AIR BREATHING CATHODE-MICROBIAL FUEL CELL WITH SEPARATOR BASED ON IONIC LIQUID APPLIED TO SLAUGHTERHOUSE WASTEWATER TREATMENT AND BIO-ENERGY PRODUCTION

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Abstract

The present work explores the catalytic and electric performance of a microbial fuel ell (MFC) implemented with high chemical oxygen demand (COD) industrial wastewater from Spain. The polymer inclusion membrane based on 70% [MTOA+][Cl-] IL was used as separator and showed a good efficiency in power production and COD removal. Outputs of 72% in COD conversion, 200 mV voltage and 32 mW m^{-3} power density were obtained, demonstrating that slaughterhouse wastewater is a good feedstock for the scale-up of this technology. Furthermore, the effect of the microbial fuel cell on the physical/chemical parameters of the slaughterhouse wastewater was analyzed. The concentration of nitrite, orthophosphate, sulfate and ammonium was reduced by more than half. Air breathing cathode-microbial fuel cells based on polymer ionic liquids inclusion membranes allow the treatment of an industrial and high load slaughterhouse wastewater with good depuration and electrical performance efficiency.

INTRODUCTION

Nowadays, there is a general consensus in society and the scientific community that we are facing two major problems of global scope: water scarcity and the used of non-renewable energy. The number of people facing water stress (water supplies below 1700 m^3 per person) or water scarcity (below 100m3 per person) situations is increasing. Currently 40% of the world population live in areas with hydric problems and it has been estimated that by 2025 this figure will have increased to 66%. This lack of access to safe water supply and sanitation has devastating consequences such as hundreds ofmillions of

cases of water related diseases and more than 5million deaths every year as well as large unquantified impacts on economic productivity (1). Nearly half the cost of current standard wastewater is related to the energy input into the processes and the major percentage is related to aeration and treatment of the sludge generated during the treatment process (2) These high costs and difficulties in maintaining treatment infrastructure make it difficult to implement environmental strategies in countries with waste treatment and supply limitations. One way to reduce wastewater treatment cost is to directly reduce the energy inputs needed in each part of the treatment process. In this critical social context, microbial fuel cells (MFCs) have emerged as a promising technology that can overcome these two global problems. Microbial fuel cell microbes oxidize organic matter under anaerobic condition in the anodic compartment and transfer resulting electrons directly to a cathode, where the protons and electrons produced in the anode could be combined with oxygen to form water. Current may be produced from simple substratum (e.g. acetate, lactate or glucose) but what is really innovative was the finding that current can also be generated from complex substrates like domestic and industrial wastewaters. By using organic matter in wastewaters as a fuel, we can simultaneously produce energy and purify wastewater (3). Furthermore, the use of membranes based on ionic liquids could open up this field of improvement in MFCs. Ionic liquids could reduce the cost of the expensive proton exchange membranes and improve the efficiency of MFCs. Ionic liquids (ILs) are organic salts remaining as liquids near room temperature. They consist of an organic cation such as imidazolium, pyridinium, pyrrolidinium, phosphonium, ammonium, and a polyatomic inorganic anion or organic anion such as tetrafluoroborate, hexafluorophosphate, chloride, trifluoromethylsulfonate, bis[(trifluoromethyl)sulfonyl]imide) (4). In this work, we have investigated the application of new MFC technology based on ionic liquid membranes to the treatment of high COD content wastewaters from industrial slaughterhouses. Chemical and electrical performance of microbial fuel cells (MFCs) fed with a non-conventional slaughterhouse industrial wastewater was analyzed. New polymer inclusion membranes based on the ionic liquid [MTOA+][Cl−]was used as separator. The power density of the microbial fuel cells as well as the COD reduction and other physical-chemical parameters such as total suspended solids (TSS), nitrite, orthophosphate, sulfate and ammonium was analyzed.

MATERIALS AND METHODS

Reagents

The ionic liquid methyltrioctylammonium chloride, [MTOA+][Cl−] (purity >97%), poly(vinylchloride) (PVC) of high molecular weight and tetrahydrofuran (THF) were purchased from Sigma-Aldrich- Fluka Chemical Co. (Madrid, Spain). Substrates, solvents and other chemicals were purchased from Sigma-Aldrich-Fluka Chemical Co, and were of the highest purity available.

