

Alternative Proton Exchange Membrane Equipped in a Microbial Fuel Cell in Batch Operation

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ABSTRACT

At present, Nafion is the most commonly used membrane in *MFC* due its good transport properties. Yet, its commercial price is up to \$1470/m² and it determines *ca.* 40% of the *MFC* total cost. Therefore, the objective of this work was to compare the effect of membrane type on the performance of *MFC* operated in long batch process. The tested *PEMs* were Nafion 117 (*NF*) and a new organic membrane (*NOM*). The treated influent was a very recalcitrant, actual leachate from Mexico City sanitary landfill. The *MFC* was seeded with an inoculum previously enriched in electrochemically-active bacteria using a selective medium of Fe(III) salts and acetate. The batch lasted 15 days. *MFC* characteristics improved with time of operation; this suggested an in-cell enrichment process or acclimation of inoculum. During the batch operation, the cell equipped with *NOM* outperformed the one equipped with *NF*. In the first period, 0 to 8 d, average volumetric powers (P_V) were 9,000 and 4,000 mW/m³ for the *MFC* equipped with *NOM* and *NF*, respectively. Considering the subperiod from 8 to 15 d when the external resistances were adjusted, the average P_V were 20,000 and 6,800 mW/m³ for *NOM* and *NF*, respectively. At the end of the operational period, deposits of dry salts appeared on the external side of the cathode carbon cloth of the cell equipped with *NF*. This effect was not observed for the cell with *NOM*. Presumably, these deposits could be responsible for the decrease of power output during 11 to 15 d in the cell equipped with *NF*.

Keywords: Natural Organic Membrane; Batch Operation; Microbial Fuel Cell; Leachate



1. Introduction

Nowadays the humanity depends heavily on the use of petroleum oil and consequently faces two great risks: the inevitable depletion and the environmental pollution caused during exploration, transport, combustion of oil-based fuels. Thus, the investigation to develop new renewable energy sources as well as bioenergies has notably increased in the last years [1-3].

Microbial fuel cells (*MFCs*) are a promising technology that generates electrical power from a wide range of soluble substrates (organic or inorganic), wastes included. The *MFCs* constitute an interesting alternative to produce electrical energy and provide wastewater treatment simultaneously. [4-6].

In order to increase the *MFC* efficiency, several conditions of *MFC* operation and components have been the subject of intensive research such as the type of biocatalysts, membrane (electrolyte) or separators, temperature, pH, substrates, the type and materials of electrodes, electrode catalysts, cell configuration and architecture, among others [2,13-16].

The protonic exchange membrane (*PEM*) is an important part of *MFCs*. The main features and purposed of the membranes in *MFCs* are listed below [17-19]:

- to separate the anodic from the cathodic chamber in order to reduce the substrate flux from the anode to cathode, to avoid the back-diffusion of the electron acceptor, and to isolate the catalyst from the cathode in single-chamber *MFCs*
- to perform as a barrier to the transfer of other ions between the chambers
- to increase the Coulombic efficiency (*CE*) reducing the flux of the oxygen from the cathode chamber to the solution in the anode chamber
- to ensure an efficient and sustainable operation along time

Yet, there are disadvantages related to the *PEM* use, such as the high cost of standard membranes [17-19]. For instance, Nafion cost has increased up to \$1733/m² [20]. Furthermore, its use might affect negatively the power generated by the *MFC* due to the increase of the internal resistance (R_{int}) [18,19,21].

Nowadays, one of the challenges of the *MFCs* is the scaling up, but it depends of the performance *MFC* and cost materials [19,22]. So, in order to replace the *NF* as *PEM*, several polymeric membranes have been studied, such as ultrafiltration and microfiltration membranes, sulphonated polyether ether ketone membrane, anion and cation exchange membranes, bipolar membrane, forward osmosis membrane [2,13,18,19,22-25]. However, these polymeric membranes also are expensive.

