INFLUENCE OF SLUDGE AGE ON THE PERFORMANCE OF MFC TREATING WINERY WASTERWATER

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ABSTRACT

The objective of this paper was to determine the influence of sludge age on microbial fuel cell (MFC) performance for generating electricity and removing organic matter from winery wastewater. Six Solid Retention Times (SRT) were used: 1.2, 1.4, 1.8, 2.3, 3.5 and 7.0 d. Results demonstrate that the electricity generation increases by decreasing the SRT, selecting electrogenic microorganisms, once the specific organic loading rate (SOLR) increased and the competition for substrate was reduced. Decreasing the SRT, coulombic efficiency can be increased from 3.4% to almost 42.2% and maximum power density from 58 to 890 mW m^{-2}. However the SRT did not influence on organic matter removal in biological treatment, because only a small part of COD was removed oscillating around 600 mg L^{-1} $d^{-1}$and it was very similar at all SRT studied.

Keywords: ►wastewater treatment ►energy recovery ► winery wastewater ►microbial fuel cell ►sludge retention time

HIGHLIGHTS

- Power generation increases by decreasing the SRT selecting electrogenic microorganisms
- SRT did not influence on organic matter removal in biological treatment
- Decreasing the SRT, Coulombic efficiency can be increased from 3.4% to almost 42.2%
- maximum power density of 890 mW m^{-2} can be achieved with MFC with SRT 1.2 d

INTRODUCTION

Microbial fuel cells (MFC) have become an emerging and promising technology that converts the chemical energy stored in organic and inorganic molecules directly into electricity, using microorganisms as biocatalysts (Rodrigo et al., 2007). Microorganisms oxidize organic matter on the anode producing electrons, which move through an external electrical circuit towards the cathode reducing an electron acceptor. Transport of ions through the bulk liquid or through an ion selective membrane keep the charge balance in the cell (Logan et al., 2006; Rabaey & Verstraete, 2005; You et al., 2006). MFC permits dual benefits like the wastewater treatment and power generation, seeming to be a promising approach to mitigate the environmental impact caused by wastewater. Nevertheless, the low power generation is one of the main bottlenecks for MFC technology, which greatly limits its development and industrial application (Rabaey & Verstraete, 2005).

Several parameters, such as operating conditions, reactor configuration, electrode material, membrane type, electrode surface area and external resistance, are known to affect MFC performance and are typically studied in works found in the literature (Akman et al., 2013; Gonzalez del Campo et al., 2013; Larrosa et al., 2009; Li et al., 2013; Patil & Gogate, 2012; Rahimnejad et al., 2011; Wei et al., 2012; You et al., 2006). However, to best of our knowledge, there are no studies on the effects of Solid Retention Time (SRT) on MFC performance.

The SRT or sludge age is an outstanding parameter for the design and operation of biological wastewater treatment processes (Rodrigo et al., 1996) and, consequently, it is expected to have a critical influence on the MFC performance, as well. SRT represents the average time spent by microorganisms in the biological reactor; and it is directly related to the population of microorganisms and the distribution of species, being a very effective method for the selection of populations. The lower the SRT, the faster should be the growth rate of microorganisms remaining in the biological reactor to avoid their wash out.

Although the choice of an SRT can lead to many consequences related to the biological wastewater process performance, its influence on the performance of the MFC were not yet well studied. Opposite, what it has been studied in the literature related to SRT is the influence on performance of the MFC of the Hydraulic Retention Time (HRT), although the literature is also scarce at this point (Kim et al., 2015b) and it is focused on the results obtained by changing HRT in a very limited range by feeding the MFC with different fuels such as domestic wastewater (Min & Logan, 2004) (Puig et al., 2011), milk processing wastewater (Kim et al., 2015a), synthetic wastewater (Wang et al., 2014) and urban wastewater enriched with glycerol (Guimaraes & Linares, 2014).

