



Efficient removal of nitrobenzene and concomitant electricity production by single-chamber microbial fuel cells with activated carbon air-cathode



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HIGHLIGHTS

- Single chamber MFCs with AC air cathodes show high nitrobenzene tolerance.
- Electricity production can be achieved using nitrobenzene-containing wastewaters.
- Nitrobenzene can be efficiently removed in single-chamber MFCs.

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ABSTRACT

Single-chamber microbial fuel cells (S-MFCs) with bio-anodes and activated carbon (AC) air-cathodes showed high nitrobenzene (NB) tolerance and NB removal with concomitant electricity production. The maximum power over 25 W m^{-3} could be obtained when S-MFCs were operated in the NB loading range of $1.2\text{--}6.2 \text{ mol m}^{-3} \text{ d}^{-1}$, and stable electricity production over 13.7 W m^{-3} could be produced in a NB loading range of $1.2\text{--}14.7 \text{ mol m}^{-3} \text{ d}^{-1}$. The present S-MFCs exhibited high NB removal performance with NB removal efficiency over 97% even when the NB loading rate was increased to $17.2 \text{ mol m}^{-3} \text{ d}^{-1}$. The potential NB reduced product (i.e. aniline) could also be effectively removed from influents. The findings in this study means that single-chamber MFCs assembled with pre-enriched bio-anodes and AC air-cathodes could be developed as effective bio-electrochemical systems to remove NB from wastewaters and to harvest energy instead of consuming energy.

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1. Introduction

Nitrobenzene (NB) is widely used as an important chemical for the production of aniline (AN), dyes, pharmaceuticals, and pesticides (Couto et al., 2016; Ren et al., 2015), and as a solvent in products like paints, shoes and floor metal polishes. The highest NB concentration in effluents was $>1.5 \text{ mM}$ in the organic chemicals and plastics. Reported NB concentration in raw and treated industrial wastewaters from several industries range from 0.1 to 4.8 mM (Kuşçu and Sponza, 2009). Due to its recalcitrance and mutagenicity, NB has been listed as a priority pollutant, with strict limitations in environmental surface water (e.g. 17 µg/L in the US and 20 µg/L in China).

To remove NB from wastewater, methods based on different mechanisms such as adsorption, ozonation, advanced oxidation processes and reduction have been developed over recent decades. However, it is still a challenging task to improve the efficiency of these methods. For example, novel adsorption material with high adsorption performance should be developed to overcome the low adsorption of NB caused by low water solubility (Rauthula and Srivastava, 2011; Wen et al., 2012), and catalysts with high performance are required to improve the NB oxidation (Ayati et al., 2016; Chen et al., 2014; Rabaoui et al., 2013; Shen et al., 2014). Moreover, most methods based on chemical processes generally need to consume significant amounts of energy and/or require significant quantities of chemicals, which are cost-intensive and may cause secondary pollution (ElShafei et al., 2010; Rabaoui et al., 2013; Xie et al., 2014). Comparatively, biological methods are more cost-effective, but generally suffer from lower degradation rates and toxicity of NB to microorganisms at

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high concentrations. Bacteria could degrade NB by oxidation mechanisms coupled to reducing oxygen or sulfate (Kirui et al., 2016; Zheng et al., 2009), or by reduction mechanisms coupled to aniline production (Spain, 1995; Wang et al., 2011).

Microbial fuel cells (MFCs) aimed at the generation of electricity or removal of waste compounds have received significant attention over the past decade (Logan et al., 2015). Up to now, in addition to effective removal of complex organic wastes by oxidation processes in the anode chamber, MFCs have also been shown to successfully remove various contaminations including nitrate (Clauwaert et al., 2007), hexavalent chromium (Scialdone et al., 2014; Wang et al., 2008), by reduction processes in the cathode chamber. However, there are few studies of NB removal by MFC operation (Li et al., 2010). Recently, Mu et al. indicated that NB can be converted to aniline in the cathode chambers in bio-electrochemical systems with a bio-anode and a chemical cathode (Mu et al., 2009). Subsequently, other researchers found that the efficiency of NB transformation in the cathode chamber of bio-electrochemical system can be enhanced by using a bio-cathode instead of a chemical cathode (Liang et al., 2014; Wang et al., 2011, 2012; Zhang et al., 2015). However, instead of operating the systems in MFC mode, power input was needed to operate the bio-electrochemical systems, i.e. microbial electrolysis cells, to effectively convert NB to other less toxic products (e.g. aniline), although the energy consumption was much lower than required in pure electrochemical systems. For instance, a power input for NB removal was reported to be *ca.* 0.075 kWh mol⁻¹ NB in a membrane-free, continuous feeding up-flow biocatalyzed electrolysis reactor (Wang et al., 2012). Thus, it is attractive proposition to develop MFCs for efficient removal of nitrobenzene and concomitant electricity (Li et al., 2010).