Membrane-less *MFCs* have been studied because of a membrane is not strictly necessary in a *MFC*. The water conducts the protons by itself, however, the most of the works operated without a membrane, the *CE* is low [13,17-19,23]. Liu and Logan (2004) explored the bioelectricity generation in a membrane-less *MFC*, in order to increase the energy output and reduce the cost [17]. They reported a power density of 146 mW/m² and 20% of *CE* for their membrane-less *MFC*. In contrast, their *MFC* equipped with *NF* membrane displayed a power density of 28 mW/m² and 28% of *CE*.

Regarding new materials as *PEMs*, to reduce costs but to keep the *CE* and obtain an attractive volumetric power (P_v), there are few studies that have focused on glass fibers or glass wool, salt bridge, as well other materials and configurations such as assemblies [18,19,26,27].

Therefore, the objective of this work was to compare the effect of membrane type on the performance of *MFC* operated in long batch process, using actual leachates from Mexico City sanitary landfill and inoculum previously enriched (*E-in*) in electrochemically-active bacteria (*EAB*). The tested *PEMs* were a new organic membrane (*NOM*) and *NF* as reference.

2. Experimental

2.1 Experimental design

The experiment consisted of the operation in a long batch process of the *MFCs* equipped either with a *NOM* or *NF* (as control) as *PEMs*. The *MFCs* were packed with graphite flakes (*GF*) as anode and loaded with a mixture of inoculum previously enriched in *EAB* and a very recalcitrant, actual leachate from Mexico City sanitary landfill. The mix was in a proportion 80% inoculum and 20% actual leachate. The *MFCs* were operated along 15 d.

The long batch process was divided in two periods; first, from 0 to 8 d, and a second one, from 8 to 15 d. The first period was operated under the first external resistance (R_{ext}) defined by the first electrochemical characterization in time 0 d. The second electrochemical characterization was carried out at 8 d, then, the R_{ext} value was readjusted and the second period started.

The main response variables studied in the characterizations electrochemical were the maximum volumetric power ($P_{v,max}$) and the internal resistance (R_{int}) of the *MFCs*, whereas along the long batch process the average P_v , the chemical oxygen demand (*COD*) removal (η_{COD}), and the current (I_{MFC}) were the response variables analyzed.



The experiments were carried out at ambient temperature, with no mechanical mixing not heating and in a single compartment, air-cathode *MFCs*.

2.2. Microbial fuel cell

The *MFCs* were two horizontal cylinders built in Plexiglas 80 mm long and 57 mm internal diameter. The anodic chambers were packed with *GF* as anodic material with surface area of 0.28 m². Its anodic material was treated heating to 550 °C. The surface area was obtained as follow. A large sample of material was screened and the fraction collected was between meshes 10 and 6 (diameters 2 mm and 3.55 mm, respectively); of this fraction, five 20 g subsamples were taken and weighed. Afterwards, the number of particles in each subsample were determined and annotated. An average number of particles was estimated. With this number, the average weight of particle of each material was estimated and using the equations shown below, it was possible to calculate the surface area of the mass of material loaded into the *MFC*. The shape factor of the material (also called sphericity factor in other textbooks) was taken into account as described in Perry [28]. For instance, 0.43 was chosen for *GF*.

On the other hand, the net volume of the only anodic chamber in our *MFCs* was calculated as the geometric volume of the chamber minus the physical volume of the anodic material. With the surface area of the anodic material and the net volume, the specific surface area of the anode (A'_s) was finally calculated with Eq. 2 below

$$A'_s = \frac{\frac{M (6^2 m_p^2 \pi^3)^{1/3}}{\Phi_s (m_p^3 \pi^2 \rho^2)}}{V} = \frac{\frac{M (36\pi)^{1/3}}{\Phi_s m_p \rho^2}}{(V_{cell} - \frac{M}{\rho})} \quad (2)$$

where

- D_p average particle diameter, defined as the diameter of a sphere of the same volume as the particle
- Φ_s shape factor of the particle defined as the quotient of the area of a sphere equivalent to the volume of the particle divided by the actual surface of the particle
- m_p average weight of a particle of the given size fraction
- M total mass of anodic material loaded into the *MFC*
- ρ actual density of the material
- V_{cell} geometric volume of the cell chamber