Experimental setup

The experiments were carried out in 250 mL microbial fuel cells provided with a single anodic chamber and with a cathode and membrane clamped at the tip of the anodic chamber leaving the cathode exposed to the air. Biofilm was grown on 100 mL of graphitic granules (Graphite Store, USA) at the anode (150 mL of wastewater) with electrons conducted by a graphite rod (Graphite Store, USA). Cathodes were made out of platinum dispersed on Vulcan (Alfa Aesar) on a carbon cloth substrate (0.5mg cm^{-2}).

As ion exchange membrane a polymer ionic liquid inclusion membrane based on 70% methyltrioctylammonium chloride ([MTOA+][Cl−]) on a polyvinylchloride matrix was used. Based on previous experiments (5) a 70% [MTOA+][Cl−] load was considered an optimum ionic liquid load. Experiments were conducted under external electrical load where anodes and cathodes were connected in closed circuit with a 1 kΩ resistor (see Fig. 1).

Experiments were conducted at a controlled temperature of 25 °C with two replicates per condition (average values are reported). In every case, reactors additional to the MFCs, named ‘Controls’ and ‘Baselines’ have been used simultaneously. Baselines consisted of sealed anaerobic reactors containing carbon granules as sole biofilm support (but unconnected to external components) with 100 mL of graphite granules and 160 mL of wastewater. They served to compare the outputs from MFCs under certain conditions with those from an anaerobic digester with biofilm under the same conditions. Controls consisted of sealed anaerobic reactors with 160 mL of wastewater; they served to compare

the outputs from MFCs under certain conditions with those from an anaerobic reactor with suspended microflora. The experiments were maintained for 240 h (10 days). In each experiment, voltage measurements were taken every 4 h and samples taken for wastewater characterization and electrical polarization measurements.

Wastewater characterization

The wastewater used as substrate for the experiments was obtained from a local slaughterhouse (Spain). A thorough characterization of the water was conducted in order to assess the initial values for the critical parameters, which are reported in the first column of Table 2. It is interesting to note that this water has a high COD content (5520 mg L^{-1}) compared with typical values found in domestic sewage water (500–1500 mg L^{-1} range).

Analytical methods

Microbial fuel cells were sampled by withdrawing a 5 mL aliquot using a plastic syringe. The sample was centrifuged at 5000 rpm for 5 min and filtered in a 0.45 μm membrane filter. An aliquot portion was reserved for COD analysis using the method AFNOR T90-101.6 For COD measurements, a Spectroquant Nova 30 spectrophotometer (Merck, Germany) was used. Soluble COD removal (COD)R is defined as the ratio between the total COD consumed in the process and the initial, the COD consumed being the difference between the initial chemical oxygen demand $[COD]_0$ and the chemical oxygen demand at a given time:

$$COD_R(\%) = \frac{([COD]_0 - [COD]_f}{[COD]_0} \times 100$$

total suspended solids, hardness, alkalinity and ion analysis were determined using common standard methods (6,7).

Polarization method

Polarizations were carried out through a self-made variable resistor box (11 MΩ–1Ω).

The voltage value in each case was taken once the cell had reached pseudo-steady-state at the corresponding resistor value (it was taken as approximately 1 min). The polarization method and its calculation is based on Logan's method (8).

Coulombic efficiency

The coulombic efficiency (CE) is the application of the concept of selectivity in microbial fuel cells. It corresponds to the electric charge that has accumulated during processing by the substrate removed, and is defined as the ratio between the number of coulombs transferred to the anode from the substrate and the maximum number of coulombs transferred if the entire substrate was able to produce current, i.e.:

$$Y_Q = \frac{coulombs\ produced}{total\ theoretical\ coulombs} \times 100$$

YQ = coulombs transferred total theoretical coulombs produced

The total coulombs obtained is determined by integrating current versus time in order to obtain the coulombic efficiency of a MFC fed batch mode, Cb, evaluated over a period of time tb, and is calculated as:

$$Y_Q = \frac{M_m \int_0^t i(t)dt}{F \cdot \Delta COD \cdot b \cdot V} \times 100 = CE(\%)$$

where:

CE: Coulombic efficiency

M_m: molecular mass of oxygen mm (32 g mol^{-1})

i(t): rated current (A = C s^{-1})

F: Faraday constant (96485 C per mol of e^-)

ΔCOD: variation of COD during the time t_b ($COD_0 - COD_t$)

b: moles of electrons produced per mole of oxygen (b = 4)

V: volume of liquid in the anode chamber (0.25 L)

The integral of the numerator, which is equivalent to the accumulated charge, was calculated by the trapezoidal method.

