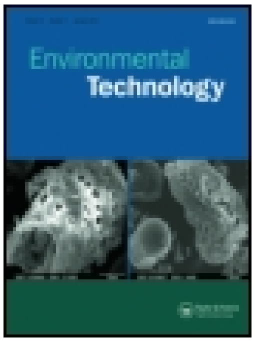

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Influence of carbon electrode material on energy recovery from winery wastewater using a dual-chamber microbial fuel cell

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ABSTRACT
The aim of this work was to evaluate three carbon materials as anodes in microbial fuel cells (MFCs), clarifying their influence on the generation of electricity and on the treatability of winery wastewater, a highly organic-loaded waste. The electrode materials tested were carbon felt, carbon cloth and carbon paper and they were used at the same time as anode and cathode in the tests. The MFC equipped with carbon felt reached the highest voltage and power (72 mV and 420 mW m^{-2}, respectively), while the lowest values were observed when carbon paper was used as electrode (0.2 mV and $8.37 \cdot 10^{-6}$ mW m^{-2}, respectively). Chemical oxygen demand (COD) removal from the wastewater was observed to depend on the electrode material, as well. When carbon felt was used, the MFC showed the highest average organic matter consumption rate (650 mg COD L^{-1} d^{-1}), whereas by using carbon paper the rate decreased to 270 mg COD L^{-1} d^{-1}. Therefore, both electricity generation and organic matter removal are strongly related not to the chemical composition of the electrode (which was graphite carbon in the three electrodes), but to its surface features and, consequently, to the amount of biomass adhered to the electrode surface.

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KEYWORDS
Wastewater treatment; energy recovery; winery wastewater; microbial fuel cell; electrode material

Highlights

- Power generation in a microbial fuel cell increases by increasing the electrode roughness and surface area.
- Electrode material influences the organic matter removal rate and electricity generation.
- By increasing the electrode roughness and surface area, the biomass adhered can be increased.
- A maximum power density of 420 mW m^{-2} can be achieved with carbon felt electrode.

Introduction

A microbial fuel cell (MFC) is a bioelectrochemical device that converts chemical energy into electrical energy using bacteria as catalysts. In the anode, microorganisms oxidize the organic matter of a fuel (which may be wastewater) and produce electrons and protons. As an example, Equation (1) shows the oxidation reaction when glucose is used as fuel. The electrons pass through an external circuit to the cathode, where they reduce an electron acceptor; in most of the studies, when oxygen is used as the oxidant in the cathode compartment, water is generated as a product on the cathode (Equation (2)). To maintain charge neutrality, the protons are transported from the anode to the cathode,[1,2] often through an ion-selective membrane, which helps to prevent the oxygen crossing to the anode compartment, improving the efficiency of the energy conversion device.

$$C_6H_{12}O_6 + 6H_2O \rightarrow 6CO_2 + 24H^+ + 24e^-, \quad (1)$$

$$4H^+ + 4e^- + O_2 \rightarrow 2H_2O. \quad (2)$$

In the last two decades, many studies have demonstrated that the MFC technology is capable to treat wastewater and generate electricity simultaneously.[3–8] However, there are several challenges to make this technology feasible for large-scale application. Even with the most recent advances in the research about MFC performance, the power density produced is very low, and it should be largely increased; meanwhile the costs associated with their construction need to be dramatically reduced. The very low current densities obtained nowadays by MFCs clearly seem to be their main

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drawback and their main handicap in the search for actual applications. In this way, the choice of an adequate electrode material may play an important role in the increase in performance and decrease in costs of the MFC technology.[9]

There are several characteristics necessary for an electrode material to be used in an MFC. A good candidate for electrode material has to show a high electric conductivity, good chemical stability, high mechanical strength and low cost.[9,10] Nowadays, the most widely used materials are the carbon electrodes, because they meet several of the general requirements. Also, carbon materials have high biocompatibility for microbial growth as a consequence of their surface characteristics, in particular the specific surface area and the surface roughness.[11]

Graphite rod, graphite fiber brush, carbon cloth, carbon paper, carbon felt and reticulated vitreous carbon are the most widely used anodic electrodes in MFC studies. In this context, although the choice of an electrode material is known to be important for the optimization of the performance of MFCs, comparison of untreated carbon electrodes in MFCs has not been well studied yet. Therefore, this paper focuses on the evaluation of the effect of different carbon electrodes (felt, cloth and paper) in the performance of a micro dual-chamber MFC, fed with winery wastewater (an already well-characterized fuel for the research group, with a high chemical oxygen demand [COD].[12] Special attention will be paid to the evaluation of the COD removal of winery wastewater and to the energy produced during this removal, because both outputs are the two main challenges to be faced in order to check the potential viability of the technology in the near future.[13,14] At this point, it is important to take into account that the main advantage searched for with the use of the MFC technology in wastewater treatment is the production of certain amount of electricity, which can partially compensate the high demands of energy associated with environmental remediation technologies.[15] At this point, highly loaded wastes, such as the winery wastewater, are the best target,[16] because they contain large amounts of substrate, which in the MFC behaves as a fuel, meaning a high amount chemical energy that can be converted into electricity.

