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Electricity generation and treatment of paper recycling wastewater using a microbial fuel cell

Liping Huang · Bruce E. Logan

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Abstract Increased interest in sustainable agriculture and bio-based industries requires that we find more energy-efficient methods for treating cellulose-containing wastewaters. We examined the effectiveness of simultaneous electricity production and treatment of a paper recycling plant wastewater using microbial fuel cells. Treatment efficiency was limited by wastewater conductivity. When a 50 mM phosphate buffer solution (PBS, 5.9 mS/cm) was added to the wastewater, power densities reached 501± 20 mW/m^2, with a coulombic efficiency of 16±2%. There was efficient removal of soluble organic matter, with 73± 1% removed based on soluble chemical oxygen demand (SCOD) and only slightly greater total removal (76±4%) based on total COD (TCOD) over a 500-h batch cycle. Cellulose was nearly completely removed (96±1%) during treatment. Further increasing the conductivity (100 mM PBS) increased power to 672±27 mW/m^2. In contrast, only 144±7 mW/m^2 was produced using an unamended wastewater (0.8 mS/cm) with TCOD, SCOD, and cellulose removals of 29±1%, 51±2%, and 16±1% (350-h batch cycle). These results demonstrate limitations to treatment efficiencies with actual wastewaters caused by solution conductivity compared to laboratory experiments under more optimal conditions.

Keywords Microbial fuel cell · Paper recycling wastewater · Cellulose · Solution conductivity · Power production

Introduction

With the continued increased consumption of paper products and other natural fiber products, the recycling and use of recovered paper is growing worldwide. The average amount of recycling content in paper production was increased by 22% from 1990 to 1998. In 2005, 78% of paper and paperboard mills in America used some recovered paper, and 149 mills used only recovered paper. By 2012, it is projected that the paper industry will recover 55% of all the paper Americans consume (Lens et al. 2002). The strength of wastewater in a paper recycling plant generally increases with the percent of recycled content. Thus, an increase in the relative proportion of recovered paper and an increase in the amount of paper produced will lead to increased energy demands for wastewater treatment using conventional treatment processes. In addition, this wastewater contains soluble organics and particulate matter such as cellulose which are not effectively degraded by traditional wastewater treatment technologies (Lens et al. 2002). Many paper recycling industries therefore have an interest in reducing water use, finding more effective methods to treat their wastewater as well as decreasing costs for wastewater treatment.

One new promising method for wastewater treatment is the use of microbial fuel cells (MFCs). Bacteria in an MFC grow under anoxic conditions, which can benefit cellulose fermentation and degradation, with the added benefits of electricity generation rather than power consumption (Huang et al. 2008; Logan and Regan 2006). Several types of wastewaters have been successfully treated with simul-

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L. Huang
School of Environmental and Biological Science and Technology,
Dalian University of Technology,
Dalian 116024, China

L. Huang · B. E. Logan (✉)
Department of Civil and Environmental Engineering,
The Pennsylvania State University,
University Park, PA 16802, USA
e-mail: blogan@psu.edu

taneous electricity generation, including municipal, food processing, brewery, and animal wastewaters which have been found to be biocatalysts for directly power generation and waste treatment in MFCs (Feng et al. 2008; Liu et al. 2004; Min et al. 2005; Min and Logan 2004; Oh and Logan 2005).

Cellulose and chitin have been shown to be suitable substrates for electricity generation in laboratory MFCs under ideal conditions (Niessen et al. 2005; Ren et al. 2007; Rezaei et al. 2007; Rismani-Yazdi et al. 2007), but so far, actual cellulosic wastewaters from pulp or paper-processing plants have not been previously investigated. Of particular concern is the efficiency of an MFC to remove cellulose in the presence of other organic matter in the wastewater and the potential adverse effect of low conductivity of the wastewater. While the effect of solution conductivity on electricity generation is now well known in laboratory MFCs, it was recently demonstrated that low conductivity can have a detrimental effect on power production using a brewery wastewaters (Feng et al. 2008). We therefore wanted to determine to what extent it might be possible to treat a paper recycling wastewater (PRW) under more optimal conditions compared to those for the actual (unamended) wastewater.