Preparation and SEM-EDX characterization of polymer ionic liquid inclusion membrane.

The polymer ionic liquid inclusion membranes (PILIMs) were prepared by dissolving 210mg of [MTOA+][Cl−] and 70mg of PVC in 3mL of THF. This solution was poured into a Fluka glass ring (inner diameter 28 mm, height 30 mm) on a Fluka glass plate, and allowed to settle overnight until total evaporation of THF had occurred, to obtain a thin plastic membrane. A scanning electron microscope (SEM) Hitachi S-3500 N and an Bruker AXS analyzer for energy-dispersive X-ray (EDX) analysis with high vacuum mode and variable pressure mode was used to study the morphological appearance, the overall chemical composition and the distribution of the chemical elements of interest present in the membranes. The PILIMs were characterized by SEM-EDX immediately after preparation (fresh membranes) and after use in the microbial fuel cell.

RESULTS AND DISCUSSION

Characterization of polymeric ionic liquid membrane used as separator in MFC

Polymer inclusion membrane based on [MTOA+][Cl−] ionic liquid was used as the separator in an MFC for slaughterhouse depuration. The ionic liquid membranes were characterized before and after use by scanning electron microscope (SEM-EDX) with the purpose of providing information about the topography, morphology and chemical composition of the membrane. Figure 2 shows SEM micrographs of the [MTOA+][Cl−] before (fresh) and after use in the MFCs. The micrograph of the fresh sample of [MTOA+][Cl−] 70wt% is shown in Fig. 2(a), highlighting the completely mottled surface of this sample. As can be observed in Fig. 2(b), the membrane appearance changed after use in the microbial fuel cell. Figure 3 (EDX spectrum) and Table 1 (EDX normalize % wt) show the results of the analysis before and after working as MFCs with slaughterhouse wastewater. In Fig. 3 we can observe from EDX analysis an important reduction of the Cl content of the membrane, since the spectrum of the membrane after use involves an increase of the relative peak heights C/Cl with respect to the fresh membrane. The relative peak heights of identical elements in the different compounds are

related to their respective concentrations. So we can conclude that some ionic liquid has been released from the membrane. In spite of the important reduction of the Cl content in the EDX analysis, it does not mean that the ionic liquid has been completed removed from the membrane. The stability of polymer ionic liquid membranes based on [MTOA+][Cl−] in water has also been studied. The membrane based on the ionic liquid [MTOA+][Cl−]was capable of retaining an important amount of ionic liquid after the fourth operating cycle.5 The reduction of Cl− could be due not only to losses of the ionic liquid but also to the EDX technics. The EDX spectra are taken from a sample of only a few micrometers thick/depth. After using the membrane in the MFC some of the ionic liquids from the surface membrane could be released and some material could be deposited on the membrane surface, as can be observed in micrographs (Fig. 2). These both could reduce the signal due to the ionic liquid. Furthermore, new chemical elements (Na, Mg, P, S, Ca, Fe, Al, Si) were found absorbed in the membranes after use which could come from the slaughterhouse wastewater. Besides the EDX of the global membrane, two points of different SEM morphology were analyzed (point 1 and point 2). Higher O and C concentration was found in point 1 than in point 2. This could be due to CO2 − o haemoglobin deposits on the membrane. Higher chloride content in point 2 than in point 1 could be explained because of the lower deposits in 2 than in 1, which allows the EDX technic to ‘see’ the ionic liquid of the membrane. These membranes act in the microbial fuel cell as proton exchange membranes. Considering that the active phase in the membrane is an ionic liquid, which is an organic salt, the transport mechanism could be explained by the solution diffusion model due to the interaction between the proton and the negative charge of the ionic liquid.

Slaughterhouse waste water treatment by the microbial fuel cell.