Materials and methods

MFC configurations and operation

The MFC used in this work was a dual-chamber cell, separated by a Sterion® proton exchange membrane (Figure 1). Both chambers were built on resin-coated wooden boards. Between both boards, there were isolating rubber joints and the proton exchange membrane. The volumes of the anode and cathode chambers were 4 mL each.

The MFC was operated in parallel in semi-continuous mode and at room temperature (20 ± 3°C). The anode chamber was connected to a reservoir of 110 mL and a peristaltic pump was used to circulate the anolyte at a flow rate of 25 mL min^{-1}. To regulate the solid retention time at 2.2 d, everyday 50 mL of liquid was removed from the anode chamber and replaced by fresh winery wastewater. The cathode compartment of the MFC was connected to a water reservoir of 250 mL and a peristaltic pump was used to circulate an HCl solution (pH 3.5) from the reservoir through the cathode chamber of the MFC at a flow rate of 25 mL min^{-1}. An aquarium compressor and a porous stone diffuser were used for supplying oxygen to the cathode reservoir and chamber.

In order to minimize internal resistance (related to ohmic loses), both electrodes were placed in direct contact with the exchange membrane. An external resistance of 120 Ω was connected to the electrodes.

Carbon felt (KFA10, SGL Carbon Group®), carbon cloth (Panex®30 PW03, Zoltex®) and carbon paper (TGPH-120, Toray®) with 20% of Teflon were used in cathode and anode chambers as electrode materials. The characteristics of these electrodes are shown in Table 1.

Inoculum and wastewater

The inoculum was collected from the activated sludge reactor at the municipal Wastewater Treatment Plant of Ciudad Real (Spain) and, previous to its use in the MFC, it was concentrated by sedimentation. Total suspended solids (TSS) and total volatile solids of the concentrated sludge were 15.8 and 11.1 g L^{-1}, respectively. To inoculate the MFC, a medium containing microorganisms and winery wastewater (90% v/v) was prepared and added into the anode chamber of the MFC and kept for 24 h before starting the operation.

Winery wastewater samples were collected from the colleting tank of the Wastewater Treatment Facility of the winery Bodegas Crisve (Socuéllamos, Spain) and stored at 4°C before being used. The characteristics of wastewater are shown in Table 2.

$NaHCO_3$ (6000 mg L^{-1}) was used to adjust the pH to 6.5 and to buffer the wastewater during the tests. 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 and necessity of nutrients for this type of wastewater.[12]