In this study, we examined electricity generation using an unamended PRW and the same system with solution conductivity increased with a phosphate buffer solution (PBS). Power output was also examined as a function of wastewater strength, with treatment efficiency expressed in terms of removal of total chemical oxygen demand (TCOD), soluble chemical oxygen demand (SCOD), and cellulose.

Materials and methods

MFC reactor construction and operation

A single-chamber MFC containing a graphite-fiber brush anode was used in all tests, constructed as previously described (Logan et al. 2007). The brush was made of carbon fibers with an average diameter of 7.2 μm (PANEX33 160K, ZOLTEK) and was 6.0 cm in outer diameter and 7.0 cm long (estimated surface area of 5,418 m^2/m^3 brush volume). Anodes were treated using a high temperature ammonia gas (Cheng and Logan 2006) and were connected to the circuit by a titanium wire (Logan et al. 2007). The cathodes were made from 30 wt% wet-proofed carbon cloth (type B-1B, E-TEK) using a platinum catalyst (0.5 mg/cm^2 Pt) and four diffusion layers (Liu et al. 2008). The working volume of the MFC was 300 ml. The reactor was run in fed-batch mode at room temperature (22–26°C) and was not mixed except liquid sampling time.

Analysis

The voltage (*V*) across an external resistor in the MFC circuit was monitored at 20-min intervals using a multimeter (Keithley Instruments, OH, USA) connected to a personal computer. Current (*I*), power ($P=IV$), and CE were calculated as previously described, with the power density normalized by the projected surface area of the cathode and volumetric power density normalized by the volume of the liquid medium (Huang and Angelidaki 2008). Internal resistance was calculated from the slope in a plot of voltage and current (Liu et al. 2008).

TCOD and SCOD of wastewater samples were measured in duplicate (except as noted) using Standard Methods (APHA 1998). All samples for SCOD, gas chromatography (GC), and high-performance liquid chromatography (HPLC) analysis were filtered through a 0.22-μm pore diameter syringe filter, while TCOD measurements were conducted without pretreatment. Measurements of cellulose were made in duplicate using procedures described by Ren et al. (2007). The concentrations of possible intermediates (acetate, butyrate, propionate, ethanol, and methanol) were analyzed (triplicate samples) using a gas chromatograph (Agilent 6890) and a 30 m×0.32 mm×0.5 μm fused-silica capillary column. Before GC analysis, 50 μl 50% formic acid (*v*/*v* in water) were added to 1 ml sample. Other intermediates such as lactate and formate were measured by HPLC (Water CO.) with pre-acidification using 0.11 ml H_2SO_4 (1.0 M) per milliliter of sample before HPLC analysis. Methane and hydrogen in the headspace were analyzed using a gas chromatograph (model 310, SRI Instruments, Torrence, CA, USA) and a molecular sieve column (Alltech Molesieve 5A, 80/100, 6 ft×1/8 in.× 0.085 μm) with nitrogen as the carrier gas. The detection limits for TCOD and COD and cellulose were 5 mg/l and 10 μg/l, respectively. For intermediates (acetate, butyrate, propionate, ethanol, methanol, lactate, and formate), 1 mg/l was detectable, and for methane and hydrogen, the minimum detection limit was 0.001%.

Wastewater

PRW was collected from the primary clarifier of the American Eagle Paper Company, Tyrone, PA and used as the sole inoculum as well as the fuel. Each time the reactor solution as replaced, 30 ml of the solution was left in the reactor to ensure an adequate inoculum of bacteria in suspension for the next cycle of treatment. In tests with PBS, chemicals were added directly to the wastewater to produce a final concentration of 50 mM (NaH_2PO_4 H_2O, 2.452 g/l and Na_2HPO_4, 4.576 g/l) or higher as noted. In some cases, the wastewater was first diluted with ultrapure water (Milli-Q system; Millipore, New Bedford, MA, USA) before adding the buffer. Wastewater supernatant was obtained by allowing a 2 l sample to settle for 48 h and using the top 1 l in tests. Typical characteristics of the supernatant (g/l) were: total COD, 0.48±0.01; soluble COD, 0.32±0.01; ammonia nitrogen, 0.18±0.01; reducing sugars, 0.28±0.02.