In previous papers of this group, the application of MFC technology to treat wineries wastewater was exhaustively studied and hence a deep understanding of the performance of this system was obtained. Taking into account this background, this paper focus on the effect of different SRT in the performance of a dual chamber MFC fed with winery wastewater, paying special attention to the study of COD removal of winery wastewater and the energy recovery.

MATERIAL AND METHODS

MFC configurations and operation

Two MFC were used in this work (Figure 1). They were made of acrylic tubes (inner diameter 40 mm; length 180 mm). Sterion® membrane (preconditioned using a 3% (v:v) hydrogen peroxide solution, 0.5 mol L^{-1} sulfuric acid and ultrapure water) was used to separate the MFC into two chambers with 70 mL (anode) and 100 mL (cathode), respectively. Carbon felts (KFA10, SGL Carbon Group®) were used as electrodes in both chambers. A stainless steel wire and an external resistance of 120 Ω connected the anode and the cathode. The electrodes in both chambers were not replaced when the SRT was changed.

The MFC was operated in parallel in semi-continuous mode with cycle time of 1 day or 24 hours and at room temperature (25 ± 3 °C). The anode compartment was inoculated only at the beginning of experiment. To regulate the SRT, every day, a volume of anolyte was taken from the anode chamber and replaced by fresh winery wastewater. It is important to point out that using this technique (the purge of mixed liquor microorganisms) only the microorganisms contained in the bulk of the MFC are directly affected and this procedure does not affect to microorganism fixed on surfaces (biofilm). It is also important to point out that at the same time, the Hydraulic Residence Time was simultaneously modified in the system. The amounts removed in the tests were 10, 20, 30, 40, 50 and 60 mL, which resulted in SRT of 7.0, 3.5, 2.3, 1.8, 1.4 and 1.2 d, respectively.

An important observation that should be taken into account in the discussion of results is that the SRT influence on performance was studied by changing this parameter in two different cells sequentially. MFC1 was operated at 7.0 d during 45 d and then the SRT was changed to 2.3 for 35 d and finally to 1.4 d for 10 days. Complementary, MFC2 was operated at 3.5 d during the firsts 41 d and then the SRT was changed to 1.8 for 35 d and finally to 1.2 d for 10 days. Overlapping of results obtained in both MFCs will be a clear indication of the reproducibility of the performance of this type of MFC and will support the conclusions drained from this work.

The cathode compartment of the MFC was connected to a water reservoir with 250 mL. A peristaltic pump was used to recirculate the solution of HCl (pH 3.5) from the reservoir through the cathodic chamber of the MFC at 1.66 mL s^{-1}. An aquarium aerator and porous stones diffusers were used in the reservoirs tank for supplying the oxygen to the cathode chamber.

Inoculum and wastewater

The anode compartment was inoculated with 90% (V:V) of activated sludge concentrated by sedimentation and collected from the activated sludge reactor at the municipal Wastewater Treatment Plant of Ciudad Real (Spain) and 10% winery wastewater. Hence, a mixed culture was used to startup the MFC. The concentration of total solids and total volatile solids were 15.8 and 11.1 g L^{-1} respectively.

The winery wastewater was collected from the regulating reservoir of the industrial wastewater treatment plant of the winery Bodegas Crisve (Socuéllamos, Spain), and stored at 4°C before being used. Table 1 shows the composition of this winery wastewater. $NaHCO_3$ (6000 mg L^{-1}) was used to adjust the pH to 6.5 and as buffer. Dibasic sodium phosphate ($Na_2HPO_4 \cdot 2H_2O$) and ammonium sulfate ($(NH_4)_2SO_4$) were added to increase the phosphorous and nitrogen concentrations to 10 mg P-PO_4^{3-} L^{-1} and 100 mg N-NT L^{-1} according to a previous study about the availability of nutrients for this type of wastewater.