The net volume of the *MFC* necessary for the denominator in the calculation of A'_s was estimated as described above in the denominator of Eq. 2. The cathode of the *MFCs* was a flexible carbon-cloth containing 0.5 mg/cm² platinum catalyst (Pt 10 wt%/C-EOTEK). On the air side, the cathode was limited by a perforated plate of stainless steel 1 mm thickness. In the liquid side, the cathode was in contact with the *PEM* (*NF* or *NOM*) [9,20].

The *NF* was pretreated to activate and to remove impurities before to use in the *MFC*. We describe a modified technique from Oh and Logan (2006) [29]. The membrane was soaked first in H₂O₂ (3% v/v), followed by soaking in deionized water, in 2 M H₂SO₄, and again in 1 h and deionized water, each stage for 1 h and at temperature of 80 °C. The *NOM* was fabricated and pretreated as reported elsewhere [20].

2.3. Enrichment of inocula

An enrichment procedure based on selective pressure using Fe (III) as an electron acceptor and sodium acetate as carbon source was implemented [20,30]. The departing inoculum consisted of soil sampled from an excavation made in the Centro de Investigación y de Estudios Avanzados del Instituto Politécnico Nacional (19°30'33"N, 99°07'46"O) at a depth of 2 m [31]. The soil was transferred to an anaerobic bottle, after this, 5 g of soil sample was suspended in anaerobic saline solution (50 mL); afterwards, 5 mL of sample was transferred to 50 mL metal-reduction medium with acetate as electron donor and Fe(III) oxide-hydroxide as electron acceptor. The enrichment of inocula was obtained with serial transfers [30,31].

Duplicate enrichments were incubated at 30 °C for 9 d in the dark condition. The enrichment procedure was repeated 3 times. The culture medium consisted of (g/L): 2.5 NaHCO₃, 0.25 NH₄Cl, 0.6 NaH₂PO₄·H₂O, 0.1 KCl, 10 mL vitamin solution and 10 mL mineral solution [32,33]. The Fe(III) oxide was synthesized as follows: a solution 0.4 M of FeCl₃·6 H₂O (pH adjusted to 7.0 with 10 M of NaOH) was added [33]. Finally, after the transferences, the *EAB* were transferred to a bioreactor of 1 L, it was incubated at 35°C in dark conditions. The inoculum was feeded every week and gassed with nitrogen to create anoxic environment [32].



2.4. Leachate

The leachates used as substrate were sampled from Mexico City sanitary landfill “Bordo Poniente”. Two types of leachates highly recalcitrant were provided: samples from Section 1 (L-1) and samples from Section 4 (L-4), where the denomination ‘section’ is related to the chronological construction of the landfill cells. Organic matter contents of leachates were 4300 and 12 300 mg COD/L for L-1 and L-4, respectively. The *MFC* was loaded with the sample from Section 4 (L-4) and their pH was slightly alkaline, *ca.* 8.0. The full characterization of the leachate is in Table 1 [31]. The relatively high organic matter content and high value of BOD₅/COD ratio indicated that the leachate is biodegradable and not quite aged [34]. Interestingly, we expected a lower pH consistent with fresh leachate. That was not the case. It is known that the “Bordo Poniente” landfill is emplaced in a site characterized by sodic-saline soil with pH of soil extract as high as 11. The local soil was likely used to cap the landfill cells during the daily operation of the landfill, possibly releasing sodium salts (carbonate, bicarbonate) as well as hydroxide that increased leachate pH. This explanation is supported by the high values of the electrolytic conductivity of the leachate (Table 1).

Table 1. Characteristics of municipal leachate.

