Bioelectricity from Recalcitrant Municipal Leachate in a Microbial Fuel Cell

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ABSTRACT

The objective of this work was to evaluate the effect of several inocula on the treatment and bioelectricity production from municipal leachate in a two-face microbial fuel cell equipped with graphite flakes as anode and (*MFC-G*) and Pt as cathodic catalyst at a dose of 0.5 mg/cm^2 Pt. Inocula tested were: two enriched in Fe(III)-reducing bacteria (i.e., one was started with soil, *In-E*$_{Fe(III)-S}$, the other was started from a sulphate-reducing bioreactorn, *In-E*$_{Fe(III)-SR}$), one enriched in Mn(IV)-reducing bacteria (*In-E*$_{Mn(IV)}$), and a plain sulphate-reducing inoculum (*In-SR*). Each face (I and II) of the *MFC-G* was characterized by separate, in series, and parallel connection. Parallel connection of faces increased the maximum volumetric power up to 14 954, 24 319, 28 112 and 28 113 mW/m^3 for the *In-SR*, *In-E*$_{Fe(III)-S}$, *In-E*$_{Fe(III)-SR}$ and *In-E*$_{Mn(III)}$ respectively. Parallel connection of electrode faces also significantly decreased the R_{int}. In the batch operation where the cells were connected to an external resistance of 100 Ω, the average volumetric powers $P_{V\text{-}ave}$ were 26 424, 25 548, 25 752 and 13 379 mW/m^3 for the *In-E*$_{Fe(III)-S}$, *In-E*$_{Fe(III)-SR}$, *In-E*$_{Mn(IV)}$, and *In-SR* respectively. The high $P_{V\text{-}ave}$ achieved in our work with enriched inocula could be attributed to the combined effects of increased concentrations of exoelectrogenic bacteria as well as the high total anodic surface area by the use of granular graphite. This, in turn, could have improved the electron transfer microbe-to-anode. The power values registered in this work (26 W/m^3) were in the range of power yields typical of the anaerobic digestion of municipal wastewaters (5 to 50 W/m^3). To the best of our knowledge, it is the first time that volumetric powers as high as 26 W/m^3 are reported in the treatment of recalcitrant, actual leachate in *MFC*. Our results constitute a firm step towards sustainable remediation of this recalcitrant and aggressive effluent.

Keywords: enriched inocula; leachate; microbial fuel cell

1. Introduction

A microbial fuel cell (*MFC*) is an electro-biochemical reactor capable of directing converting organic matter into electricity [1-2]. Leachate is heavily polluted wastewater with a complex composition containing four groups of pollutants: dissolved organic matter, inorganic macro-components, heavy metals and xenobiotic organic compounds [3]. Leachate is generated from the landfill, composting and pyrolysis pretreatment of municipal solid waste. This sewage is always mixed with the liquid from microbial anaerobic decomposition, settlement of rainwater and surface water lixivium, which can be named as leaching water or leaching solution. This complex composition and the variance in quality and quantity usually observed, make leachate one of the most difficult of wastewaters to be treated [4]. All kinds of leachate treatment options have been reported such as *(i)* leachate recirculation, *(ii)* co-treatment with domestic wastewater, *(iii)* physico-chemical systems, *(iv)* biological processes, and *(v)* combined treatments [4]. Because of the high costs, generation of waste sludges, as well as emission of objectionable odors of the ordinary treatment, finding an efficient, stable and economic method for municipal landfill leachate treatment is very necessary.

The objective of this work was to evaluate the effect of several inocula on the treatment and bioelectricity production from municipal leachate in a two-face microbial fuel cell equipped with graphite flakes as anode and (*MFC-G*) and Pt as cathodic

catalyst. Inocula tested were: two enriched in Fe(III)-reducing bacteria (i.e., one was started with soil, *In-*$E_{Fe(III)-S}$, the other was started from a sulphate-reducing bioreactorn (*In-*$E_{Fe(III)-SR}$), one enriched in Mn(IV)-reducing bacteria (*In-*$E_{Mn(IV)}$), and a plain sulphate-reducing inoculum (*In-SR).*

2. Experimental

2.1. Microbial fuel cell architecture

The *MFC-G* consisted of a horizontal cylinder built Plexiglass 90 mm long and 57 mm internal diameter. The opposing faces of the cylindrical shell were fitted with corresponding sets of an assemblage of (inside to outside) proton exchange membrane (Nafion 117), a Toray flexible carbon-cloth containing 0.5 mg cm^{-2} platinum catalyst (Pt 10 wt%/C-ETEK), and a perforated plate of stainless steel 1 mm thickness. Each assemblage was corresponded with anodes made of granular graphite and a graphite rod as collector (80 mm long and 5 mm diameter). The average separation between cathode-anode in *MFC-G* was 17.5 mm. The anode chamber volume was 100 mL.