Analytical methods

The pH, conductivity and dissolved oxygen were measured using a GLP22 Crison® pH-meter, a GLP 31 Crison® conductivity meter and an Oxi538 WTW® oxy-meter, respectively. The total suspended (TSS) and volatile suspended (VSS) solids were measured gravimetrically (Rodrigo et al., 2009). The COD and concentration of phosphorous were measured using a spectrophotometer (DR2000, HACH®). The total nitrogen was monitored using a Multi N/C 3100 Analytik Jena analyzer.

Electrochemical measurements

A digital multimeter (Keithley® 2000) was connected to the system to continuously record the value of the cell potential and the data were recorded in a personal computer. The polarization curves from the MFC were obtained by varying the resistance in the circuit and measuring the voltage. Power densities (mW m^{-2}) and current densities (mA m^{-2}) were based on the surface area of anode (7.0 cm^{-2}). The current (I) was calculated using Ohm's Law (I = E/R), and the output power of the cell using P= I E, where I (A) is the current, E (V) is the voltage, R (Ω) is the external resistance and P (W) is the power. Coulombic efficiency (CE) was based on total current generation and the maximum current that can be produced from COD oxidation and was calculated according a previous research (Rodrigo et al., 2009).

Results & Discussion

Figure 2 depicts the average values of COD, total nitrogen (TN) and total phosphorous (TP) before and after the daily feeding cycle for each SRT studied. The MFCs were operated in semi-continuous mode and every day a fixed volume of the anolyte (well homogenized) was replaced by fresh wastewater. Values shown in the Figure 2 were calculated once stabilized the COD, after 7 days of operation and the average value was calculated in order to avoid effects of fluctuations. It is important to take into account that the higher values observed for the COD, TN and TP concentrations for the lower SRT tested should not be considered as a direct consequence of the SRT but of the operating procedure planned, as can be seen in Figure 3. The lower the SRT, the higher is the amount of fresh winery wastewater added to the system and, hence, the lower is the dilution of the carbon and nutrient sources in the anolyte caused by the replacement with fresh wastewater. Another important observation that should be taken into account in the discussion of results is that the SRT influence on performance was studied by changing this parameter in two different cells sequentially. MFC1 was operated at 7.0 d during 45 d and then the SRT was changed to 2.3 for 35 d and finally to 1.4 d for 10 days. Complementary, MFC2 was operated at 3.5 d during the firsts 41 d and then the SRT was changed to 1.8 for 35 d and finally to 1.2 d for 10 days. A good reproducibility of results and the drawn of sound conclusions in spite of having used two independent cells is an additional proof of the robustness of the MFC technology and it will strengthen the conclusions obtained in this work.

Once clarified these important points, in comparing the daily COD removal, it can be observed that just a very small fraction of the organic matter contained in the feeding winery wastewater was removed during the operation of the MFC. In addition, this fraction did not seem to depend on the SRT: it is around 600 mg L^{-1} d^{-1} regardless the SRT (Figure 2). This observation should be explained in terms of the high fraction of recalcitrant substances contained in winery wastewater (Pepe Sciarria et al., 2015).The daily COD removal efficiency was only around 10%, being this value much lower than those observed by other authors (Cusick et al., 2010; Pepe Sciarria et al., 2015) who reported efficiencies of about 67 % and 27 %, respectively, for the same type of wastewater. Anyhow, the great variability of this type of wastewater, caused by the very different processes involved in the manufacture of wine and by the seasonality in the quality of this wastewater, can help to explain this divergence. Even with this low COD removal, the organic matter seems not to be a limiting factor for the MFC performance.

In comparing the TN and TP consumptions rates for the different SRT tested (Figure 2b and 2c), it can be noticed that the lower is the SRT, the higher is the resulting consumption of nutrients, suggesting a more active population of microorganisms. Moreover, even for the lower SRT, another important observation is that concentrations of nitrogen and phosphorous were not limiting the MFC operation because the concentration at the end of each daily feeding cycle is not negligible.