Parameters	Value
pH	8.26 ± 0.02
Conductivity (mS/cm)	36.7 ± 0.1
Total Kjeldahl nitrogen (g/L)	2.9 ± 0.03
SO ₄ ²⁻ (g/L)	0.281 ± 0.01
COD (g/L) ^a	12.3 ± 0.5
BOD ₅ (g/L) ^b	10.6 ± 0.2
BOD ₅ /COD	0.86

Notes: ^a chemical oxygen demand; ^b biochemical oxygen demand

2.5. Electrochemical characterization of the microbial fuel cells

The *MFC* characterization was performed by duplicated. The internal resistance and power density curve of the *MFC* was determined by duplicate, using the polarization curve method by varying the external resistance and recording both the voltage and the current intensity [9,18,21,35].

The *MFCs* were operated at open circuit for 1 h; afterwards different resistors were varied, 10 to 1 MΩ and viceversa, to determine the power generation and another response variables as a function of load. After this, the cell was set to open circuit conditions for 1 h in order to check the adequacy of the procedure (values of initial and final open circuit voltages should be close). The voltage was measured and recorded with a Multimeter ESCORT 3146A.

The current was calculated by the Ohm's law (Eq. 3) and the R_{int} was calculated as the slope of the linear section of the curve voltage versus the current intensity [9,23]. The P_V was calculated according to the Eq. 4 below.

$$I_{MFC} = \frac{E_{MFC}}{R_{ext}} \quad (3)$$

$$P_V = \frac{(E_{MFC})^2}{V_{cell} \times R_{ext}} \quad (4)$$

where I_{MFC} is the current intensity of the *MFC* in A, E_{MFC} is the voltage delivered by the cell in V, R_{ext} is the external resistance connected to the cell in Ω and V_{cell} is the net volume of the anodic chamber.

The initial *COD* and biomass concentration in the *MFC* liquor were *ca.* 2000 mg O₂/L and 1900 VSS/L respectively. The pH and the electrical conductivity were 9 and 39 mS/cm, respectively. The *COD* and VSS of the inoculum enriched in Fe (III)-reducing bacteria seed bioreactor and *MFCs* were determined according to the Standard Methods [36].

3. Results and discussion

3.1. Electrochemical characterizations



The physical characteristics of the anodic material *GF* are showed in the Table 2. This anodic material has a large surface area *c.a.* 0.3 m^2 . The *MFCs* equipped with their respective *PEMs* were electrochemical characterized at 0 d, it leads to define the R_{ext} to start the power generation. The *MFC* equipped with *NOM* showed discouraging values (Fig. 1, Table 3.).

The R_{int} and $P_{v,max}$ were 650Ω and 9 mW/m^3 , whereas the *MFC* equipped with *NF* displayed a R_{int} and $P_{v,max}$ of 350Ω and 1100 mW/m^3 , respectively (Fig. 2, Table 3.). In this first test, the *NF* was far better than *NOM*. However, after 8 d, in the second electrochemical characterization the *MFC* equipped with *NOM* improve their performance, reaching a $P_{v,max}$ of $22,500 \text{ mW/m}^3$ (Fig. 1, Table 3.). The R_{int} decreased 94% (40Ω) and the $P_{v,max}$ increased 4 magnitude orders, it was very encouraging.

Table 2. Selected physical characteristics of graphite flakes.

Characteristics	Values
Working net volume (m^3)	$7.22 \times 10^{-5} \pm 5.05 \times 10^{-6}$
Anodic actual surface (m^2)	0.28 ± 0.08
A_s^a (m^2/m^3)	1302 ± 91
Conductance (S) ^b	0.13 ± 0.04

Notes:

^a Relationship between the anode surface area to cell volume, also known as specific surface area of the anode.

^b Electrical conductance of the material, expressed in Siemens.

On the other hand, regarding the second characterization of the *MFC* equipped with *NF*, the R_{int} and $P_{v,max}$ observed were 80Ω and $8,600 \text{ mW/m}^3$ (Table 3, Fig. 2). The R_{int} only decreased 77%, whereas the $P_{v,max}$ increased 87% respect the initial value. The *MFC* performance equipped with *NOM* at 8 d, was better than the *MFC* equipped with *NF*.