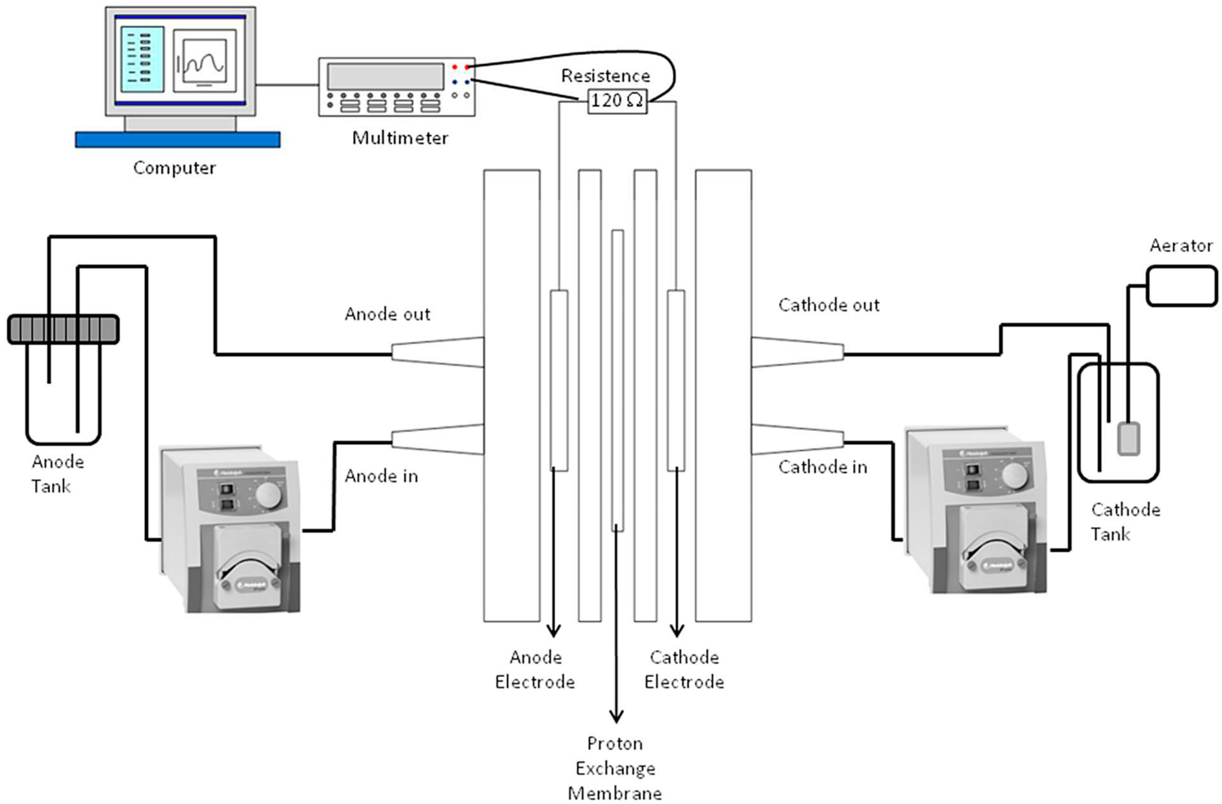

Figure 1. Experimental setup used in this study.

Table 1. Characteristics of carbon paper, carbon felt and carbon cloth used as electrode in the MFC.

Parameter	Carbon paper	Carbon felt	Carbon cloth
Specific surface area ($m^2\ m^{-2}$)	16	17,700	3500
Roughness (µm)	8	30	15

Table 2. 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
SO_4^{2-} (mg L^{-1})	810
Cl^-(mg L^{-1})	39.90

Analytical methods

The pH, conductivity and dissolved oxygen were measured using a GLP22 Crison® pH-meter, a GLP31 Crison® conductivity meter and an Oxi538 WTW® oxy-meter, respectively. The COD and concentration of phosphorous were measured spectrophotometrically (DR2000, HACH®). Total nitrogen (TN) was monitored using a Multi N/C 3100 Analytik Jena analyzer.

A digital multimeter (Keithley® 2000) was connected to the system to continuously monitor the value of the cell potential and the data were recorded on a personal computer. The polarization curves from the MFC were obtained by varying the resistance in the circuit and measuring the voltage. Power density (mW m^{-2}) and current density (mA m^{-2}) were based on the surface area of the anode (7.0 cm^2). The current (I) was calculated using Ohm's Law ($I = U/R$), and the output power of the cell using $P = IU$, where I (A) is the current, U (V) is the voltage, R (Ω) is the external resistance and P (W) is the power.

For the determination of biomass adhered in the electrode material, the electrodes were washed with glass beads and distilled water for biomass detachment. TSS and volatile suspended solids (VSS) were measured gravimetrically according to the standard methods previously used in this type of studies.[17]

The electrochemical impedance spectroscopy (EIS) was measured using a potentiostat/galvanostat AUTOLAB PGSTAT 30 equipped with a frequency response analysis module. The EIS measurements were performed at a frequency range from 0.001 to 10,000 Hz, with a potential difference of 0 V and amplitude of 0.01 V.

Results and discussion

To compare the performance of the MFC equipped with the three anode materials studied in this work, three cells (each equipped with one of the studied materials) were

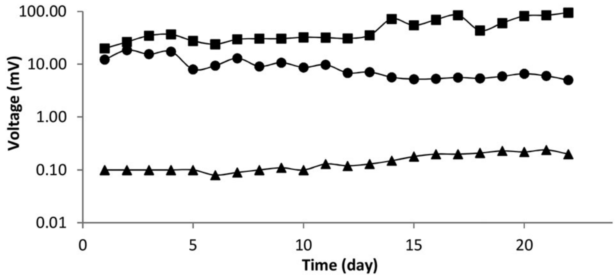

Figure 2. Temporal variation of voltage of the MFC with different electrode materials studied: carbon felt (■), carbon cloth (●) and carbon paper (▲).

started-up, connected to a resistance of 120 ohms and operated for more than two weeks before the stabilization of the cell voltage was achieved. Once the start-up stage was completed, normal operation was maintained for more than three weeks and several characterization tests were carried out.

