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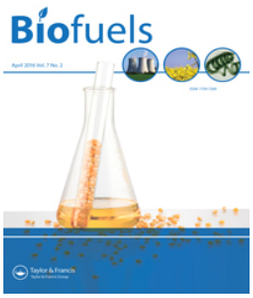

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Stacked-MFC into a typical septic tank used in public housing

Liliana Alzate-Gaviria*, Orlando García-Rodríguez, Manuel Flota-Bañuelos, Francisco Del Rio Jorge-Rivera, Gerardo Cámara-Chalé and Jorge Domínguez-Maldonado

Yucatan Center for Scientific Research (CICY), Renewable Energy Unit, Calle 40 No. 130, Colonia Chuburná de Hidalgo, 97200 Mérida, Yucatán, Mexico

ABSTRACT

To test the viability of incorporating a stacked-microbial fuel cell (SMFC) within a typical septic tank, 15 cartridges of MFCs with proton exchange membrane without catalysts were installed in a real system considered with a pretreatment. Both chemical oxygen demand (COD) removal and electricity generation using super capacitors by electricity storage were investigated under continuous flow mode. Three MFCs A1, B4 and C2 with 109.40 ± 34.25 mW/m^3, 131.58 ± 27.75 mW/m^3 and 124.01 ± 27.57 mW/m^3, respectively, were chosen for testing. The organic loading rate was 0.24, 0.52 and 1.05 kg DQO/m^3-d corresponding to 200, 500 and 1000 ppm. Total COD removal and total coulombic efficiency were 89.67 ± 5.19% and 48.07 ± 2.33%. The results of this study suggest that MFCs may be suitable for deployment in a septic tank. This research has demonstrated the great challenges in applying a stack of MFC in scale-up. R_{int} showed that anode resistances are higher than cathode; however, the configuration seems to be indicated for this kind of system depuration in developing countries. An electrochemical model must be developed for scale up which explains performance and electrochemical data. It is necessary to develop experiments in scale up and to test the feasibility of implementation.

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stacked-microbial fuel cells; scale-up; septic tank

1. Introduction

Water is one of the basic elements for the existence of life because it is impossible for biological processes to be performed without it. For centuries water was considered an infinite resource but is becoming scarce in areas where previously it was not. The total amount of fresh water supply in urban areas in Mexico was 12,000 million m^3 in 2014, while the total volume of discharged wastewater in urban areas was 6,623 million m^3, which was about 55.20% of the total water supply in urban areas (the data is collected from National Water Commission-CONAGUA 2014). This indicates that more fresh water could be saved in production if wastewater in urban areas could be effectively purified. Mexico treated about 60% of wastewater; however, in the state of Yucatan this percentage is less than 10%. [1] Water has now been recognized as a strategic issue involving national security and has become a central element to the current environmental and economic policies, as well as a key factor of social development. Surface water must be kept free from wastewater discharges in order to avoid affecting their natural capacities of assimilation and dilution and to ensure that all water resources in the country regain their health.

Mérida city has soils of permeable calcareous sedimentary rocks that hinder the installation of sewerage systems, due in part to the impact of a meteorite of Baptistine family 65 million years ago which formed a crater of 180 kilometers in diameter forming one of the biggest impact areas of the world (Chicxulub Crater).

Wastewater generated in Yucatan homes is arranged in septic tanks but is a potential source of water pollution. However, their contribution to freshwater eutrophication and impacts on human health are uncertain and difficult to quantify.[2]

The only source of drinking water is the karstic aquifer beneath the city. This aquifer is vulnerable to contamination by these effluents.[3] One solution to this problem is to implement technology of microbial fuel cells that are basically bioreactors that use bacteria as electrocatalysts to convert residual biomass into bioenergy.[4]

One additional challenge in the use of microbial fuel cells (MFCs) for domestic wastewater treatment that has not received sufficient attention is the performance of reactors containing many electrodes.[5–9]

Likewise, Nafion 117 membrane is still undeniably the most commonly used membrane in MFC applications because of its good conductivity and should be modified to improve its properties, such as preventing biofouling formation on its surface easily and reducing oxygen and substrate crossover. Until now, none of the membrane separators have been able to avoid the formation of biofilm on their surfaces. Hence, the

CONTACT Liliana Alzate-Gaviria lag@cicy.mx

membranes have to be replenished once they are severely fouled; indirectly increasing the MFC's cost. [10]

The objective of this study was to design, assemble, install and check performance (in terms of removal of organic matter and energy generation) of a stack of microbial fuel cells in a conventional septic tank design used in Yucatán, Mexico.

