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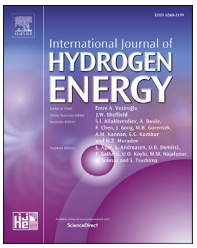

Effect of hydraulic retention time on continuous electricity production from xylose in up-flow microbial fuel cell

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ABSTRACT

Aerobic wastewater management is energy intensive and thus anaerobic processes are of interest. In this study, a microbial fuel cell was used to produce electricity from xylose which is an important constituent of lignocellulosic waste. Hydraulic retention time (HRT) was optimized for the maximum power density by gradually decreasing the HRT from 3.5 d to 0.17 d. The highest power density (430 mW/m^2) was obtained at 1 d HRT. Coulombic efficiency decreased from 30% to 0.6% with HRTs of 3.5 d and 0.17 d, respectively. Microbial community analysis revealed that anode biofilm contained known exoelectrogens, including *Geobacter* sp. and fermentative organisms were present in both anolyte and the anode biofilm. The peak power densities were obtained at 1–1.7 d HRTs and xylose degraded almost completely even with the lowest HRT of 0.17 d, which demonstrates the efficiency of up-flow MFC for treating synthetic wastewater containing xylose.

Introduction

Sustainability in wastewater management requires energy and performance efficiencies. The energy-rich compounds in wastewater should be converted to useful energy. One possibility to recover energy from wastewaters is production of electricity using microbial fuel cells (MFCs) [1,2]. In MFCs, microorganisms oxidize wastewater constituents and convert their chemical energy into electricity with simultaneous wastewater purification [3].

In Finnish paper, cardboard and pulp mills, in 2013, approximately 500 Mm^3 of wastewater was produced [4] containing cellulose and hemicellulose. Glucuronoxylans with xylose as the most abundant monomer, are hemicellulose that

Abbreviations: CE, Coulombic efficiency (%); COD, chemical oxygen demand; DGGE, denaturing gradient gel electrophoresis; HRT, hydraulic retention time (d); MFC, microbial fuel cell; OLR, organic loading rate (g/L/d); PCR, polymerase chain reaction; SL, sequence length; UV, ultraviolet; VFA, volatile fatty acid.

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is present in high concentrations especially in hardwood [5]. The occurrence of hemicellulose and thus xylose in forest industry wastewaters decreases the cost-effectiveness of the treatment process if xylose is not degraded [6]. For example, a yeast *Saccharomyces cerevisiae* cannot utilize xylose for bioethanol production without gene modification [7]. However, it has been reported that in MFCs xylose can be anaerobically converted to electricity [8–11].

Continuous treatment is a prerequisite for efficient and low-cost wastewater treatment. Only a few studies have reported continuous electricity production from xylose [8,10]. In continuous operation, organic loading rate (OLR) has a remarkable effect on electricity production [12] and the OLR is controlled by the HRT used. By now, several different reactor configurations have been tested for simultaneous electricity production and wastewater treatment, from which up-flow reactors are easily scalable and have comparatively low space requirements and thus have potential for future applications [12–16]. Up-flow reactors can be operated with high OLRs [17], i.e. low HRTs, and to treat wastewaters containing compounds, such as phenol [18]. Recently, granular activated carbon (GAC) has been reported at the MFC anodes to increase the surface area and performance of anodes as well as their wastewater treatment efficiency [19,20]. GAC can be combined with up-flow reactors, i.e. fluidized bed reactors [21], which further highlights the importance of up-flow configuration for bioelectrochemical systems in the future [20]. To make MFCs economically feasible for wastewater treatment, the treatment time should be close to the conventional processes. This makes HRT an important operational parameter [22].

This study examined the effects of HRT and organic loading rate on the ability of an up-flow MFC to convert xylose to electricity by further optimizing the operation parameters reported by Lay et al. [10]. The COD removal efficiencies and microbial communities at the anolytes were determined for each tested HRT. In addition, the microbial community of the biofilm was characterized in the end of the experiment.