Figure 2 shows the time course of cell voltage produced after this start-up period by the three MFCs, each of them equipped with one of the carbonaceous materials used in this work as electrodes: carbon felt, carbon cloth and carbon paper. They were run under the same operating and feeding conditions.

As it can be seen, there is a clear influence of the anode material on the production of electricity. The cell voltage increased by almost three log-units, only by changing the anode from carbon paper to carbon felt, despite the main composition of the three anodes being sp^2 carbon in the three MFCs. Carbon cloth shows an intermediate behavior, much better than carbon paper but worse than carbon felt. It is worth to mention that the carbon paper is doped with Teflon (20%), which may decrease the electricity production because of the high hydrophobicity of this electrode. In previous tests, it was found that the use of carbon papers with lower content of Teflon in MFCs should be avoided, because the electrodes degrade very rapidly.[1] Moreover, carbon paper has a lower roughness than the other materials, which decreased the easiness of microorganism adhesion. This is clearly shown in Figure 3 where it can be observed that the microorganism adhesion (measured in VSS cm^{-2}) follows the same fashion of the production of electricity, and the highest value is obtained for the carbon felt electrode (Table 1).

Figure 3 shows pictures of the anodes (naked eye) and they depict macroscopically the distribution of microorganisms adhered to each electrode studied. It is worth to notice that the carbon felt exhibits a more homogeneous distribution of microbial community than the carbon cloth and carbon paper electrodes, and this fact can be explained in terms of the roughness and specific area of the three materials tested. The carbon felt has the highest roughness and specific area. The microorganisms adhered to the carbon cloth and the carbon paper formed layers that decreased the direct contact between the microorganisms and the electrode, and in turn, the transfer of electrons.

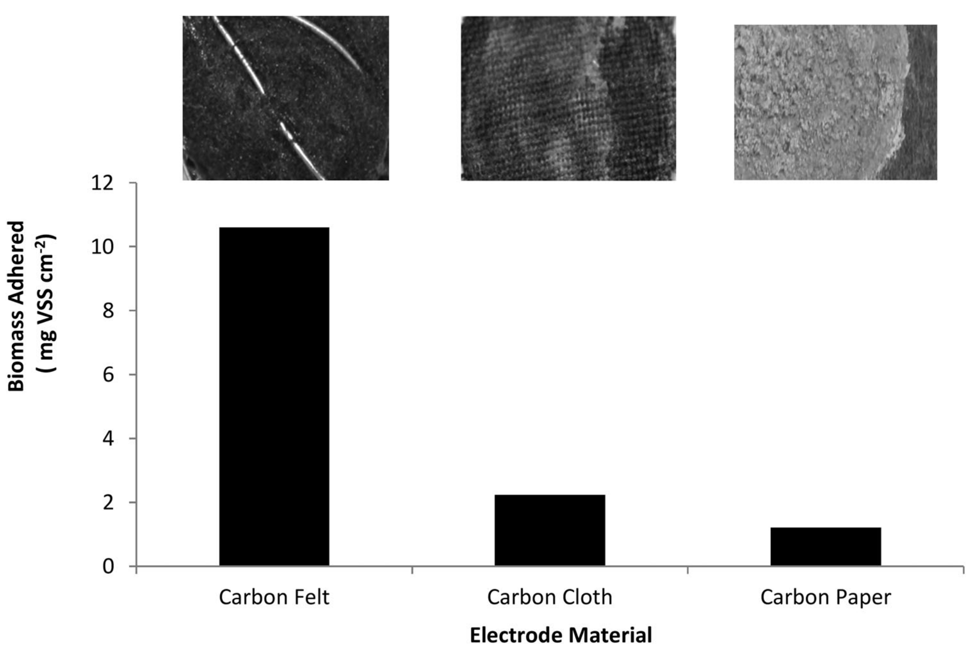

Figure 3. Concentration of microorganisms adhered in the anode of the MFC with different electrode materials studied and the photograph of distribution of microorganisms in carbon felt, carbon cloth and carbon paper.

Hence, the physical characteristics of the electrode material are very important, even more than the chemical composition, and they can justify the huge differences observed in the performance of the MFCs. To further study this influence, for each electrode material tested, polarization curves were recorded periodically during the operation of the MFCs. Figure 4 shows three polarization curves obtained on three different representative days of operation (5th, 12th and 19th day of operation). It can be observed that the performance of the MFCs gets better with the operation time and it is also interesting to point out the huge differences found among the results obtained with the three electrode materials. Typically, polarization curves are expected to show three different zones: activation losses, ohmic losses and mass transfer losses.[2] When carbon felt was used (Figure 4 (a,b)), only two zones were observed, activation and ohmic losses, showing that the third zone (typically associated with mass transfer losses) was not significant for this material. On the contrary, in using carbon cloth and carbon paper as electrodes in MFCs, it is possible to identify three zones, clearly pointing out that under high demand of current, mass transfer can limit the performance of MFCs with these two electrodes, because of the lower content of microorganisms available for the production of electricity and the more difficult access of substrate to the points on the surface of the anode in which microorganisms are fixed.