Results

Power generation during start-up

After a lag period of 108 h after first inoculation using PRW (50 mM PBS), there was an increase in cell voltage over the next 60 h, reaching an initial peak voltage in the first cycle of 71 mV. Power was immediately generated at the start of the second fed-batch cycle. It was observed in the third cycle that there were two successive peaks in power output at 330 and 295 mV, lasting 108 and 118 h, respectively. After the fourth cycle, there were consistent power cycles exhibiting two stable and repeatable voltages produced each time of ~400 mV and ~420 mV (data not shown).

Relationship between initial COD, power generation, and CE

The power generated by the MFC was monitored for TCOD concentrations ranging from 181 to 1,464 mg/l (50 mM PBS). The maximum power densities, CEs, and TCOD removals at different initial TCODs are shown in Fig. 1. The maximum power production increased from 156 to 506 mW/m^2 (208 to 675 mW/m^3) with TCODs of 181 to 1,464 mg/l, demonstrating that power production was dependent on the initial TCOD (Fig. 1a,b).

Bimodal power production was observed for higher initial TCOD concentrations of 723 and 1,464 mg/l (Fig. 1a,b). At 1,464 mg/l, the peaks produced 340–420 mW/m^2 and 410–430 mW/m^2 of power for the initial 200 h and during 280–410 h, respectively. It was suspected that the reason for the two peaks was due to a change in the dominate source for current generation from soluble to particulate matter. Therefore, additional tests were conducted with the wastewater supernatant, and only one peak in power was observed, producing 400 mW/m^2 over the initial 200 h (Fig. 1b). This result supported our hypothesis that the second peak was due to solubilization of the particulate matter.