2. Experimental

2.1. Stacked-MFC (SMFC) construction

MFC construction type PEM, passive air cathode MFCs (2 L anode and 0.5 L cathode), was constructed as described. Each MFC contained granular carbon and stainless steel mesh (size 400×400, alloy 316) using metal mesh current collectors.

Each MFC contained two cylindrical chambers: anode chamber with a diameter of 10 cm by 40 cm with SW as affluent and the cathode with a diameter of 8 cm by 20 cm and synthetic greywater (SGW) as influent. The stack of 15 MFC's was separated by a proton exchange membrane (PEM) Nafion® 117 with a diameter of 7.7 cm. Assembling was done on a flat support with fixing holes which prevented deforming and leaking. The cathode is contained in the anode chamber so that the membrane is in contact with both anolyte and catholyte. The anode's bottom was reduced in size because it was necessary to adapt it to a second chamber of the septic tank. The influent of anode was at the bottom (upstream) and effluent at the top. The cathode was fed by the top (downstream) and its effluent at the bottom. The electrodes were placed parallel to the flow path in continuous flow operation.

2.2. SMFC within typical septic tank

The material used for civil works was of conventional construction such as cement, sand, gravel and smooth finished walls. The total volume of the septic tank was 2.93 m^3 with a useful volume of 2.44 m^3.

SMFC consisted of 15 individual cells distributed in the second chamber, which initially correspond to the filter holes.

The septic tank was covered with polycarbonate lids to prevent entry of rain water, insects and trash, while allowing for the achievement of anaerobic conditions and quick access for physical chemical and electrochemical tests.

2.3. SMFC operation

Leak testing of the septic tank was performed according to Mexican law (NOM-006-CNA-1997).[11]

The oxygen used in cathodes comes from SGW with a concentration of approximately 5 ppm, according to literature.[12,13]

SW and mixture inoculum [14,15] were added in the first chamber of the septic tank.

The system was initially operated in batch mode for 15 days and 22 days in continuous flow (Start-up). A carbon source (glucose) was provided every 24 hours and thereafter continuously fed for 90 days with SW concentration as follows: 200, 500 and 1000 ppm.[16]

The design was made for a house inhabited by five people and the hydraulic system had a gravity discharge flow from its tributary until effluent.

2.4. Energy storage

Due to the fact that SMFC shares the same anolyte, it is not possible to add the voltages of the 15 MFC's. Therefore, the energy generated by each MFC was harvested externally through a card (supercapacitors connected in series) that allows storage of energy and voltage rise at the same time.[17] The electronic component used was a charge pump circuit topology consists of analog switches to control the connections of voltages to several capacitors. The basic 2x boost charge pump is shown in Figure 1. For this simple boost application, analog switches SW1, SW2 and SW3 are closed when the control is asserted, charging C1 and C2 to the voltage present at VIN. This is typically called the charge phase. During the second phase switches SW1, SW2 and SW3 are opened when the switch control is deasserted, connecting C1 and C2 in series resulting in VOUT which is effectively double the voltage of VIN. The pulsing or switching noise is filtered by the capacitor COUT.[18]

2.5. Calculations and measurements

Polarization curves and impedance

To measure the internal resistance, the SMFC was connected to a Biologic VSP potentiostat in a three-electrode mode: one electrode serving as the working

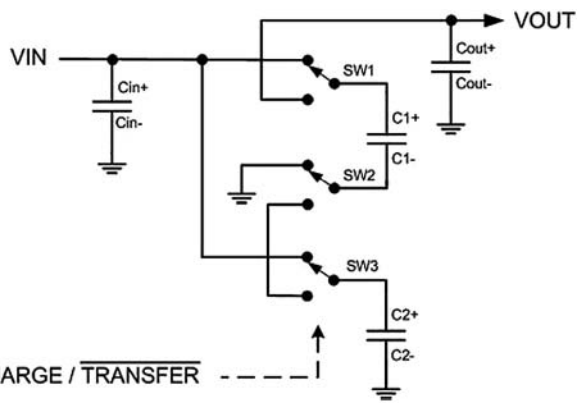

Figure 1. The simplified 2x charge pump topology.

electrode (anode or cathode) and the others acting as reference (saturated calomel) and counter electrodes (platinum mesh). The electrochemical impedance spectra (EIS) measurements were performed by using an Interface instrument Biologic VSP. After this, each MFC was operated under open circuit conditions for 2 h. The impedance spectra were recorded in the frequency range from 0.01 to 100,000 Hz by applying a sinusoidal excitation signal of 10 mV. The data were fitted to an equivalent electrical circuit by using the EC-Lab® (version 10.37) impedance analysis software.