Regarding numerical values, the electrode material clearly influences the power density produced by each cell. When carbon felt was used as electrode, the MFC showed a maximum power density of 420 mW m^{-2}

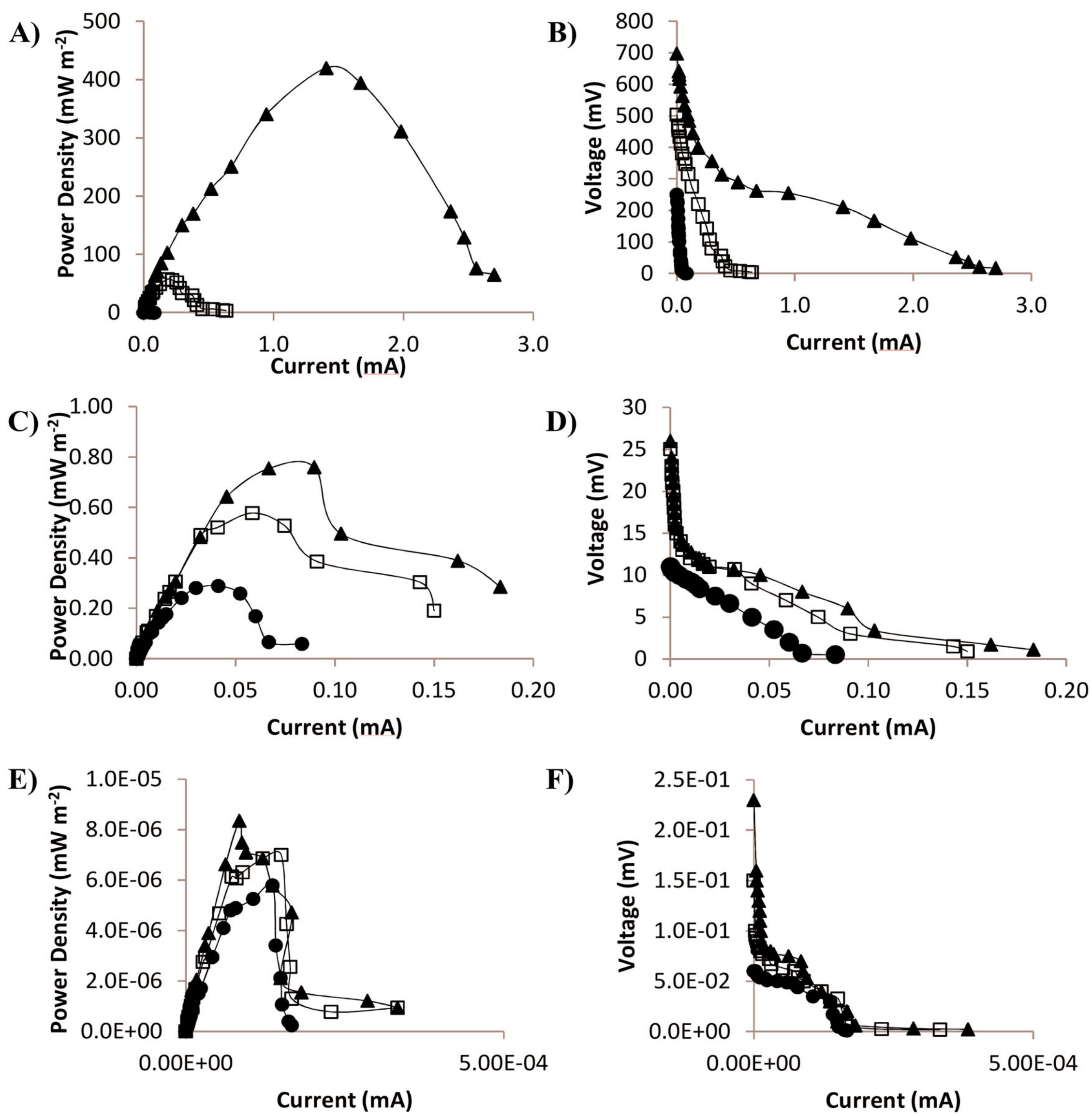

Figure 4. Polarization curves obtained in the MFC fed with winery wastewater using different electrode materials: carbon felt (a and b), carbon cloth (c and d) and carbon paper (e and f) and on different days of operation: 5th (●), 12th (□) and 19th (▲).