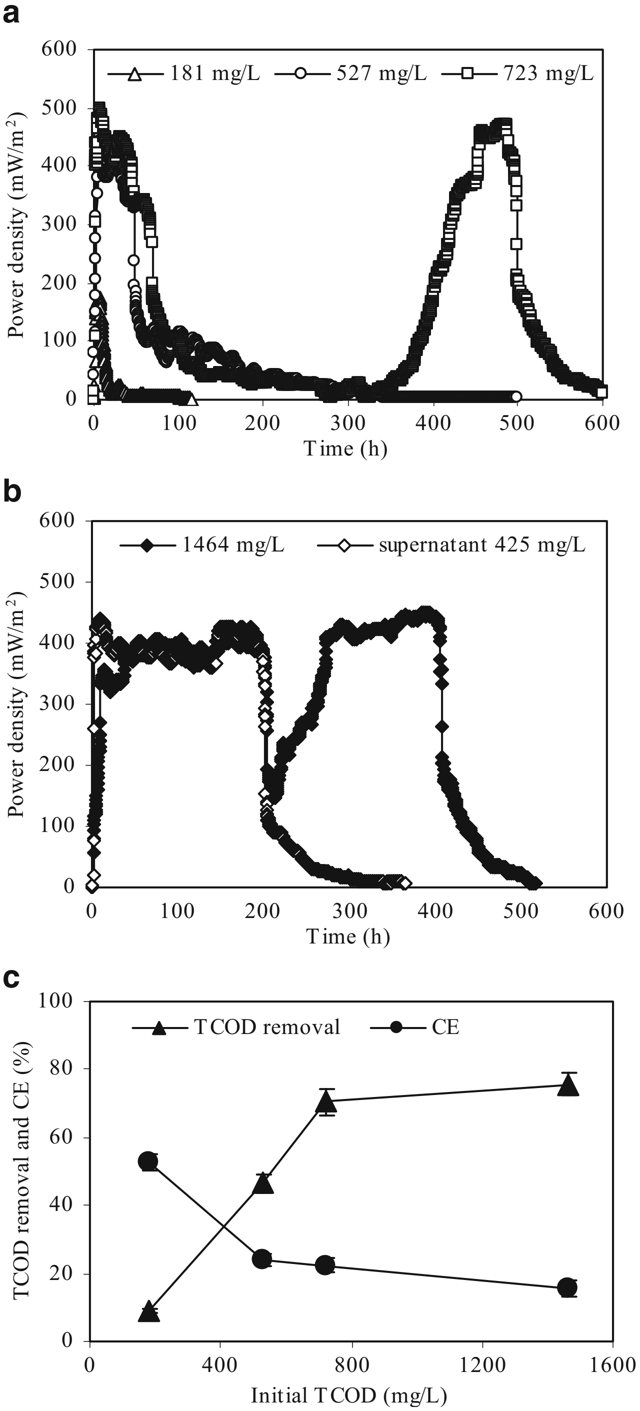

Fig. 1 Relationship between initial PRW concentrations and power generation (**a**) and (**b**), and COD removal and CE (**c**) in the MFC (R_{ex}, 1000 Ω)

The overall CE changed with the initial TCOD (Fig. 1c). An increase in the TCOD from 181 to 527 mg/l decreased the CE from 53±3% to 24±2%, but increased TCOD removal from 9.1±0.9% to 47±3%. A further increase in initial TCOD concentration to 723 mg/l resulted in slight reduction in CE to 22±2% and a TCOD removal of 70±4% (Fig. 1c). This relationship between CE and substrate concentration may be due to the effect of oxygen transfer into the solution through the cathode. The higher the substrate concentration the longer the period of time needed to fully degrade the substrate. As the time period increased, more oxygen could diffuse into the system, causing aerobic removal of the substrate and a decrease in the CE (Min et al. 2005).

Removal of TCOD, SCOD, and cellulose, and the production of intermediates

While the amount of substrates that was captured as current in MFC tests was low, electricity generation enhanced treatment efficiency beyond just conversion of the organic matter to current. TCOD removal in an open-circuit control was 27±2% compared to 76±4% removal in the MFC (1,464 mg/l initial TCOD; Fig. 2a). This result indicated that removal of TCOD was substantially enhanced by electricity generation from organic matters in PRW. The CE under these conditions was 16±2% (Fig. 1c), and thus the capture of electrons alone cannot fully explain the increase in TCOD removal from 27% to 76% by current generation. Reasons for this enhanced removal need to be further explored. The final TCOD (358±34 mg/l) after 500 h of treatment could not be further utilized for power generation and thus may reflect non-biodegradable organic matter (Lens et al. 2002).

There was a peak in SCOD concentration (206±14 mg/l) in the MFC tests at 350 h using the wastewater, but this peak was absent in tests using only the supernatant (Fig. 2a). This increase in SCOD occurred around the same time as the second peak in power production, as previously noted, at 280 h (Fig. 1a). This SCOD result provides additional evidence that the second peak in power production was due to the hydrolysis and fermentation of the particulate substrate, which resulted in the release of intermediates measured as SCOD.

Cellulose concentrations continually decreased over the time course of the experiment, from 1.18±0.05 to 0.051±0.02 g/l, achieving 96±1% cellulose removal after 500 h (Fig. 2a). Cellulose hydrolysis is the slowest step in cellulose degradation (Schwarz 2001; Drissen et al. 2007). While the initial peak in power production was likely sustained by soluble substrates originally present in the wastewater, lower power was generated when this soluble