Chemical analyses

The pH and temperature were measured with a Thermo Scientific Orion® multiparameter meter. Chemical oxygen demand (COD) was measured with the potassium dichromate in digestion solution technique (high range COD reagent from 0 to 15,000 ppm).

3. Results and discussion

The start-up was done with 200, 500 and 1000 ppm, respectively, and finished when three periods or cycles of electricity generation were carried out and SMFC were acclimated with the same external resistance (1000 Ω).[19] The nomenclature used for naming the each MFC is from 1 to 5 for rows and A, B and C for the three columns (Figure 2). Each fed-batch cycle was typically 4−5 days.[8]

The organic loading rates were 0.24, 0.52 and 1.05 kg DQO/m^3 d (200, 500 and 1000 ppm of COD, respectively). Likewise, average total COD removal and total coulombic efficiency were 89.67 ± 5.19% and 48.07 ± 2.33%, respectively.

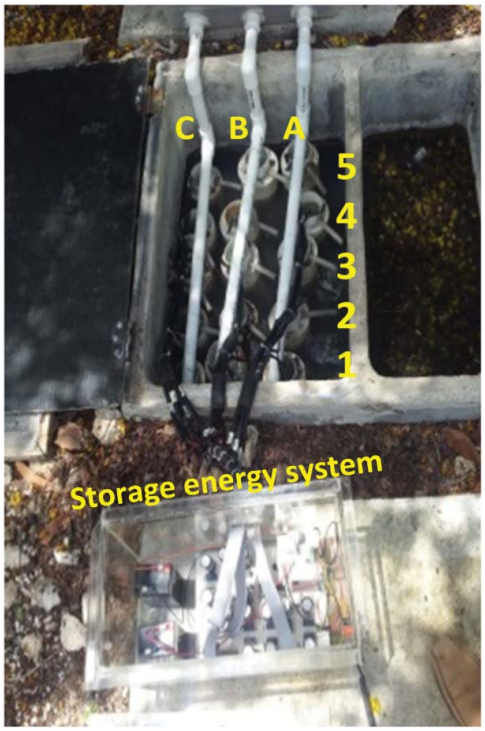

Figure 2. Stacked microbial fuel cells connected to an energy storage circuit.

3.1. Energy storage and the maximum power harvesting

Fifteen MFCs share the same electrolyte in continuous flow conditions. Although the resistors make it straightforward to measure the MFC power generation, this scheme cannot be used for practical purpose.[20] For efficient harvesting and using of the MFC energy, a power conversion circuitry is indispensable for capturing the electrical energy from MFCs and shaping it into a usable form.[21] Furthermore, the MFC units in the stack connected either serial or parallel connections; this increased the total available electrode surface area but did not change the reactor volume that involves the voltage reversal.[22] Therefore, in this study an external connection was made in series of 15 supercapacitors initially connected to each MFC, using a charge pump power supply that is a DC to DC converter that uses supercapacitors as energy storage elements to efficiently create either a higher or lower output voltage from each MFC.[18] Likewise, the charge pumps may not affect parameters as microbial activity and community much because these devices just receive whatever amount of power provided by the MFC.[23]

Figure 3 A, B and C show the polarization and power density curves obtained in the steady-state. The maximum power density produced by the MFC with 0.24, 0.52 and 1.05 kg DQO/m^3 d were 146.67, 151.2 and 152.7 mW/m^3, with an average of 302, 323 and 351 mV, respectively. The corresponding external resistors at the peak power density were 73 Ω, 53 Ω and 39 Ω.

The charge pump was able to harvest the SMFC energy. During the initial load conditions the voltage of the super-capacitors was 0 V causing a short circuit on the CCM;[17,24] with increased R-value the capacitor stores more energy and so increased voltage of the capacitor and the cell.