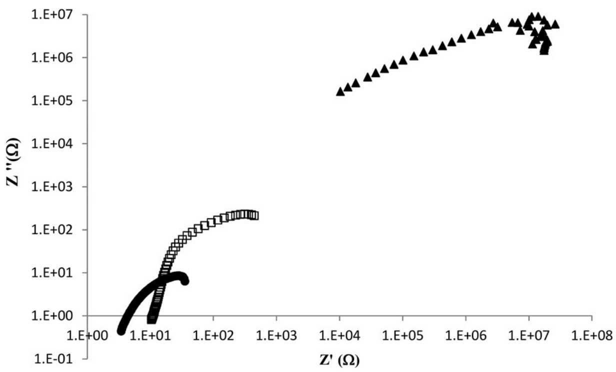

Figure 5. EIS for different electrodes used at the end of the operation: carbon felt (●), carbon cloth (□) and carbon paper (▲).

(211 mV, Figure 4(a)). Using carbon cloth as electrode resulted in a substantial two log-unit decay in power density. The maximum power density reached was 0.76 mW m^{-2} (Figure 4(c)) in this case. When carbon paper was used as electrode, the maximum power density was much lower than when the MFC was operated with carbon felt, reaching a maximum value of only 8.37·10^{-6} mW m^{-2} (Figure 4(e)). These significant differences in power densities should be a consequence of the amount of biomass adhered on the electrode and also of the reduction of the ohmic losses, although the macroscopic information obtained from the pictures about the microorganisms fixed (naked eye) is not as relevant as differences in the power densities pointed out.

Figure 5 shows the results of the EIS characterization of the different electrode materials. As it can be observed, the curves obtained are not as they were expected for a typical fuel cell.[18] Instead of semicircles, only tails are observed, suggesting a very complex behavior of the system different from what is expected of a fuel cell. For this reason, the only valuable information that can be obtained, in addition to the observed non-conventional behavior of the MFC