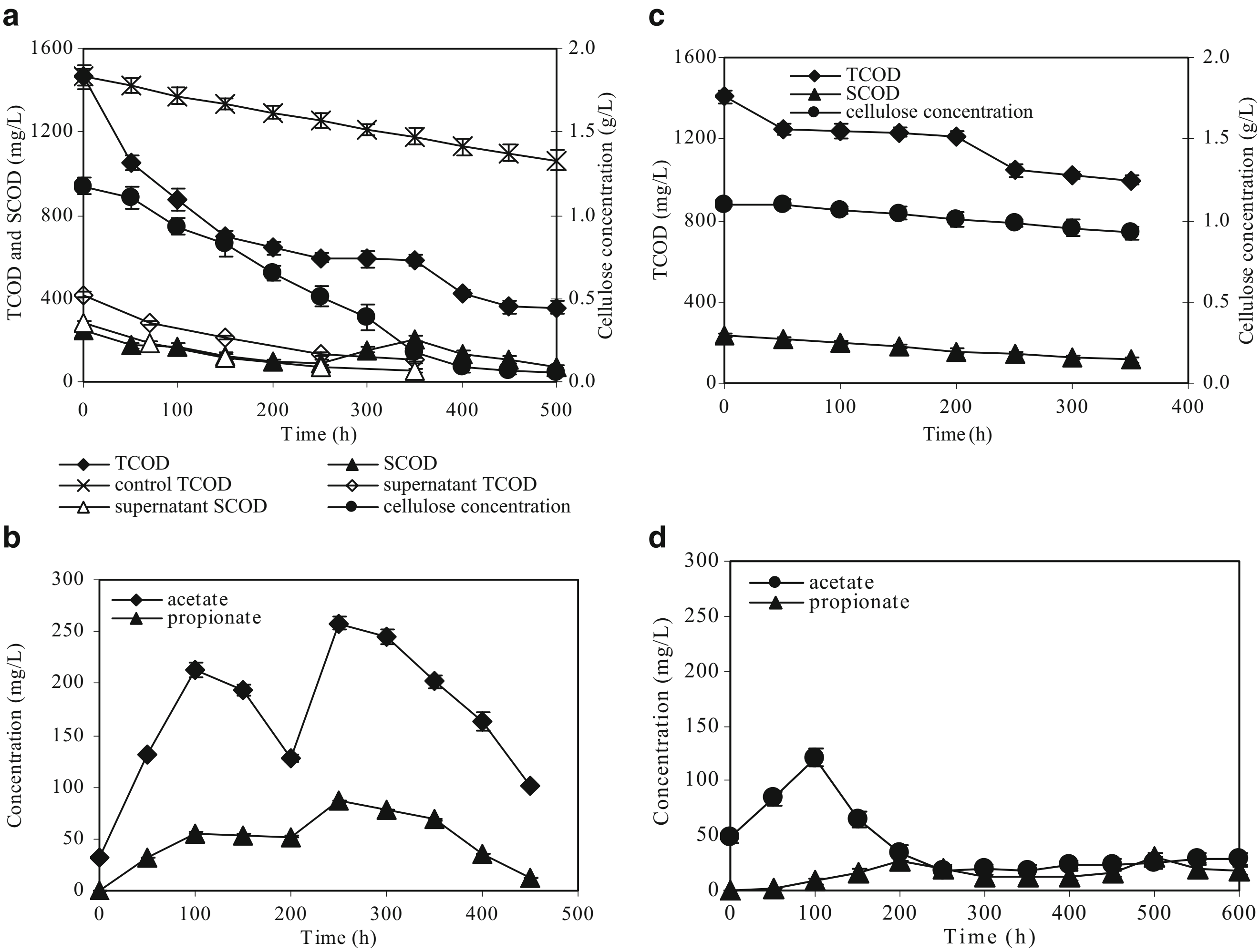

Fig. 2 Change of TCOD, SCOD, cellulose, and major intermediates over duration of MFC running using PRW with 50 mM PBS (**a**, **b**) or without PBS addition (**c**, **d**) (R_{ex}, 1000 Ω)

substrate was depleted. As cellulose degradation proceeded over time, the second peak in power generation was likely a result of an increased rate of cellulose hydrolysis (due to exponential bacterial growth), resulting in increased release of volatile acids and other intermediates over time.