The energy is extracted from the SMFC during the Charge mode; around the time (t) = 18.12 h the capacitor voltage equals the open circuit voltage of the MFC, R is much greater than the internal resistance of the MFC and there is no electron flow to the capacitor (Figure 4A), showing the ability of the fuel cell to charge the capacitor.[24] The maximum power transferred was 8.75 mW in 12.59 minutes after starting the capacitor charging process (100 F). By integrating the power curve in 9,216 s, the total energy transferred was 38.76 J.

Once loaded the 15 capacitors were connected in series with each other to amplify the voltage at 3.11 V, and in the discharge process of capacitors transfer

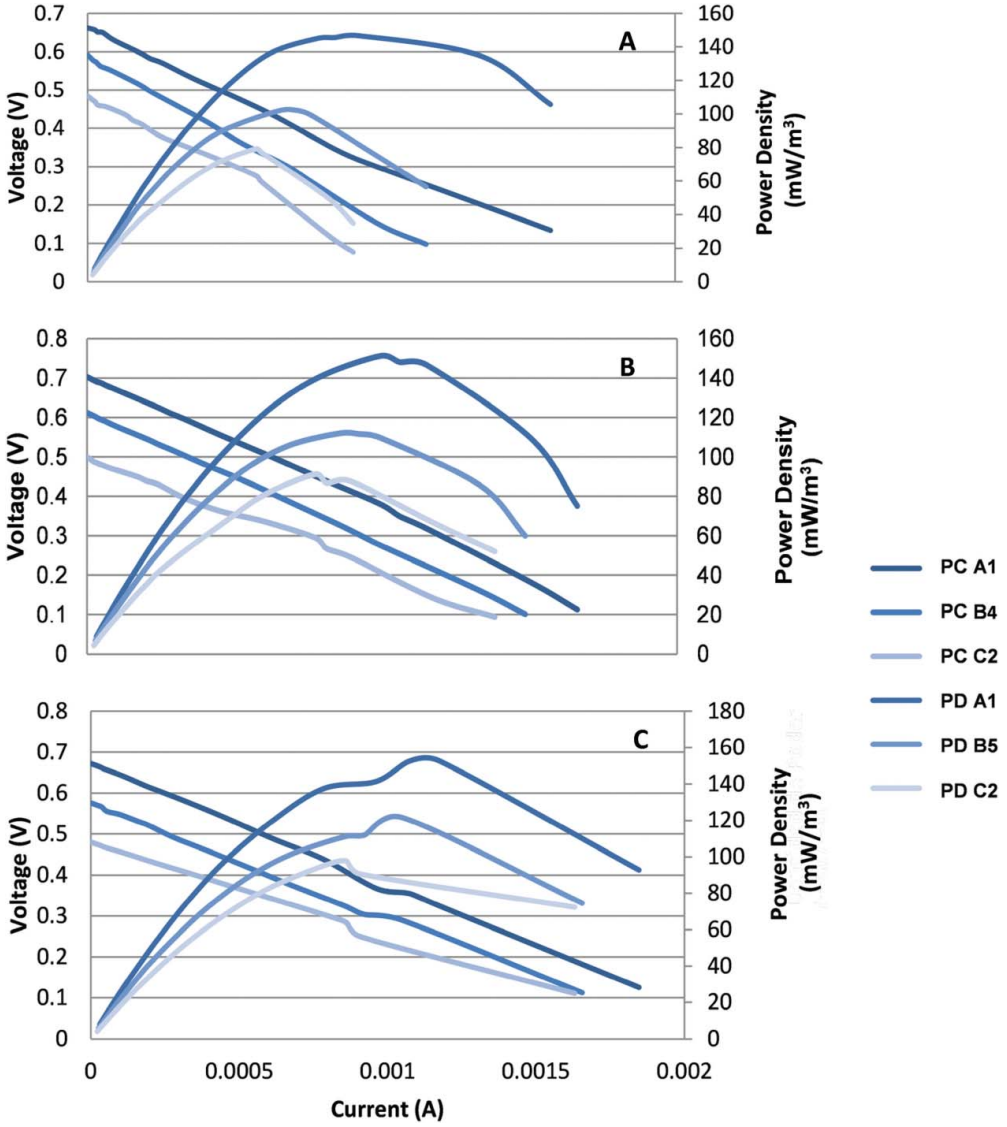

Figure 3. Polarization and power density curves (A) 200 ppm, (B) 500 and (C) 1000 ppm.
PC = Polarization Curve and PD = Power Density.