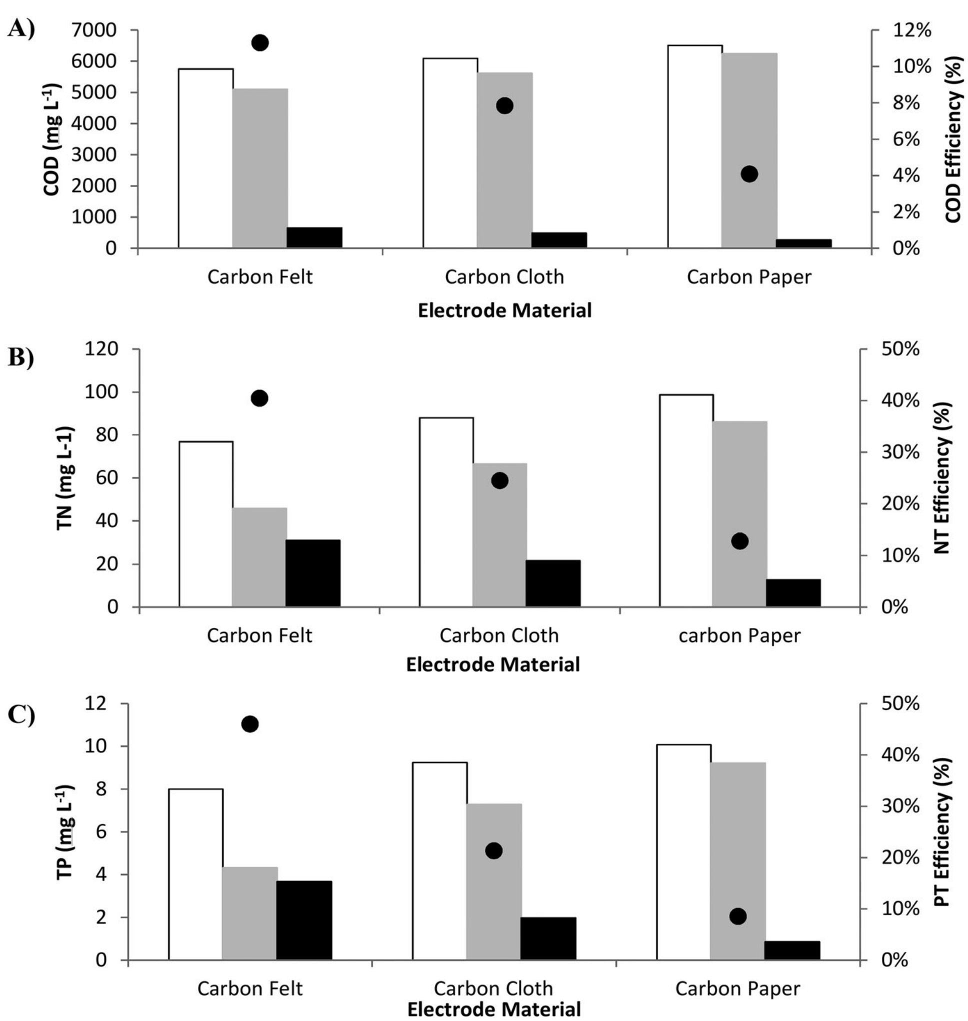

Figure 6. Average values of COD (a), total nitrogen (TN, b) and total phosphorus (TP, c) before (□) and after (■) the feeding cycle, the consuming rate (■) and efficiency (●) in the different electrode materials.

as fuel cells, is the lower resistance of the carbon felt as compared to that of the other two carbonaceous materials.

Figure 6 shows the average values of COD, nitrogen and phosphorous before and after the daily feeding cycle in each MFC studied. It can be observed that only a small fraction of the organic matter contained in the feeding winery wastewater was removed during the operation of the MFC. This observation should be explained in terms of the large fraction of biorecalcitrant substances contained in winery wastewater,[4] which becomes a handicap for the application of the MFC technology with this type of wastewater, since the amount of readily biodegradable substrate is not high and much lower than the organic content.

In addition, the COD removal seemed to depend on the electrode material. When carbon felt was used as electrode, the MFC showed the maximum average consumption rate (650 mg L^{-1} d^{-1}) and, as a consequence, it yielded the highest COD removal efficiency (around 11%). In using carbon cloth, the COD consumption rate decreased almost 170 mg COD L^{-1} d^{-1}, reaching the value of 480 mg COD L^{-1} d^{-1}. The lowest value was observed in the MFC equipped with the carbon paper anode, which exhibited a COD consumption rate of only 270 mg COD L^{-1} d^{-1}. These differences in the