To further examine the reasons for the bimodal peak in power output, we measured the concentration of volatile acids and these other intermediates over the course of a fed-batch cycle. Acetate was the dominant intermediate followed by propionate (Fig. 2b). Acetate reached a maximum of 258±6 mg/l at 250 h and then decreased, showing good agreement in the timing of acetate accumulation and power production (Fig. 1a). Propionate, another key intermediate in anaerobic degradation of cellulose, increased to 54±2 mg/l within 100–200 h, reaching a maximum of 86±1 mg/l at 250 h and then decreasing in concentration. Others have shown that acetate and propionate are the dominant intermediates during cellulose degradation by mixed microorganisms (Hu et al. 2004; Gallert and Winter 2008). Anaerobic oxidation of propionate by acetogenic bacteria is only possible if the hydrogen and formate concentrations are kept extremely low (de Bok et al. 2004), e.g., by the action of synthrophic hydrogen or formate utilizing bacteria such as methanogens or sulfate reducers. Formate and hydrogen gas were not detected here, allowing for anaerobic oxidation of propionate by acetogenic bacteria. Oxidation products from propionate by *Smithella propionica* include acetate and butyrate. The pathway of this propionate conversion is condensation of propionate to a six-carbon intermediate, which, after some rearrangements, is ultimately cleaved into butyrate and acetate (de Bok et al. 2004; Liu et al. 1999). While acetate was measured in high concentrations, the butyrate concentration here was only ~10 mg/l. Other intermediates (isobutyrate, valerate, ethanol, and lactate) in the liquid were undetectable (data not shown). CH_4 was not detected in the headspace, indicating that methanogenesis had been effectively inhibited (data not shown).

Effect of solution conductivity on power generation, removal of TCOD, SCOD and cellulose, and the production of intermediates

Solution conductivity was an important factor for maximizing power generation (Fig. 3a). Adding 100 mM PBS raised the solution conductivity from 0.8 mS/cm to 10.2 mS/cm and increased power density by up to 245%. This increase reflects the importance of solution conductivity to reduce the bulk phase internal resistance for power generation in the system as well as facilitate transport of ions through the anode biofilm (Torres et al. 2008).

Polarization data were obtained by varying the circuit external resistance (Fig. 3b). Maximum power densities increased from 144±7 mW/m^2 with unamended wastewater to 672±27 mW/m^2 (100 mM PBS). The internal resistances calculated from the polarization curves were 993 Ω (no buffer, R^2=0.998), 389 Ω (50 mM, R^2=0.996), and 292 Ω (100 mM, R^2=0.990). These results demonstrate that increased power resulted from a reduced internal resistance.

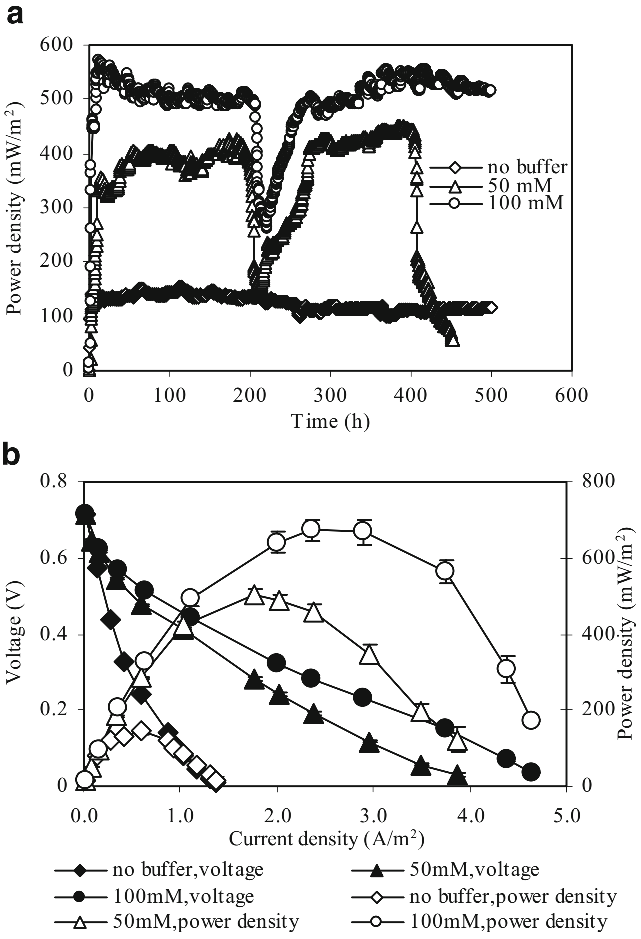

Fig. 3 Comparison of power density (**a**) and polarization curve (**b**) with or without PBS addition (R_{ex}, 1,000 Ω)

Solution conductivity also affected TCOD, SCOD, and cellulose removal of PRW as shown in Fig. 2c. Without buffer addition, removals of TCOD, SCOD, and cellulose (350 h) reached 29±1%, 51±2% and 16±1%, respectively. These values are much less than those with 50 mM PBS addition (conductivity of 5.9 mS/cm) of 60±2% (TCOD), 17±1% (SCOD), and 85±2% (cellulose) over the same period of time (Fig. 2a). Part of these differences can be attributed to the effect of the buffering capacity of the PBS. The final pH without PBS addition decreased from 7.0 to 5.9±0.2 after 350 h compared to 6.8±0.1 with 50 mM PBS.