Figure 4 shows the microorganisms concentration, quantified as VSS and the resulting specific organic loading rate (SOLR). As expected, the VSS increased with the increase in the SRT. Longer SRT allowed more types of microorganisms to reproduce efficiently, increasing the VSS concentration. At this point, it is important to bear in mind that microorganisms with a low growth rate are washed out from the system if the removal rate (by purging) is higher than the growth rate and this washing-out may happen at low SRT. SRT control is an important method for the selection of populations because SRT does not only affect the concentration of VSS but most importantly the distribution of the different types of microorganisms. When the MFC was operated with a SRT of 7.0 d, the VSS was 0.643 g L^{-1}. From that value, the decrease of the concentration is almost linear and for the lowest SRT tested (1.2 d) the VSS was up to 72% lower (0.18 g L^{-1}). It is interesting to observe that SOLR increased reducing the SRT, indicating that more organic matter was available per microorganism, as direct consequence of the VSS decline. As explained before this higher availability does not reflect on the COD consumption rate, although it does in the TP and TN consumptions.

Once clarified the role of the SRT on the degradation of the winery wastewater, it is important to focus on the energy aspects of the MFC performance. To assess if the SRT influence the electrical generation the cell voltage and the polarization curves were monitored.

The temporal variation of voltage at different SRT in semi continuous MFC is shown in Figure 5. There is a clear influence of the SRT on the production of electricity and a one-fold difference is observed in comparing the two extreme values tested in this work. The highest average cell voltage was observed in MFC operated with 1.2 d SRT (178 mV) and the lowest was reached in reactor operated in 7.0 d SRT (18 mV). The cell voltage increased as the SRT decreased from 7 to 1.2 d (Figure 5A), which indicates that lower SRT enhanced the MFC power generation because non exoeletrogens microorganism were washed out and more organic matter could be used by exoeletrogens bacteria. The daily voltage profile, as can been seen in Figure 5B, showed an unstable profile, the voltage instantaneously increased but late it dropped. One possible reason for this increase was the addition of hydrochloric acid to cathode to control the pH in 3.5, which raise the concentration of protons reacting with oxygen and electrons and it generates more energy. As the time passed by, the concentration of protons in cathode decreased, and consequently the voltage reduced and pH raised.

For each SRT, polarization curves were recorded and showed the same behavior than that observed in cell voltage, as can be seen in Figure 6 with better performance for lower SRT. Thus, when the cell was operated with the longest SRT (7.0 d), the MFC showed a maximum of power density of 58 mW m^{-2} (95 mV). Decreasing the SRT to 1.2 d lead to a significant increase in the power densities: the maximum power densities were fifteen times higher than that reached with 7.0 d SRT (891 mW m^{-2} and 275 mV). It is interesting to observed that the slope of polarization curve (Figure 6) decreased when the SRT decreased from 7.0 to 1.2 d, because the ohmic loss was reduced due to the increment of conductivity of solution (Table 2) as consequence of the MFC operation mode (the lower the sludge age the higher the fresh winery wastewater amount added) and the microbial community selection in lower SRT.

The increase in cell voltage and in power densities observed in polarization curves varied linearly with the SRT, as shown in Figure 7. These relationships suggest that electrogenic microorganisms have a higher growth rate than non-electrogenic microorganisms and they prevail in the biological culture when sludge age is decreased. In fact, under low SRT those microorganisms with a slow growth rate are washed out because they required a larger time to reproduce. This may help to eliminate microorganisms such as acidogenic and methanogenic when the system was operated in low SRT decreasing the competition for substrate, as can been seen by the increment of SOLR in low SRT (Figure 4).