2.2. Municipal leachates

The cells were loaded with 20 mL of municipal leachates sampled from the Mexico City landfill "Bordo Poniente" stage 4. The characterization of the leachate is given in Table 1. The relatively high organic matter content and high value of BOD5/COD ratio indicated that the leachate is biodegradable, and likely not very aged. Interestingly, we expected a lower pH consistent with fresh leachate [4]. That was not the case. It is known that the landfill is emplaced in a site whose soil is sodic-saline soil with pH as high as 11 [5]. 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 hydroxides 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)	12.3± 0.5
BOD_5 (g/L)	10.6± 0.2
BOD_5/COD	0.86

2.3. Sample collection and enrichment of inoculum

The enriched inoculum was obtained after three serial transfers, described below. Sample from soil was collected in the CINVESTAV-IPN (19° 30´33"N, 99° 07´46"O) to a depth of 2 m. The sample was transferred to an anaerobic bottle. 5 g of soil sample was suspended in 50 mL of anaerobic saline solution; afterwards, a 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. A sample of sulphate-reducing sludge was taken from a complete mix sulphate reducing semi-continuous bioreactor that acted as seed reactor [5-7] and handled in a similar way as the soil sample. The enrichment of inoculum were obtained with serial transfers. Duplicate enrichments were incubated at 30 °C for 9 d in the dark condition. The enrichment procedure was repeated 3 times. For the procedure of enrichment Mn(IV) of soil sample was similar way as the enrichment Fe(III). Duplicate enrichments were incubated at 30 °C for 15 d in the dark condition. The enrichment procedure was repeated 3 times.

The culture medium consisted of (g/L): 2.5 $NaHCO_3$, 0.25 NH_4Cl, 0.6 $NaH_2PO_4.H_2O$, 0.1 KCl, 10 mL vitamin solution and 10 mL mineral solution (Lovley & Phillips, [8]; Lovley & Phillips, [9]). The Fe(III) oxide was synthesized as follows: a solution 0.4 M of $FeCl_3.6\ H_2O$ (pH adjusted to 7.0 with 10 M of NaOH) was added, according to the technique described by Lovley & Phillips [8]. The MnO_2 was synthesized by slowly adding a solution of $MnCl_2$ (30 mM) to basic solution of $KMnO_4$ (20 mM) which was stirred with a magnetic stir bar. This procedure is similar to a previously described technique (Lovley & Phillips, [8]).

2.4. Electrochemical characterization and batch operation of the microbial fuel cells

Potential sweep experiments were carried out from open-circuit cell voltage (EOC), to the final potential of 0.02 V at a scan rate of 1mVs-1, performed in a potentiostat/galvanostat Voltalab model PGZ402 [10,11]. Values of R_{int} were estimated from the slopes of corresponding regression lines selected in the linear range of the polarization curves. The current (I_{MFC}), power (P_{MFC}) and volumetric power (P_V) were calculated as previously described [12]. The power density (surface area) was normalized to the projected cathode surface area (surface power density P_S). The cells were loaded with 80 mL of inoculum and 20 mL of actual municipal leachate. The initial biomass concentration in the cell inoculum was *ca.* 1,300 mg $VSSL^{-1}$.

The batch *MFC*s were loaded with municipal leachate and i enriched inoculum. The organic matter concentration in MFCs was ca. 2 g CODL-1. The cells were loaded with 80 mL of inoculum; the initial biomass concentration in the cell inoculum was ca. 1,300 mg $VSSL^{-1}$. The cells were operated for two Periods; in Period I the cells were run until a decrease organic matter concentration was observed. At the end of Period I, the cell was loaded with new municipal leachate (ca. 2.0 g $CODL^{-1}$) although the electrodes and membrane as well as the microbial community remained the same, and further operated for what we denominated In Period II. At the start of Period I the cells were operated for 24 h to open circuit voltage. Afterwards the cells were batch-operated for a total 250 h, at ambient temperature (23°C average) without mixing. The circuit of each MFC was fitted with a corresponding external resistance of 100 Ω for Pt.

