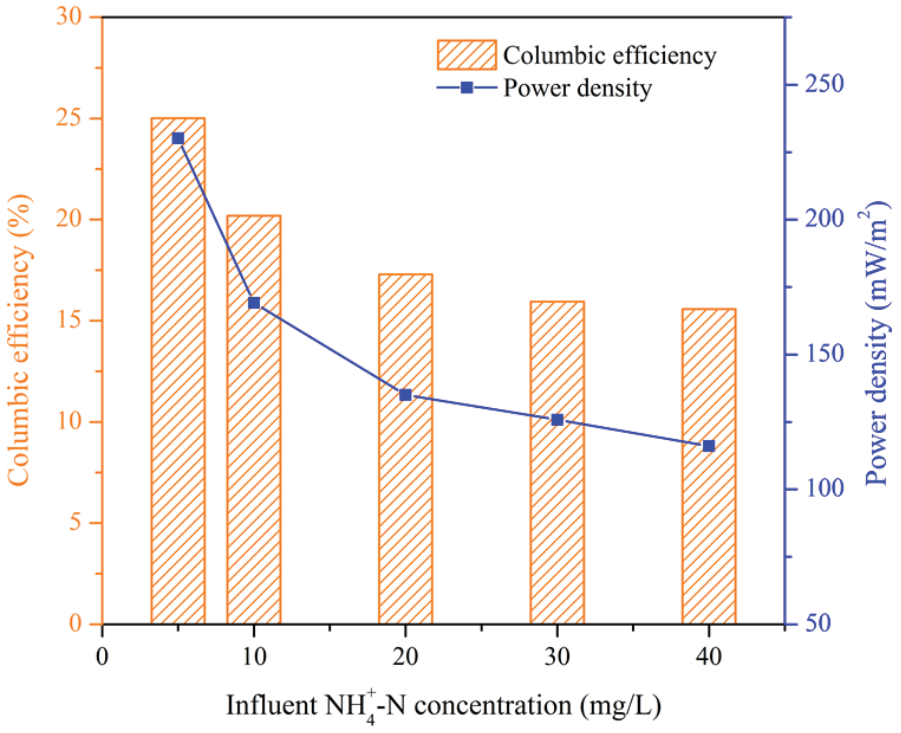

Fig. 2

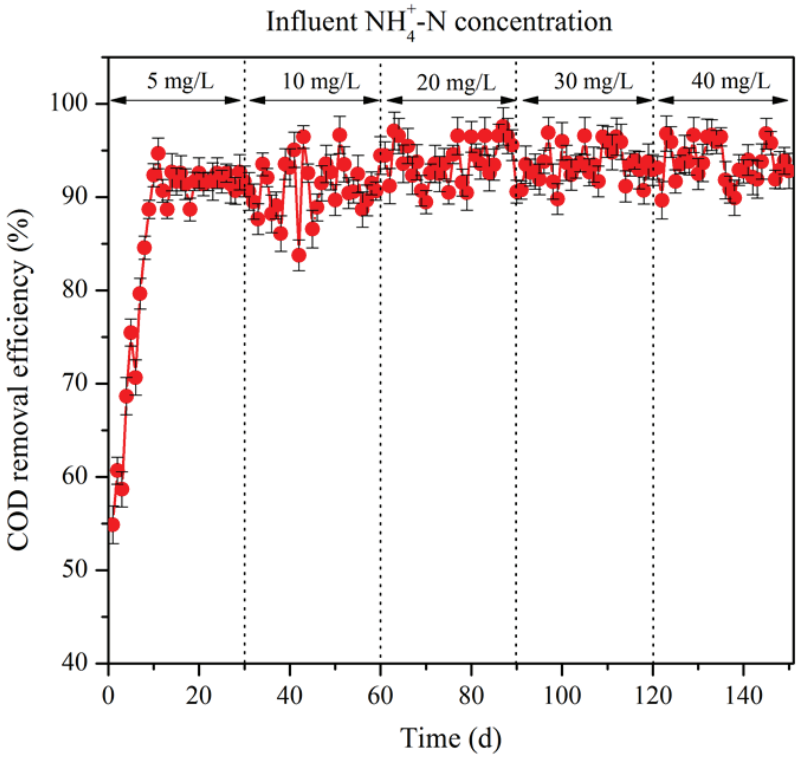

Fig. 3

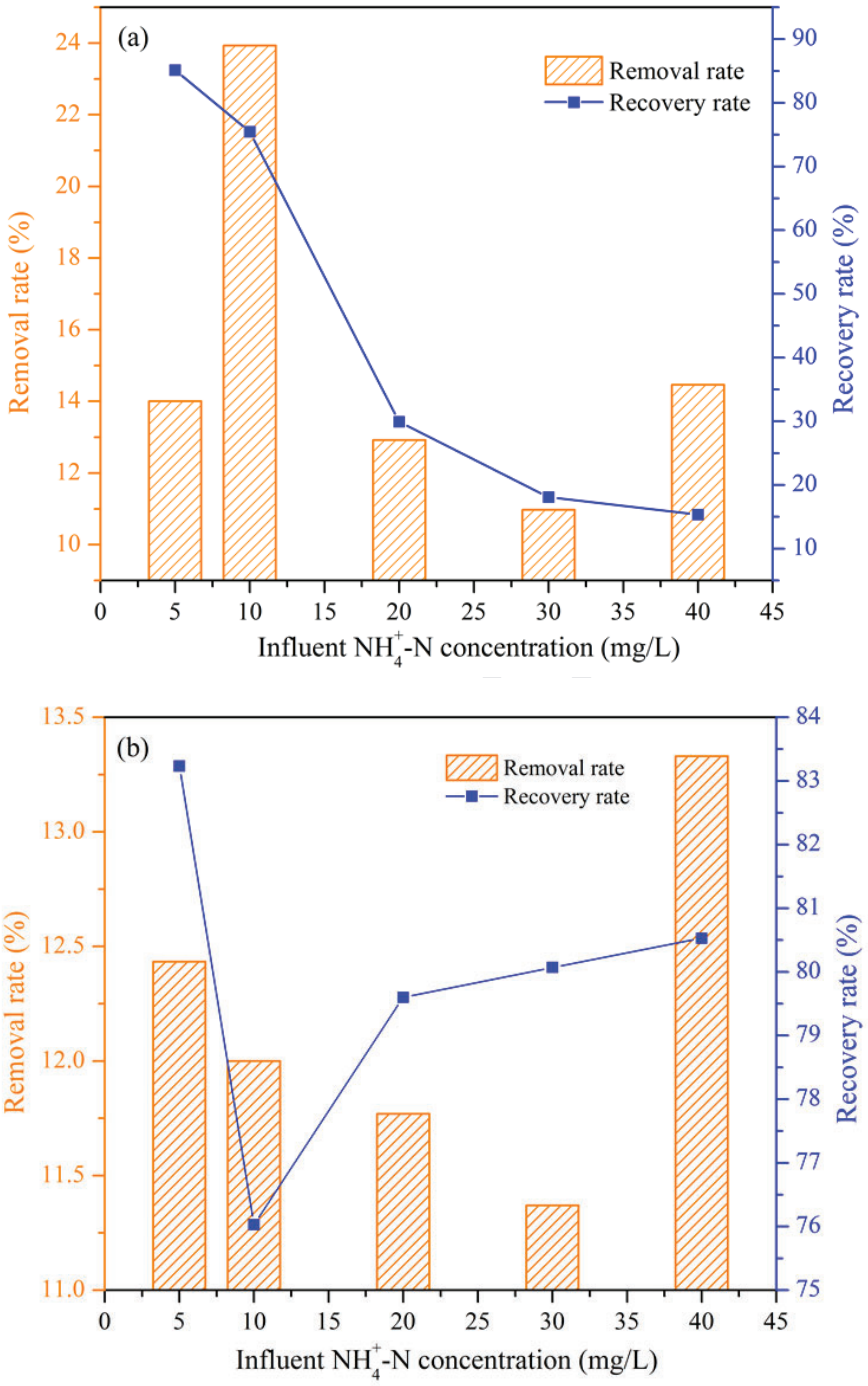
(a)
Removal rate
Recovery rate
Removal rate (%)
Recovery rate (%)
Influent NH_4^+-N concentration (mg/L)
(b)
Removal rate
Recovery rate
Removal rate (%)
Recovery rate (%)
Influent NH_4^+-N concentration (mg/L)

Fig. 4

Table captions

Table 1 Operational conditions of the dual-chamber MFC

Table 1. Operational conditions of the dual-chamber MFC

Parameters	Experimental period				
	i	**ii**	**iii**	**iv**	**v**
Days	1-30	31-60	61-90	91-120	121-150
Flow rate (mL/min)	0.35	0.35	0.35	0.35	0.35
Hydraulic retention time (d)	0.69	0.69	0.69	0.69	0.69
Influent NH_4^+-N concentration (mg/L)	5	10	20	30	40
OLR (mgCOD/L·d)	435	435	435	435	435