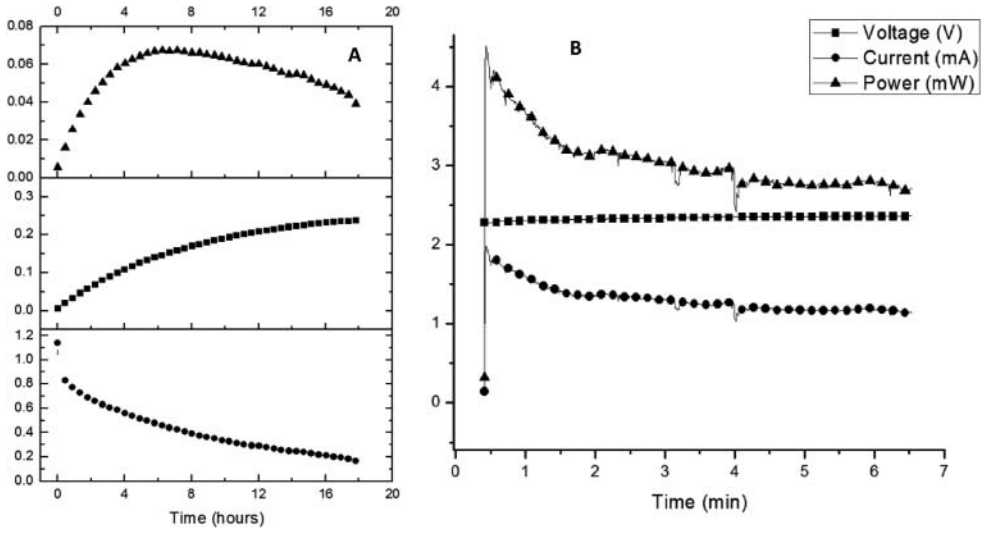

Figure 4. Energy transfer from (A) MFC to capacitor (Charge Mode) and (B) capacitors to the battery (discharge mode).

energy to a battery with a stable voltage of 2.2 V (Figure 4B).

The initial voltage transfer from capacitors to the battery was 2.2 V up to the theoretical value of battery charge of 2.4 V (t = 378 s). The maximum current transferred by connecting the capacitors to the battery was 1.98 mA, reaching a minimum value of 1.14 mA and maximum power transferred of 4.51 mW (Figure 4B).

Likewise, the integration of the power transferred over time, it allowed to obtain the total energy transferred from the capacitors to the battery (1.117 J).

3.2. Performance of MFC units

For testing, three of 15 MFCs corresponding to the highest (A1), intermediate (B4) and lower (C2) power density were chosen for each organic loading rate evaluated (Table 1).

As shown in Figure 3 A, to the substrate concentration of 200 ppm, the MFC A1 had the highest power density of 146.67 ± 2.12 mW/m^3 and lower internal resistance 99.68 ± 5.44 Ω; MFC B4 was an intermediate power density of 102.24 ± 6.03 mW/m^3 and an internal resistance of 122.24 ± 12.78 Ω and the lower power density corresponded to the MFC C2 with 79.30 ± 3.11 mW/m^3 and an internal resistance of 139.66 ± 11.69 Ω.

At a substrate concentration of 500 ppm (Figure 3B), the MFC A1 had the highest power density of 151.20 ± 9.77 mW/m^3 and lower internal resistance 87.93 ± 8.83 Ω. Meanwhile, MFC B4 had a power density intermediate of 111.96 ± 4.32 mW/m^3 and an internal resistance of 95.09 ± 10.32 Ω and the lower power density corresponded to the MFC C2 of 91.12 ± 7.12 mW/m^3 with an internal resistance of 96.78 ± 12.64 Ω.

When it used 1000 ppm, as shown in Figure 3C, MFC A1 had the highest power density 152.70 ± 8.34 mW/m^3 with an internal resistance of 76.68 ± 6.85 Ω. MFC B4 had an intermediate power density of 121.63 ± 9.42 mW/m^3 and an internal resistance of 77.48 ± 6.54 Ω and the lower power density corresponded to MFC C2 with 97.71 ± 2.69 mW/m^3 and an internal resistance of 77.74 ± 8.52 Ω, respectively.