The main variable responses of this experiment were the average volumetric power ($P_{V\text{-}ave}$), the efficiency of organic matter removal (η_{COD}), and the coulombic efficiency (η_{Coul}).

2.5. Analytical methods and calculations

The COD and VSS of the liquors of sulphate-reducing seed bioreactor and cells were determined according to the Standard Methods [13]. Manganese (Mn II) contents were analyzed by the method of Brewer and Spencer [14] as modified by Armstrong et al. [15] whereas the presence of Mn (IV) was assessed with a benzidine acetate reagent [16]. The main response variables were calculated as reported elsewhere [2].

3. Results and discussion

3.1. Electrochemical characterization of microbial fuel cells loaded with municipal leachates.

Each face (faces I and II) of the *MFC-G* was characterized by separate (I and II), in series, and parallel connection. Parallel connection of faces increased the maximum volumetric power ($P_{V\text{-}max}$) up to 24,319 and 28,112, 28,799, and 14, 984 mW/m^3 for *In-*$E_{Fe(III)\text{-}S}$, *In-*$E_{Fe(III)\text{-}SR}$, *In-*$E_{Mn(IV)}$ and *In-SR*, respectively (Fig. 1). In general, parallel connection of electrode faces significantly decreased the R_{int} (18, 13, 10 and 19 Ω for *In-*$E_{Fe(III)\text{-}S}$, *In-*$E_{Fe(III)\text{-}SR}$, *In-*$E_{Mn(IV)}$ and *In-SR*, respectively).

The $P_{V\text{-}max}$ for the *MFC-G* for *In-*$E_{Fe(III)\text{-}S}$, *In-*$E_{Fe(III)\text{-}SR}$, *In-*$E_{Mn(IV)}$ and *In-SR* when faces were connected in series and parallel were higher than that reported by Ortega-Martínez *et al.* [17], Vázquez-Larios *et al.* [178, Ortega-Martinez *et al.* [19] and Ieropoulos *et al.,* [20] . Ortega-Martinez *et al.* [17] evaluated the effect of an enriched inoculum on the characteristics of a parallelepiped *MFC*. The values of $P_{V\text{-}max}$ were 1,772 and 5,804 mW/m^3 for the faces connected in series and parallel. Vazquez-Larios *et al*. [18] evaluated a MFC equipped with a cathode painted with Pt as a catalyst, graphite triangular pieces as anodic material, inoculum sulphate-reducing and semisynthetic leachate. The $P_{V\text{-}max}$ for faces I and II connected in parallel and series were 2,094 and 3,098 mW/m^3. Ortega-Martinez *et al.* [19] characterized a multi-face parallelepiped *MFC*; this cell was fitted with a 'sandwich' cathode-membrane-anode assemblage in five of their faces. When the 5 faces of the cell were connected in series and parallel, the $P_{V\text{-}max}$ achieved values of 62 and 570 mW/m^3. Ieropoulos *et al.,* [20] compared the performance of two small size *MFCs* connected in parallel using acetate as the substrate and anaerobic sludge and regiested a $P_{V\text{-}max}$ of 860 mW/m^3.

The relatively high value of $P_{V\text{-}max}$ obtained in our *MFCs* could be attributed to the increase of the total electrode surface area by the application of granular graphite, and this, in turn, could have improved the electron transfer microbe-to-anode process [18]. Also, our work demonstrated that parallel connection of cell faces was more appropriate. On the other hand, the increase of power could be ascribed to the use of the enriched inoculum. Dissimilatory metal reducing bacteria, were capable of the reduction of soil metal oxides have been particularly noteworthy with regard to this ability and to date, many species of electrochemically active bacteria [19].