The evolution of the COD in the microbial fuel cells during the experimental run is illustrated in Fig. 4. As can be seen, the speed of removal of organic matter is high in the first 72 h. The experiments were maintained for 240 h (10 days). In absolute terms, the COD removal after 10 days of experiment was 72%. Despite being a remarkable value, however this level of COD conversion is not as high as those reported for urban and industrial wastewaters (between 85 and 95%) (8–12). One possible explanation for this lower value is inhibition of microbial growth due to the common presence of antibiotics in slaughterhouse waters (13,14). This fact could also be explained by the high COD of

the slaughterhouse wastewater compared with conventional wastewater. Regarding the COD removal rate (Fig. 4), this parameter is higher at the beginning of the experiments in the MFCs than the removal rate observed in the controls and baseline reactors. Since in these reactors only conventional anaerobic digestion is taking place, it seems reasonable to infer that the kinetic of the organic matter degradation is improved in the microbial fuel cell system with respect to the conventional anaerobic system. This could be explained because the biofilm formed and selected electroactive bacteria facilitates organic matter degradation more efficiently than the pool compounded by all kinds of bacteria in suspension and because degradation of the organic matter in the anode is facilitated by the catalytic reduction of oxygen in the cathode. In addition to the COD evolution, a number of physicochemical parameters were also monitored. Table 2 shows absolute values for the results of the characterization of these parameters at the end of the experiment both for the MFC and for the control and baselines. First, the results show moderate variation of pH and alkalinity both towards the alkalinity side. The pH of the anodic chamber decisively influences the metabolic activity and therefore affects the mechanism of proton and electron generation from electroactive bacteria. It is worth noting that protons are not accumulated in the anodic chamber and are transported through the ionic liquid membranes. With regards to the speciation of the main ions a different behaviour is observed among them. For example, it is interesting to note that the vast majority of ions decrease their concentration (nitrite, orthophosphate, sulfate and ammonium) by more than half. To explain this behaviour, we have to focus not only on conditions of oxidation in the anodic chamber but also on other phenomena such as the consumption by microorganisms in the biofilm. It is likely that more than one mechanism is acting on each ion so that results can be a consequence of several conflicting trends. The decrease in the orthophosphates values could be due to th conversion into organic phosphates because of the growth of microflora. As can be seen in Table 2 the nitrites and sulphate concentration is also reduced. In the absence of oxygen, nitrites and sulphates act as oxygen donors and also they could be used as nutrients for growing microorganisms. That reduction is more extensive in the microbial fuel cell than in the control and baseline which could be because growing the biofilm and water oxidation is faster in microbial fuel cells that in controls and baselines. The ammonium concentration is also reduced with respect to the initial values. This is also used as a nutrient by microorganisms, and the reduction is also higher in MFCs than in controls and baselines. Kjeldahl nitrogen is increased in microbial fuel cells, controls and baselines. The increase

in the former could be explained by the conversion of inorganic ammonium in organic ammonium. From Table 2 it can also be observed that total phosphate, nitrites, ammonium and sulphates ions concentration were higher in baseline and MFC (which contains graphitic granules) than in controls (without graphitic granules). In order to study this behaviour, 100 mL of graphitic granules were put in contact with 160 mL of distilled water for 10 days, and the ions content in the water analyzed. The results are shown in Table 3. As can be seen in Table 3, an increase in phosphate, nitrites, ammonium and sulphates ions was produced in water after contacting the graphitic granules. The release of anions from the graphitic granules could explain the higher concentration in systems containing graphiticmaterial. Surprisingly, anions such as chlorides, and hardness remained practically unchanged in the MFCs.

Electric performance of the microbial fuel cell

Voltage evolution during the experiments is reported in Fig. 5, which shows an initial induction period during the first 24–48 h in which voltage is low (lower than 50mV) corresponding to stage of rupture of long-chain molecules and formation of the biofilm. It is important to note that fresh slaughterhouse water was used rather than urban wastewater, the former containing a higher concentration of microorganisms. After that, a period of fast exponential growth is observed followed by a plateau where the electrical potential is maintained around 200mV. Figure 6 displays the polarization curve for this water after 96 h. As can be seen, the maximum power density obtained was 32 (mWm−3) corresponding to an internal resistance of 3.91 kΩ. The internal resistance value is not as lower than literature probably due to the high organic matter concentration of slaughterhouse wastewater and having in mind that the first 48 h could be considering in this case as an ‘activation time’. Starting the experiment at 48 h could decrease the value of the internal resistance and increase the maximum power density.15,16 Figure 7 shows the evolution over time of the coulombic efficiency for the different membranes tested. As can be seen from Figs 5 and 7, the behaviour of the coulombic efficiency was similar to that observed for the voltage. As the experiment progressed the coulombic efficiency increased. This behaviour could be explained by growth of the biofilm converting the organic matter into electricity (17).