It was also observed that when PBS was not added to the PRW, there was only one acetate peak at a value of 121 mg/l, a concentration much lower than the two peaks of 213 and 258 mg/l with 50 mM PBS addition (Fig. 2d). The accumulation of propionate in the PRW without PBS addition followed a similar trend to that observed with PBS addition, although the concentration was at a lower level (Fig. 2d).

Discussion

The use of MFCs to treat wastewater from a paper recycling plant is technically feasible, but these results demonstrate that solution conductivity and buffer capacity are important factors that can limit the amount of power that can be extracted from this wastewater. Adding a phosphate buffer (100 mM PBS, 10.2 mS/cm) showed that it was possible to achieve 672±27 mM/m^2 under optimal conditions. However, with unamended wastewater, the power output was only 144±7 mM/m^2 due in large part to the low solution conductivity (0.8 mS/cm). In addition, the pH of the wastewater decreased to 5.9, which may have affected the extent of treatability in terms of COD removal. There was 29±1% removal of TCOD of the wastewater without PBS addition compared to 73±1% removal under more optimal conditions (100 mM PBS).

While it is clear that PBS addition improved treatment, we are not advocating that PBS be added to the wastewaters to achieve increased power production, as this would not be cost-effective and the addition of phosphate would not be suitable for wastewater discharged at an industrial site. However, these results do show that power and treatment efficiencies can be severely affected by a lack of solution conductivity and buffer capacity. It has also been recently shown that solution conductivity was an important factor in the treatment of brewery wastewater (Feng et al. 2008). Therefore, it is suggested that in all future MFC reports of the treatment of wastewaters, data be included on the conductivity of the wastewaters tested. With such data, we will be able to better gage the importance of this factor in treatment efficiency. The solution conductivity has not previously been an important factor in conventional wastewater treatment using aerobic processes, except when salinity is very high and bacterial activity is inhibited.

Under optimal conditions, cellulose removal was very efficient, with 85±2% cellulose removed (100 mM PBS) compared to 16±1% with no PBS addition at an MFC running time of 350 h. Direct and indirect evidences shows the microbial ability to degrade cellulose is associated with the cellulosome (membrane-bound enzymatic complex). The binding of microorganisms to cellulose by hydrophilic peptide sequences present in the glycocalix proteins and/or in cellulolytic enzymes is necessary for its degradation (Paggi et al. 2004; Pegden et al. 1998; Zaldivar et al. 2001).

Cellulose degradation normally involves two separate steps: cellulose hydrolysis and then fermentation of the sugar, releasing fermentation end products into the solution (Schwarz 2001; Drissen et al. 2007). Cellulose conversion in an MFC therefore requires that bacteria be dispersed in the solution so that they can bind to the cellulose. These fermentation end products released into solution can then be used by the exoelectrogenic bacteria on the anode to produce current. There are no known microbes capable of both cellulose hydrolysis and electricity generation (Niessen et al. 2005; Ren et al. 2007). Thus, two separate steps are needed for particle breakdown, making cellulose degradation much more complex in MFCs than the degradation of just soluble substrates. However, the present study demonstrates that it is possible to accomplish both of these processes in the same reactor. Indeed, the removal of fermentation end products by anodic bacteria may be helpful in accelerating the rate of cellulose degradation through the removal of compounds (e.g., butyrate and ethanol) that can inhibit the growth of fermentative bacteria (Sakai et al. 2007). Further investigation of the bacterial community in an MFC degrading PRW and the use of continuous flow systems need to be explored in future studies to help understand and improve the performance.

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