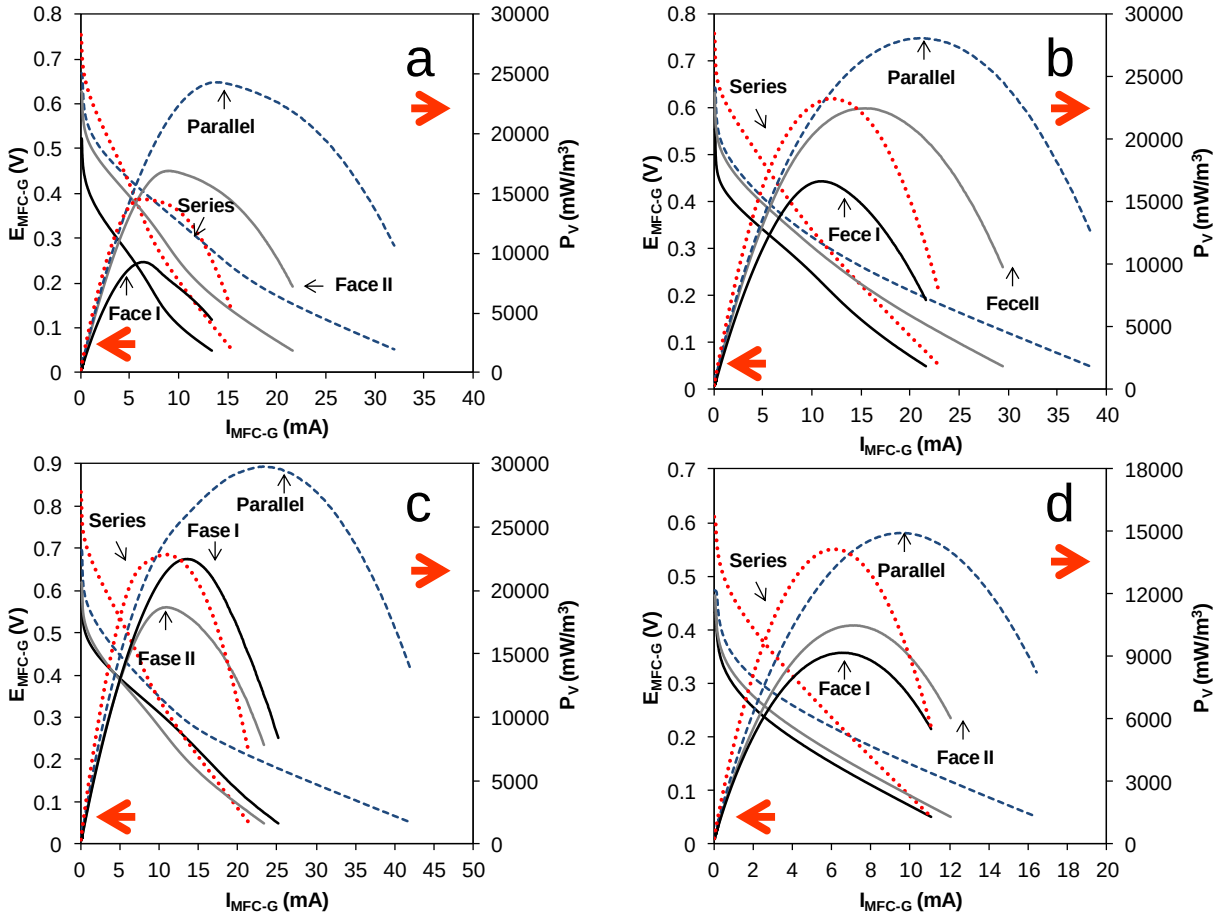

Fig. 1. Polarization curves by linear sweep potential studies in electrochemical characterization of microbial fuel cells, a) *In-*$E_{Fe(III)\text{-}S}$, b) *In-*$E_{Fe(III)\text{-}SR}$, c)*In-*$E_{Mn(IV)}$ and d) *In-SR*. Keys: convex curves stand for volumetric power, read on the right axis; descending parabolic curves represent the E-I relationship, read on the left axis; curve in blue-hyphen stand for parallel connection of the two faces of the cell; red-dot stands for series connection of the faces; continuous gray corresponds to face II alone, and continuous black stands for face I alone.