Table 3. Electrochemical characterization of microbial fuel cells equipped with different membranes.

Parameter	Time (d)			
	0		8	
	Membranes			
	NOM ^a	NF ^b	NOM	NF
R_{int} (Ω)	649.3 \pm 21.8	350.0 \pm 218.1	40.8 \pm 6.7	79.7 \pm 1.3
$P_{v,max}$ (mW/m ³) ^c	9.31 \pm 3.2	1142.7 \pm 379.2	22560.0 \pm 2727.0	8594.9 \pm 1069.8
I_{MFC} (mA) ^d	0.026 \pm 0.004	0.49 \pm 0.08	8.59 \pm 0.05	2.75 \pm 0.17
P_{cath} (mW/m ²) ^e	0.26 \pm 0.09	32.34 \pm 10.73	638.40 \pm 77.20	243.22 \pm 30.30
$E_{MFC,max}$ (mV) ^f	25.6 \pm 4.3	163.0 \pm 27.7	189.0 \pm 11.5	225.2 \pm 14.02
$E_{MFC,OC}$ (mV) ^g	29.4 \pm 19.4	380.0 \pm 29.5	619.4 \pm 47.7	549.7 \pm 28.3

Notes:

^a new organic membrane

^b Nafion 117 membrane

^c Maximum volumetric power.

^d Current intensity value at the maximum power.

^e Maximum power density based on surface area of electrode (cathode).

^f Potential value at the maximum power.

^g Open circuit potential.

3.2. Performance of the *MFC* equipped with *NOM* and *NF* in long batch operation

The influent characteristics are described in Table 4. In the first period, 0 to 8 d, the load resistances used were 470 and 680Ω for the *MFCs* equipped with *NF* and *NOM*, respectively (defined by previous electrochemical characterizations). The average volumetric powers (P_V) recorded were 9,000 and $4,000 \text{ mW/m}^3$ for *MFC* equipped with *NOM* and *NF*, respectively (Fig. 3). The *MFC* equipped with *NOM* reached an average P_V higher than *MFC* equipped with *NF*, although the values in the first characterization. The second electrochemical characterizations were carried out due these results.

In the second period, 8 to 15 d, new load resistances were applied, 82 and 47Ω in the systems using *NOM* and *NF*, respectively. In this period the *MFC* equipped with *NOM* reached an average P_V of $20,000 \text{ mW/m}^3$, almost the $P_{v,max}$ recording during the second electrochemical characterization. Furthermore, the P_V was stable along this period (Fig. 3).



On the other hand, the *MFC* equipped with *NF*, reached only an average P_V of $6,800 \text{ mW/m}^3$ during the day 8 to 11, after this time, the P_V began to fall down (Fig. 3). Its behavior could be explained because of from the 11 d, in the *MFC* equipped with *NF*, deposits of dry salts appeared on the external side of the cathode carbon cloth of the *MFC* equipped with *NF* (Fig. 4). The influent loaded to the *MFCs* had a high salinity and high pH (Table 1) and the *NF* was affected by this parameters. However, this effect was not observed for the *MFC* equipped with *NOM*.

These deposits could be responsible for the decrease of power output during 11 to 15 d in the cell equipped with *NF*. Another issue was that the water level inside of the *MFCs* decreased by evaporation losses through the *PEMs*. In order to maintain the original level inside of the *MFCs* and guarantee a full hydration of the *PEMs*, sterile distilled water was supplied every time it was necessary.

Regarding the organic matter (*COD*, Table 4) of the effluent, both *MFCs* seemed to have enough “fuel” to convert in electrical energy. The η_{COD} at the end of the batch operation were 39.32 and 28.29% for the *MFCs* equipped with *NOM* and *NF*, respectively. Once more, in this parameter, the *MFC* equipped with the *NOM*, was higher than the device with *NF*. The deposits of dry salts on the external side of the cathode carbon cloth of the *MFC* equipped with *NF* affect the performance of the *MFC* equipped with *NF*. Furthermore, the influent color at the end of the batch operation becomes clear in both systems (Fig. 5). Overall, the performance of the *MFC* equipped with *NOM* was much better than *MFC* equipped with *NF*.