Materials and methods

MFC construction and operation

The up-flow MFC used was similar to the one used by Lay et al. [10]. Anode and cathode chambers (working volumes 500 mL and 250 mL, respectively) of dual-chambered up-flow MFC (Fig. 1) were separated with an anion exchange membrane (Ø 4.5 cm, AMI-7001, Membranes International Inc., USA). The membrane was changed on days 23, 78, 117, 132, and 159 due to membrane fouling. Flat plate graphite electrodes at the anode and cathode (0.00385 m^2, McMaster-Carr, Aurora, OH) and 100 Ω external resistance were used [10]. A reference electrode (Ag/AgCl in 3 M KCl solution, −205 mV vs. standard hydrogen electrode (SHE), SENTEK QM710X) was attached to the anode recirculation tubing on day 15 through a glass capillary (QiS, the Netherlands). Anolyte temperature was maintained at 37 °C with heating coils around the anode chamber. Temperature was measured from the circulated anolyte which had a flow rate of 60 mL/min [10]. Medium was prepared as described by Mäkinen et al. [23] without addition of EDTA, yeast extract, and resazurin. Xylose (0.5 g/L) was used as substrate and pH of the medium was adjusted to 7.0 with NaOH before feeding. During continuous operation, influent container was kept in a cool box (approximately 9 °C) to minimize microbial growth outside the reactor. The catholyte was potassium ferricyanide (50 mM $K_3Fe(CN)_6$) in phosphate buffer (100 mM Na_2HPO_4, pH 7.0). Catholyte was circulated after day 83 through a container (500 mL) with a minimum flow rate of 0.2 mL/min. MFC was started as fed-batch where 0.5 $g/L_{anode\ chamber\ volume}$ xylose was added with an interval of 4–7 days. Continuous operation was started on day 43 with 3.5 d HRT, and HRT was gradually decreased to 0.17 d. Inoculum [10] was originally enriched from a compost culture.

Analyses

Electrochemical measurements and calculations

Cell voltage and anode potential were measured at 2 min intervals with an Agilent 34970A data Acquisition/Switch Unit (Agilent, Canada). The current was calculated from cell voltage (U) and external resistance (R) with ohm's law. Current and power densities were calculated against the projected area of the anode electrode (0.00385 m^2) or the volume of the anode chamber (0.5×10^{-3} m^3).

Performance analyses were performed at the end of each HRT by measuring cell voltage and anode potential after 30 min of stabilization with different external resistances (1000 Ω, 499 Ω, 240 Ω, 100 Ω, 10 Ω) and at open circuit mode. Power density and polarization curves were drawn from performance analyses results. Internal resistances were further estimated from the slopes of polarization curves according to Ref. [24].

Coulombic efficiency (CE) was calculated at each HRT using the measured cell voltage and the added influent xylose concentration over the periods with stable cell performance according to Equation (1)

$$C_E = \frac{M_s \int_{t_1}^{t_2} \frac{U}{R} dt}{F b_{es} \frac{t_b v_a}{HRT} c}, \quad (1)$$

where M_s = molecular weight of xylose (g/mol), $t_2 - t_1$ = time period of the measurement (d), F = Faraday's constant (96,485 C/mol * e), b_{es} = number of the electrons released per mol of xylose (20e^-), v_a = working volume of anode chamber (L), HRT = hydraulic retention time (d) and c = xylose concentration (g/L).

Sampling and chemical analysis

Xylose concentration, pH, and volatile fatty acids (VFAs) and alcohols were analyzed 3 times a week. During batch mode operation, samples were taken from sample port a (Fig. 1) before substrate was added. During continuous operation, samples were taken from sample port b (Fig. 1) and from effluent and influent. Samples for VFA, ethanol and xylose analysis were filtered through 0.2 or 0.45 μm PET filter. WTW pH 330 meter was used for measuring pH.

Xylose concentration was measured with phenol-sulfuric acid method [25] using customized sample and reagent volumes (1 mL sample, 0.5 mL 5% phenol solution, and 2.5 mL

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