CONCLUSIONS

The MFC, implemented with a polymeric ionic liquid membrane as separator in the cathode and slaughterhouse wastewater as fuel in the anode, presents good catalytic and electric efficiency in terms of COD removal and power production. The 70% $[MTOA^+][Cl^-]$ ionic liquid membrane has been successfully used in the MFC air-breathing cathode and it constitutes the key to its excellent performance as a proton exchange membrane. Outputs of 72% in COD conversion, 200 mV voltage and $32mWm^{-3}$ power density were obtained, showing that slaughterhouse wastewater could be a good feedstock for MFCs. A reduction in the ions concentrations of nitrite, orthophosphate, sulfate and ammonium was also found.

ACKNOWLEDGEMENTS

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Table 1. EDX spectrum data (normalized weight) of fresh samples and used (adter operation) samples (global, point 1, point 2) of 70 wt% [MTOA+][Cl-] membranes.

		70 w/w% [MTOA+][Cl-]		
Element	(Fresh)	After operation global	After operation point 1	After operation point 2
	C. Norm (%wt)	C. Norm (%wt)	C. Norm (%wt)	C. Norm (%wt)
C	36.05	36.57	38.99	37.02
N	13.76	14.16	11.36	12.02
O	9.27	39.24	36.15	29.50
Cl	40.91	1.07	4.51	14.28
Na	-	0.80	0.95	-
Mg	-	0.54	0.49	-
P	-	1.37	0.86	0.53
S	-	3.67	2.39	4.63
Ca	-	0.85	2.26	1.45
Fe	-	1.73	0.58	-
Al	-	-	0.84	0.35
Si	-	-	0.62	0.23

Table 2. Comparison of physicochemical parameters before and after water treatment in MFC, control and baseline.

Water parameter	Method	Initial	Final Values		
			MFC	Baseline	Control
pH	[-]	6.86	7.65	7.47	7.97
TSS, mg L^{-1}	AFNOR 90-105	1.57	1.52	1.46	1.24
COD, mg L^{-1}	AFNOR 90-101	5520	1500	1650	1650
Total phosphate, mg L^{-1}	AFNOR 90-022	43.56	52.63	53.54	57.17
Ortophosphates, mg L^{-1}	AFNOR 90-110	19.24	2.36	3.54	2.72
Kjeldhal nitrogen, mg L^{-1}	AFNOR 90-110	103.6	207.2	221.2	221.2
Nitrites, mg L^{-1}	AFNOR 90-013	3.41	0.48	0.6	0.98
Ammonium, mg L^{-1}	AFNOR 90-015	88.51	40.12	51.25	62.48
Sulphates, mg L^{-1}	Rodier	350.17	115.17	106.83	183.5
Chlorides, mg L^{-1}	AFNOR 90-014	1121.8	1121.8	1121.8	1121.8
Hardness, mg L^{-1} $CaCO_3$	Rodier	1036	1036	1436	1276
Alkalinity, meq L^{-1}	Rodier	167.6	223.6	239.6	127.6

Table 3. Phosphate, nitrites, ammonium and sulphates ion concentration in distilled water and after equilibration of 160 mL of water with 100 mL of graphitic granules.

	NO_2^- (ppm)	HPO_4^{2-} (ppm)	NH_4^+ (ppm)	SO_4^{2-} (ppm)
Distilled water	LD < 0.07	LD < 0.76	1.21	0.26
Sample 1	0.48	66.61	81.20	10.72
Sample 2	0.60	48.38	163.79	9.93

Figure 1. Schematic of the device used for these experiments.