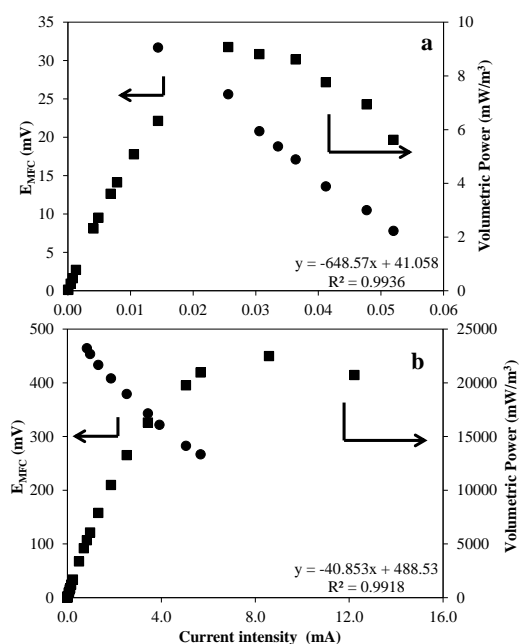


Fig 1. Electrochemical characterization of the *MFC* equipped with *NOM* at (a) 0 d and (b) 8 d.



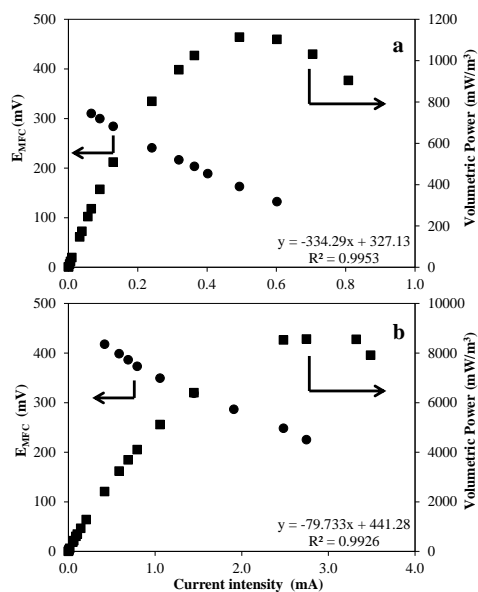


Fig 2. Electrochemical characterization of the *MFC* equipped with *NF* at (a) 0 d and (b) 8 d

Table 4. Influent and effluent characteristics of the *MFCs* equipped with both *PEMs*

Parameter	Time (d)		
	0		15
	Membranes		
	<i>NOM</i> and <i>NF</i>	<i>NOM</i>	<i>NF</i>
pH	9.12 ± 0.03	8.87 ± 0.02	7.68 ± 0.05
Conductivity (mS/cm)	38.65 ± 0.07	29.5 ± 0.05	30.6 ± 0.06
<i>COD</i> (mg/L)	2022 ± 99	1227 ± 511	1450 ± 193
Temperature ($^{\circ}C$)	28.50 ± 0.71	27 ± 0.66	26 ± 0.58



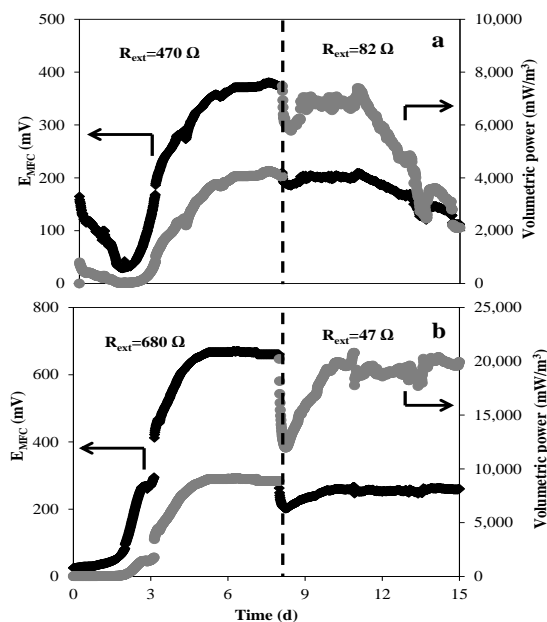


Fig 3. Time course of voltage and volumetric power outputs of MFCs using (a) *NF* and (b) *NOM*