3.2. Results of batch operation of microbial fuel cells loaded with municipal leachate

Figure 2 shows the time course of cell potential the *MFC-G* in the batch operation of the cells using two enriched inocula, namely, the inoculum enriched in Fe (III)-reducing bacteria (either one soil origin, *In-*$E_{Fe(III)\text{-}S}$ or sulphate-reducing origin, *In-*$E_{Fe(III)\text{-}SR}$), inoculum enriched in Mn (IV)-reducing bacteria, sulphate reducing and *MFC-G* without inoculum. The cells were connected to a R_{ext} of 100 Ω whereas their two faces of each cell were connected in parallel. The gray area in Fig. 2 shows that the open circuit potential (the 21 h and without a resistance in the external circuit) of the cells were 0.516, 0.499, 0.500, 0.358 and 0.061 V for *In-*$E_{Fe(III)\text{-}S}$, *In-*$E_{Fe(III)\text{-}SR}$, *In-*$E_{Mn(IV)}$, *In-SR* and without inoculum, respectively. Two repeated cycles were carried out (Period I and Period II in Fig. 2), where treated leachate was removed and replaced by a new leachate load, whereas the microbial culture and electrodes remained the same.

The $P_{V\text{-}ave}$ for period II the cells *In-*$E_{Fe(III)\text{-}S}$, *In-*$E_{Fe(III)\text{-}SR}$, *In-*$E_{Mn(IV)}$ and *In-SR* were higher than those reported by Zhang *et al.* [21], Puig *et al.* [22], Rikame *et al.* [23], Özkaya *et al.* [24] and Vazquez-Larios *et al.* [25]. Zhang *et al.* [21] evaluated an

upflow air-cathode membrane-free microbial fuel cell (UAMMFC). The experimental results demonstrated that the *UAMMFC* could continuously generate electricity from leachate for an operational period of time (50 h); a maximum volumetric power of 12 800 mW/m^3 was reported. Puig *et al*. [22] operated an air, single chamber *MFC* and Pt catalyst in the cathode to treat landfill leachate in two operation periods. In Period I with 0.507 g COD/L, the authors reported a P_V of 6.1 mW/m^3. In their Period II the organic matter content was increased to 1.48 and 8.51 g COD/L, the P_V were 106 to 344 mW/m^3, respectively.

Rikame *et al*. [23] experimented with a two-chambered *MFC* with carbon electrodes for food waste leachate, the P_V was 15,140 mW/m^3 (η_{COD} = 90%). Özkaya *et al*. [24] assessed the performance of a two-chambered, continuous *MFC* with new Ti–TiO_2 electrodes for bioelectricity generation from young landfill leachate. They found a P_V of 1 920 mW/m^3. Vazquez-Larios *et al*. [25] evaluated a new design with extended electrode surface (larger ξ, specific surface or surface area of electrode to cell volume) and the assemblage or "sandwich" arrangement of the anode-*PEM*-cathode (*AMC* arrangement) and model leachate, similar to that produced in the biological hydrogen production from the organic fraction of the municipal solid waste. A P_V of 606 mW/m^3 was achieved.

The high average volumetric powers achieved in our work could be attributed to the combined effects of increased concentrations of exoelectrogenic bacteria as well as increased total anodic surface area by the use of graphite flakes (Vazquez-Larios *et al*., [18]).