It is worth pointing out that although no significant changes in the COD depletion rate were observed for the different SRT, the activity of electrogenic microorganisms improved greatly when the SRT were reduced and a higher ratio of the COD was processed by electrogenic microorganisms, improving their prevalence with respect to non bioelectrogenic microorganisms. This prevalence can be clearly seen in Figure 8, where it is shown the changes in the Coulombic Efficiency calculated using the power generated and the COD consumed. When the SRT was 1.2 d, the Coulombic Efficiency increased twelve times from 3.4% when operated at SRT of 7.0 d to 42.2%, showing that the reduction of SRT favored the electrogenic microorganisms. These values of the Coulombic Efficiency can be considered as high, in particular if it is taken into account that maximum expected efficiency (for a pure culture of bioelectrogenic microorganisms) cannot exceed 40%, because this is the typical ratio of catabolic consumption of COD (the remaining 60% is used in biological assimilation reactions). At this point, it is interesting to observe that the reported Coulombic Efficiencies were higher than in other studies carried out by *(Cusick et al., 2010; Pepe Sciarria et al., 2015)* who used a single chamber air-cathode MFC (18 % and 15 %, respectively) for which ohmic loses are lower.

Conclusions

This paper demonstrates the relevant role of sludge retention time (SRT) on electrical power generation and winery wastewater treatment in a dual-chamber MFC. Power generation increases by decreasing the SRT, selecting electrogenic microorganisms, once the SOLR increased and the competition for substrate was reduced. However the SRT did not influence on organic matter removal, because only a small part of COD was removed and it was very similar at all SRT studied.

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Figure captions

Figure 1. Experimental setup

Figure 2: Average values of COD (A), total nitrogen (TN, B) and total phosphorus (TP, C) before (□) and after (■) feeding cycle and consuming rate in different sludge retention time (△).

Figure 3: COD temporal profile before (×) and after the feeding cycle in MFC1 (A) and MFC2 (B) in all conditions studied: in MFC1 with SRT 1.4 d (●),with SRT 2.3 d (△) and with SRT 7.0 d (○) an d in MFC2 with SRT 1.2 d (□),with SRT 1.8 d (▲) and with SRT 3.5 d (■).

Figure 4: Relationship between: the VSS (■) and SOLR (○) in different SRT studied.

Figure 5: Voltage produced during the lifetest of the MFC fed with winery wastewater in different SRTs: 1.2 (□), 1.4(●), 1.8(×), 2.3 (-), 3.5(■), 7 (○) d and the tendency (▬) in each case studied (A). Daily voltage profile in different days and conditions: with SRT 1.2 d in 83rd (□), with SRT 1.4 d in 86th (●),with SRT 1.8 d in 63rd (×), with SRT 2.3 d in 68th (-), with SRT 3.5 d in 30th 3.5(■) and with SRT 7.0 d in 31st (○) day of operation.

Figure 6: Polarization curves (A and B) obtained in the MFC fed with winery wastewater in different SRT: 1.2 (●), 1.4 (✳), 1.8 (×), 2.33 (▲), 3.5 (■) and 7 (◆) d.

Figure 7: Relationship between the average cell voltage (□, tendency ▬ ▬) and power densities (■ and tendency ▬▬▬) in different SRT studied.

Figure 8: Coloumbic Efficiencies during the life test of the MFC fed with winery wastewater in different SRTs: 1.2 (□), 1.4(●), 1.8(×), 2.3 (-), 3.5(■), 7.0 (○) d and the tendency (▬) in each case studied.

Tables

Table 1: Characteristics of winery wastewater used.

Parameter	Value
pH	4.11
Conductivity (mS cm^{-2})	2030
COD (mg L^{-1})	6850
TOC (mg L^{-1})	1030
Total Nitrogen (mg L^{-1})	18.3
Total Phosphorous (mg L^{-1})	0.95

Table 2: Average conductivities after feeding cycle for all SRT studied.

SRT (d^{-1})	1.2	1.4	1.8	2.3	3.5	7.0
Conductivity (mS cm^{-1})	10.2	9.9	8.1	7.5	6.0	5.7