Fig 4. Deposits of dry salts on the external side of the cathode carbon cloth of the MFC equipped with *NF* from the day 11th

In the open literature, there are reports on tests with organic membranes [26,27]. For instance, Min *et al.* (2005), using a pure culture of *Geobacter metallireducens* and domestic wastewater, examined the power produced by their MFC using a salt bridge as separator. The performance displayed by their MFC was 2.2 mW/m² with a high R_{int} 19,920 Ω [26]. Their system applied is very simple and attractive, however their MFC performance was poorer than that of our system (average powers 547 mW/m² and ca. 20,000 mW/m³ in the batch operation of our MFC equipped with *NOM*, Fig. 3).

In another work, Kargi and Eker (2007) used a salt-agar slab (salt bridge) in order to separate the chambers in their MFC [27]. The electrodes were made of Cu and Au-covered copper wires as anode and cathode, respectively. Although the high cost of their electrodes, the performance of their MFC was also too poor, the maximum power density registered was 2.9 mW/m². On the other hand Liu and Logan (2004) studied the power generation in an air-cathode MFC with and without (membraneless) a *NF*. Using wastewater as substrate and with *NF* as *PEM*, the power density reached was 28 mW/m², whereas the power density increased up to 146 mW/m² in the membraneless cell [17].

Sivasankaran and Sangeetha (2011) developed a sulphonated polyether ether ketone (SPEEK) to use in a MFC instead of *NF* [24]. The $P_{V,max}$ produced by their system, using dairy wastewater and domestic wastewater as influent were 5,700 and 3,200 mW/m³, respectively. Our average P_V obtained during the batch operation was ca. 20,000 mW/m³. It means our membrane could

be considered a very good option for using as a *PEM* in a *MFC* system.

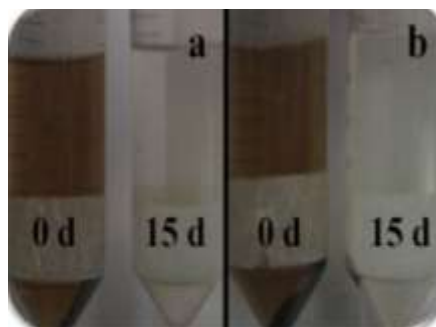


Fig 5. Visual evidence of leachate depuration after 15 d of batch operation in the *MFC* equipped with (a) *NOM* and (b) *NF*

4. Summary and perspectives

Our research was focused on the evaluation of a new membrane as *PEM* in order to replace the *NF*. The *NOM* tested in this work is a new alternative to use as *PEM* in *MFCs*. Along the batch operation, in a short time, the *MFC* performance using *NOM* delivered a volumetric power more than 2-fold the power of the *MFC* equipped with *NF*. Furthermore, the performance during the batch was more stable. Our new membrane is outstandingly more economic than *NF* and depicts good performance. Moreover, our *NOM* does not need any pretreatment; in contrast, the *NF* membrane requires a pretreatment with hydrogen peroxide and sulfuric acid that in turn generates hazardous wastes, besides the increased costs of membrane fabrication and conditioning.

Acknowledgements

The authors are grateful to CINVESTAV-IPN and SECITI-GDF (formerly ICYTDF), Mexico, for financial support to this research (PICCO-10-28). Giovanni Hernandez-Flores received a graduate scholarship from CONACYT, Mexico. Also the authors thank Mr. Rafael Hernández-Vera, and technicians of the Environmental of Biotechnology and Renewable Energy R&D Group, CINVESTAV-IPN for their excellent technical help.

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