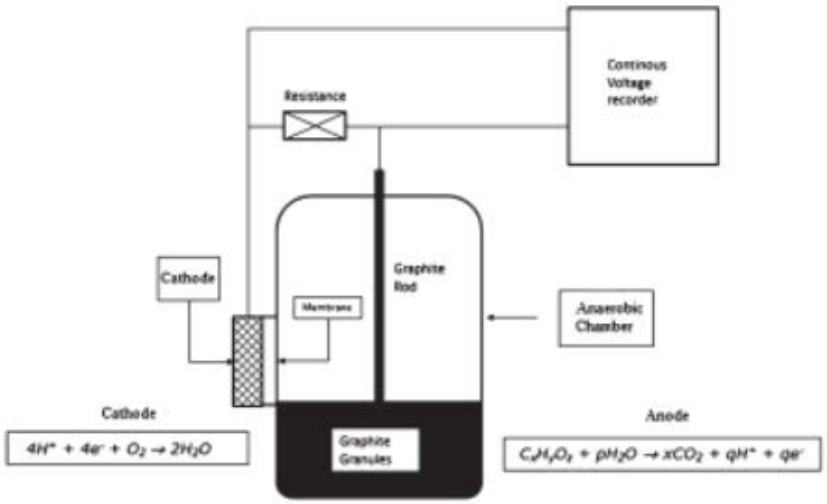
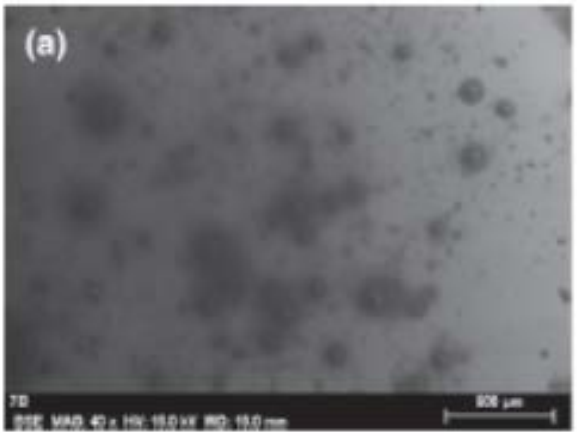


Figure 2. Scanning electron micrograph of the membrane with [MTOA+][Cl-] ionic liquid before (fresh, a) and after use (b) in the MFC 1 and 2 in Fig.2 (b) denote where EDX analysis was carried out (see below).



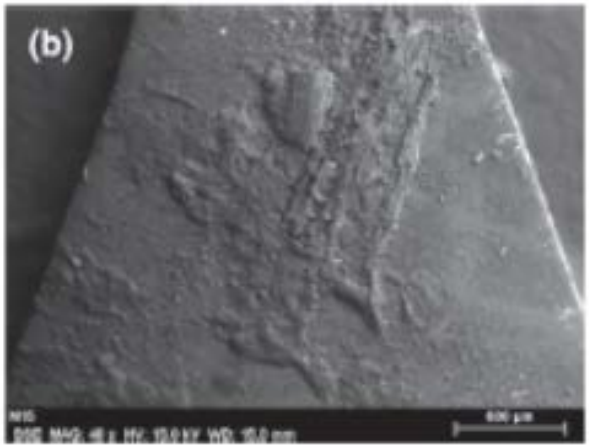


Figure 3. EDX spectra of the [MROA+][Cl-] (70 wt%) membrane, fresh and after operation (global, point 1 and point 2, see Fig.2.)

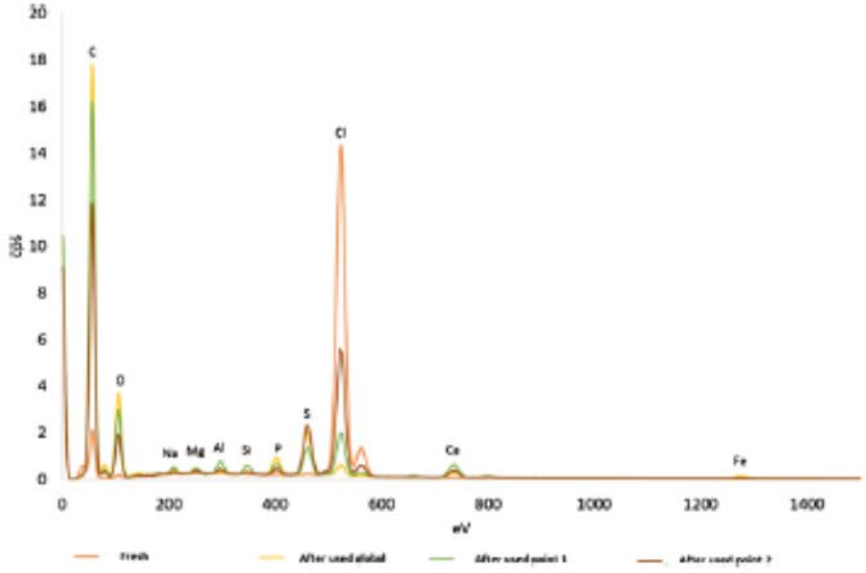


Figure 4. COD reduction in MFC with 70% (w/w) [MTOA+][Cl-] and PVC membranes, controls and baselines.