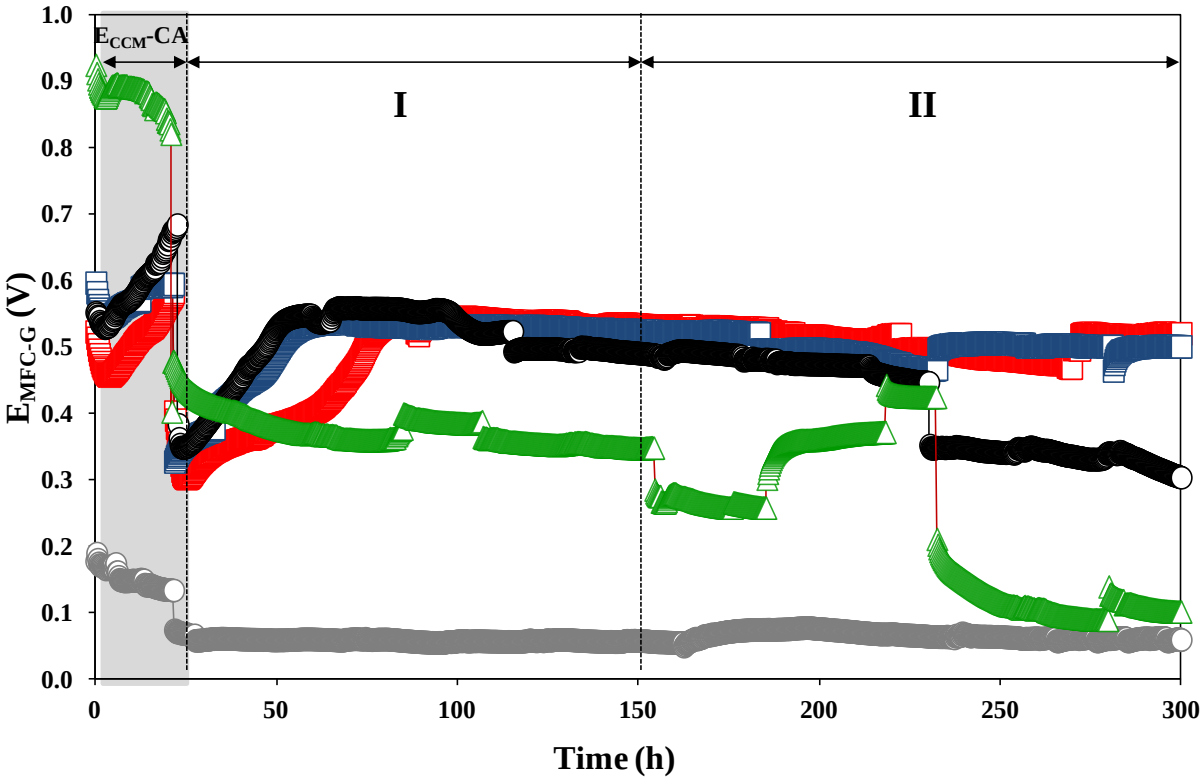

Fig. 2. Time course of voltage outputs of *MFC-G* cell using a inoculum enriched in Fe (III)-reducing bacteria (either one soil origin, *In-*$E_{Fe(III)\text{-}S}$ and sulphate-reducing origin, *In-*$E_{Fe(III)\text{-}SR}$), inoculum enriched in Mn (IV)-reducing bacteria (*In-*$E_{Mn(IV)}$), inoculum sulphate reducing (In-SR) and without inoculum.

4. Summary and perspectives

Two-face *MFCs* fitted with air-cathodes and graphite flakes anodes were characterized and batch operated for bioelectricity generation from an actual leachate. Moreover, the effect of four inocula, the enriched in Fe(III)-reducing bacteria (either one from soil origin, *In-*$E_{Fe(III)\text{-}S}$ or sulphate-reducing origin, *In-*$E_{Fe(III)\text{-}SR}$) inoculum enriched in Mn (IV)-reducing bacteria (*In-*$E_{Mn(IV)}$), inoculum sulphate reducing (*In-SR*) was assessed.

Each face (I and II) of the *MFC-G* was characterized by separate (I and II), in series and parallel connection. Parallel connection of faces increased the max volumetric power up to 24 319 and 28 112, 289 799, 14 984 mW/m^3 for *In-*$E_{Fe(III)\text{-}S}$, *In-*$E_{Fe(III)\text{-}SR}$, *In-*$E_{Mn(IV)}$ and *In-SR*, respectively, respectively. In general parallel connection of electrode faces significantly decreased the R_{int} (18, 13, 10 and 19 Ω for *In-*$E_{Fe(III)\text{-}S}$, *In-*$E_{Fe(III)\text{-}SR}$, *In-*$E_{Mn(IV)}$ and *In-SR*, respectively).

In the batch operation where the cells were connected to an R_{ext} of 100 Ω, the average volumetric power $P_{V\text{-}ave}$ were 26 424 ± 482, 25 548 ± 744, 25 752 and 13 379 mW/m^3 for *In-*$E_{Fe(III)\text{-}S}$, *In-*$E_{Fe(III)\text{-}SR}$, *In-*$E_{Mn(IV)}$ and *In-SR*, respectively. The high $P_{V\text{-}ave}$ achieved in our work could be attributed to the combined effects of increased concentrations of exoelectrogenic bacteria as well as increased total anodic surface area by the use of graphite flakes. This, in turn, could have improved the electron transfer microbe-to-anode.

The power values achieved in this work (30 W/m^3) were higher or comparable than power thresholds typical of the anaerobic digestion of municipal wastewaters (5 to 50 W/m^3). To the best of our knowledge, it is the first time that volumetric powers as high as 26 W/m^3 are reported in the treatment of actual leachates in MFC. Our results constitute a firm step towards sustainable remediation of recalcitrant leachates.

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