For wastewater treatment, single-chamber MFCs with an air-cathode are generally accepted to be one of most suitable configurations for practical application due to the economic benefits of a simplified design, a reduced total volume, and improved power output (Liu and Logan, 2004; Liu et al., 2004). Additionally, cost-effective catalysts for oxygen reduction at air-cathodes have been attractive to constructing single-chamber MFCs. Activated carbon (AC), due to its high surface area, was considered to be an attractive cost-effective catalyst alternative to noble metal catalysts for oxygen reduction for single-chamber MFC configuration (Zhang et al., 2009). The performance of the AC cathode can be maintained at high levels over long-term MFC operation (Zhang et al., 2011), and under variable pH conditions (Zhang et al., 2016). In the present study, single-chamber MFCs with pre-enriched bioanodes and AC air-cathodes were found to have a capability of effective NB removal, with concomitant electricity.

2. Materials and methods

2.1. MFC configuration and operation

For comparison, two types of MFCs were employed in this study: single-chamber MFCs (S-MFCs) with microbial anodes and AC air-cathodes and double-chamber MFCs (D-MFCs) with microbial anodes and ferricyanide-cathodes. MFC chambers were made of plexiglass with a total empty volume of 118 mL (cylinder with 5.0 cm diameter and 6.0 cm length) and effective solution volume of ~60 mL. Electrodes assembled with three pieces of graphite felt (radius 2.3 cm, thickness 1.0 cm) and stainless steel (316L) bolt and nuts were used as anodes in S-MFCs and as anodes and cathodes in D-MFCs. Air-cathodes in S-MFCs was prepared using AC (Xinshen Carbon, Fujian), PTFE microporous filtering film (0.45 μm) and stainless steel mesh (316L) as catalyst, air-diffusion layer and current collector, respectively, as described in the previous study

(Zhang et al., 2016). To operate D-MFCs with ferricyanide-cathodes, a cation exchange membrane (Shenzhen) was installed to separate two identical chambers. Details of the MFC setup are provided in Fig. S1.

Bio-anodes were first pre-enriched for S-MFCs. A constant external resistance of 100 Ω was connected between the anode and cathode. To activate the MFCs, S-MFCs were initially operated by feeding NB-free artificial growth medium (AGM) in batch mode. The original inoculum source was taken from a separately operated (long-term running) MFC anode chamber which was inoculated with anaerobic activated sludge (sampled from the waste treatment plant in Yangzhou brewery) and was operated using acetate as electron donor over one year. The AGM, of pH 7.0, was prepared with the following constituents (in grams per liter of deionized water): NaAc, 1.6; NaHCO₃, 2.5; CaCl₂·2H₂O, 0.1; KCl, 0.1; NH₄Cl, 1.5; NaH₂PO₄·H₂O, 0.6; NaCl, 0.1; MgCl₂·6H₂O, 0.1; MgSO₄·7H₂O, 0.1; MnCl₂·4H₂O, 0.005; NaMoO₄·2H₂O, 0.001; yeast extract 0.05. The initial pH of AGM was adjusted to 7.0 using HCl and NaOH solution.

After the S-MFCs were operated in batch mode over one month to obtain mature anodic biofilms, MFCs were run by feeding AGM containing NB of 0.5 mM for subsequent batch cycles over one month, enabling MFCs to acclimate to the presence of NB. Following NB acclimatization, the MFC operation was transferred from batch mode to flow mode by continuously feeding AGM containing NB and increasing the concentration of NB in steps (0.5–7.0 mM), to investigate the effect of NB loading rate on electricity production and NB removal by S-MFCs. The flow rate was controlled at 0.2 ml min⁻¹ with a peristaltic pump (S1.5-2B, Signal Liquid Co., China), resulting in a hydraulic retention time (HRT) of 5 h. To compare the effect of NB on MFCs with different configuration, mature bioanodes developed in S-MFCs acclimating to 0.5 mM NB over one month, were employed to install D-MFCs. To run D-MFCs, the NB-containing AGM and the ferricyanide solution (50 mM buffered by 50 mM phosphate), were continuously fed into the anode chamber and the cathode chamber, respectively, with peristaltic pumps at a rate identical to that in the operation for S-MFCs.

2.2. Electrochemical measurements

The voltage produced by MFCs with an external load of 100 Ω, and the electrode potentials relative to a double salt bridge electrode saturated calomel electrode (SCE, Leici 217) was continuously measured using a multiple-channel high impedance voltmeter (Keithley 2700). Polarization curves of MFCs were measured using a battery testing system (Neware CT-3008W, Shenzhen, China) in the mode of constant current discharge. During polarization measurements, the bioanode and air-cathode of the tested MFC were connected to the negative terminal and positive terminal of the battery testing system, respectively. By selecting the mode of constant current discharge in the battery testing system, the tested MFC was controlled to discharge at constant current. The discharge current was increased from 0 mA (open circuit state) step by step with a typical current step of 1 mA until voltage output close to zero. At each constant current level, stable voltage output of MFC was measured to calculate the power output (Power = Current × Voltage), and the electrode potentials (vs. SCE) were measured to describe the polarization behaviors of the bioanode and the air-cathode. Cyclic voltammetry (CV) for bioanodes was performed using a potentiostat (CHI 660c, Shanghai, China) with SCE and the cathodes as reference electrode and counter electrode, respectively. For polarization and CV measurements, MFCs were first disconnected from the circuit until the open circuit voltage plateaued before measurements. Current density and power

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