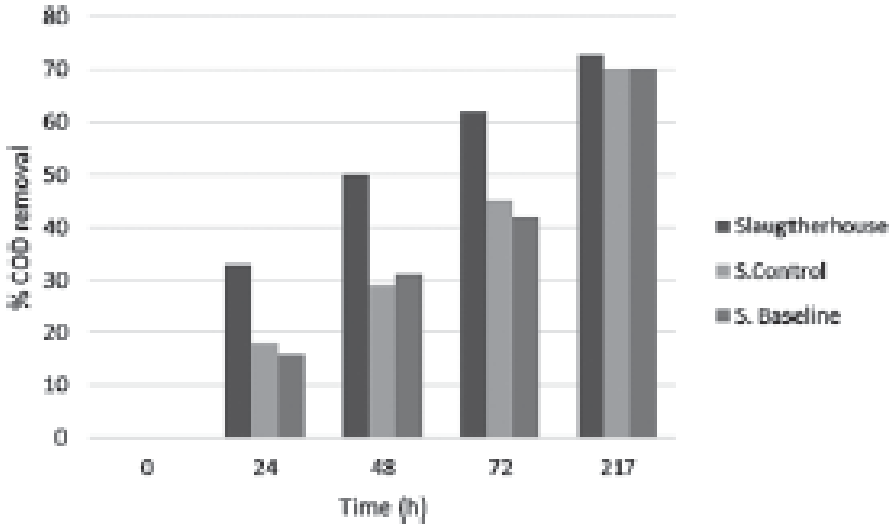


Figure 5. Voltage vs time in the microbial fuel cell using polymer inclusion membrane based on 70% (w/w) [MTOA+][Cl-] and PVC using slaughterhouse wastewater as feedstock.

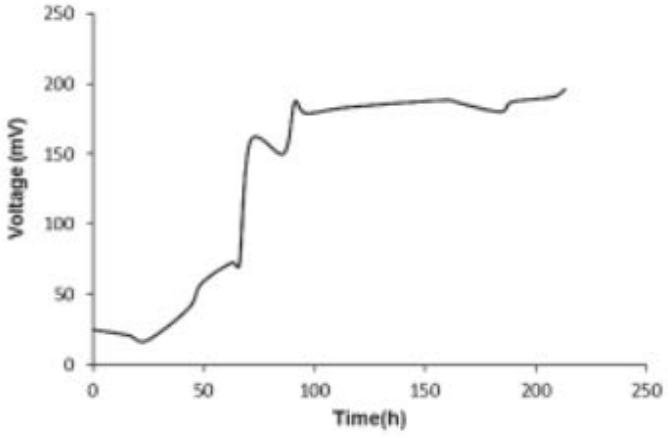


Figure 6. Polarizations of MFCs using slaughterhouse as feedstock and a polymer inclusion membrane based on [MTOA+][Cl-]. Polarization curve for MFCs (V vs. I) Power output curve (W vs. I). Calculations are based on anode liquid volume.

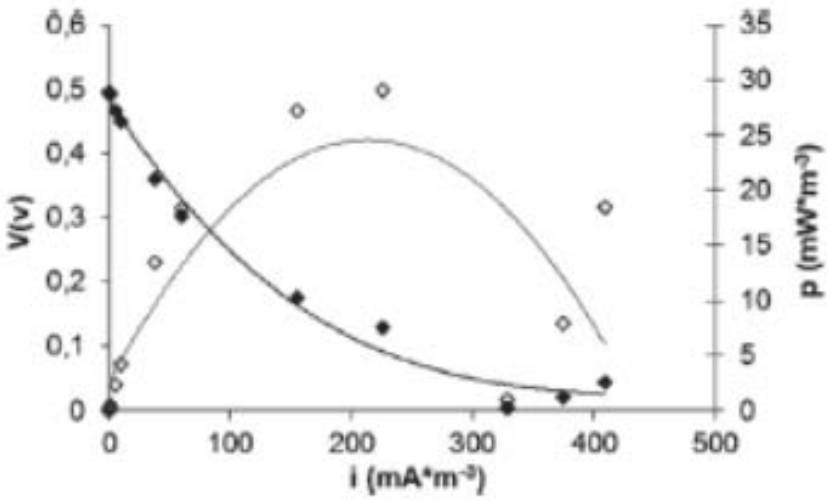


Figure 7. Profile of the coulombic efficiency (YQ) of the MFC using slaughterhouse as a feedstock and a polymer inclusion membrane based on 70% (w/w) [MTOA+][Cl-] and PVC.