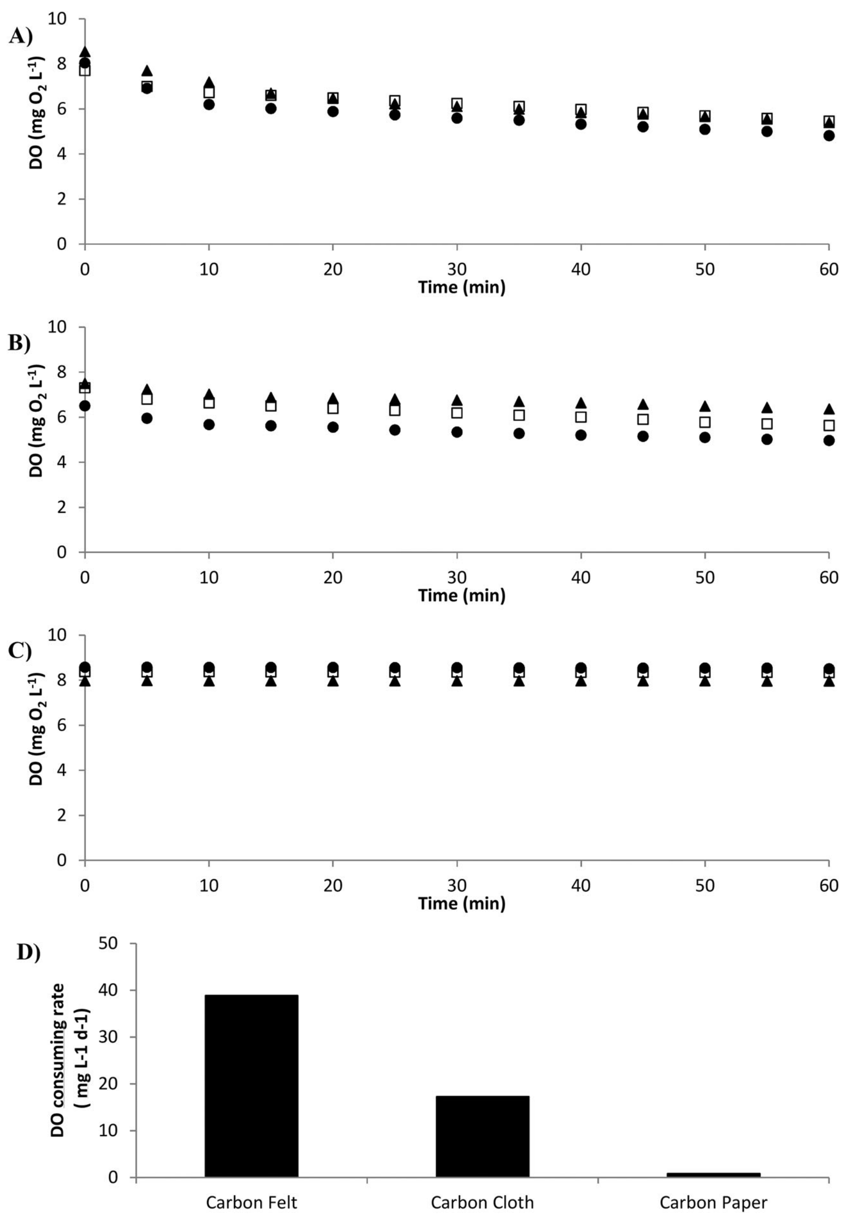

Figure 7. Dissolved oxygen consumption curve obtained in the MFC fed with winery wastewater using different electrode materials: carbon felt (a), carbon cloth (b) and carbon paper (c) and on different days of operation: 4th (●), 11th (□) and 18th (▲). Average dissolved oxygen consumption rate using different electrode materials (d).

COD consumption rate observed could be directly associated with the concentration of microorganisms adhered on the electrode surface which used organic matter to generate electricity. As shown in Figure 3, the concentration of biomass adhered to the carbon felt was almost onefold higher than that observed in the carbon paper. However, even with this more efficient electrode material, the daily COD removal efficiency was much lower than those observed by other authors [4,19] who reported yields of about 67% and 27%, respectively, for the same type of wastewater. There are many different processes involved in the manufacture of wine, and seasonality may cause great changes in the quality of winery wastewater, which may explain these discrepancies in COD removal. In addition, other factors should be influencing the MFC performance, such as temperature, inoculum, design and operation of the MFC. However, it is important to state that although the COD removal is low, organic matter was not a limiting factor for the MFC performance, because at the end of each daily cycle there was a high concentration still available for consumption (Figure 6(a)), and hence COD is not limiting the MFC performance.

The TN and total phosphorus (TP) consumption rates for the different electrode materials tested are shown in Figures 6 (b, c), respectively. The same behavior as in organic matter removal can be observed. When carbon felt was used, the MFC yielded the maximum average consumption rates of TN and TP (31 mg TN L^{-1} d^{-1} and 3.7 mg TP L^{-1} d^{-1}, respectively). Using carbon cloth, the TN and TP consumption rates decreased, reaching the values of 21 mg TN L^{-1} d^{-1} and 2.0 mg TP L^{-1} d^{-1}, respectively. The lowest values were observed in the MFC equipped with the carbon paper anode, for which values of 12.6 mg TN L^{-1} d^{-1} and 0.9 mg TP L^{-1} d^{-1} were obtained. The consumption of nutrients is related to the consumption of COD and, as expected, they follow the same trend. Furthermore, it can be pointed out that the concentrations of nitrogen and phosphorous were not limiting the MFC operation because the concentration at the end of each daily feeding cycle was not negligible.

In the cathode chamber of MFCs, oxygen is reduced to water. Despite cathodic oxygen reduction being a well-known reaction, it is worth not to forget that this is a very important process to understand the performance of an MFC, because all electrons taken from the fuel on the anode should be used in the cathodic reduction. For this reason, the oxygen consumption rate was evaluated during three different representative days within the experimental period. Figure 7 depicts the decay of dissolved oxygen in the cathode compartments after the air supply was stopped (tests lasted for more than 1 h). As it can be observed, for each electrode material tested, the dissolved oxygen consumption curves on the 4th, 11th and 18th days of operation were very similar, not varying substantially over the experimental period (Figure 6 (a–c)). It is interesting to observe that the electrode material influenced importantly the dissolved oxygen consumption rate. More efficient the material is in the production of electricity and in COD consumption, the higher the oxygen consumption is. Thus, as COD oxidation by bioelectrogenic microorganisms in the anode chamber is coupled with oxygen reduction in the cathode chamber, the COD consumption rate (Figure 7(d)) was 40 times larger in carbon felt than when carbon paper was used as the electrode in the MFC.

Conclusions

This study investigated the influence of carbon electrode material on electrical power generation and COD removal during the treatment of winery wastewater in a dual-chamber MFC. Significant power generation and organic matter removal were observed with carbon felt. By contrast, hardly any electricity was produced when carbon paper was used as anode. The improved organic matter removals and power generated obtained with carbon felt suggest that the roughness and specific area of the electrode and the concentration of microorganisms adhered to the electrode may be the determining factors to increase the MFC performance.

Disclosure statement

No potential conflict of interest was reported by the authors.

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