These values are similar to those obtained by Zhang et al. [25] who used MFC-C-AC (180 mW/m^3) in tubular MFCs that contained only activated carbon powder (5 mg/cm^2) as a cathode catalyst for more than 400 days in situ investigation in an aeration tank. Zhang et al. [25] obtained values slightly higher in an unstable electric current constructed with ion exchange membrane and cathode catalysts, MFC-C-Pt and MFC-A-Pt, that had 0.1 mg/cm^2 of Pt (10% Pt on carbon black) and 4 mg/cm^2 of activated carbon as cathode catalysts for oxygen reduction, obtaining power densities of 370 mW/m^3 and 270 mW/m^3, respectively. Rabaey et al. [26] and Zhang et al. [27] obtained from laboratory tubular shape MFCs ranges from 2 to 60 W/m.

The volumetric loading rate was increased by increasing the concentration of sucrose and buffer in the feed while maintaining constant nutrient levels. The increase in buffer concentration increased the concentration of ions in the anolyte and, therefore, decreased R_{int} from 120.53 ± 20.05 to 77.30 ± 2.05 Ω over the operating period. He et al. [28] found that the internal resistance of their MFC decreased from 5.44 to 2.41 Ω when the organic load was increased from 0.57 to 3.40 kg COD/m^3-day. However, while they used an organic load rate of 4.29 kg COD/m^3-día, its resistance increased to 2.89 Ω, likely due to the fact that substrate concentration was higher than capacity of the MFC for conversion to electricity which caused lower power density and methane production.

The SMFC increased the power density when the substrate concentration changed from 200 ppm to 500 ppm. However, in the concentration of 1000 ppm a difference in the power densities was not observed. Likewise, COD removal was similar at 500 and 1000 ppm (about 85%). This may explain why the power density does not increase significantly with increasing concentration of the substrate because the electrons obtained from degradation of the substrate are the same. Cheng and Logan [29] obtained the maximum power density when they increased the substrate concentration at 0.15 g/L, the power density was 27 W/m^3. When the substrate concentration increased, power increased by 33% to 36 W/m^3 (0.5 g/L), and by 56% to 42 W/m^3 at 1 g/L; however, further increases in the acetate concentration to 2 g/L did not appreciably affect power density; therefore they concluded that at low substrate concentrations power can be hindered by the anode.

On the other hand, methane measurement in the second chamber (SMFC assembly) was conducted with a portable biogas analyzer, resulting in 5% of total biogas composition.

Table 1. Summary of the power density and fit of experimental results from EIS for A1, B4 and C2.

MFC	Ps-max (mW/m^3)	Romh (Ω)	Rct (Ω)	Rint (Ω)*
200 mg/L (0.24 kg COD/m^3 d)				
A1	146.67±2.12	13.71±0.45	85.97±4.99	99.68±5.44
B4	102.24±6.03	18.54±1.16	103.7±11.62	122.24±12.78
C2	79.3±3.11	22.56±2.01	117.1±9.68	139.66±11.69
Average	109.40±34.25	18.27±4.43	102.26±15.62	120.53±20.05
500 mg/L (0.52 kg COD/m^3 d)				
A1	151.2±9.77	17.64±0.69	70.29±8.14	87.93±8.83
B4	111.96±4.32	14.21±2.65	80.88±7.67	95.09±10.32
C2	91.12±7.12	16.57±4.57	80.21±8.07	96.78±12.64
Average	131.58±27.75	16.14±1.75	77.13±5.93	93.27±7.68
1000 mg/L (1.05 kg COD/m^3 d)				
A1	152.7±8.34	12.72±1.44	63.96±5.41	76.68±6.85
B4	121.63±9.42	11.72±0.98	66.02±7.54	77.48±6.54
C2	97.71±2.69	13.46±2.33	64.02±4.21	77.74±8.52
Average	124.01±27.57	12.63±0.87	64.67±1.17	77.30±2.05

PS-max = maximum power density; Romh = ohmic resistance; Rct = charge transfer resistance; Rint = internal resistance.
± standard deviation
*Internal resistance calculated from impedance analysis

3.3. Electrochemical Impedance Spectroscopy (EIS)

To obtain quantitative data on the resistances, overall internal resistance (R_{int}) and its sources were analyzed by fitting experimental data into an equivalent circuit without a diffusion resistance,[28] because the diffusion resistance showed that transport limitation played a minor role, compared to ohmic and kinetic limitations (Figure 5 A, B, C). According Yuan et al. [30], at low over potentials the main contributor to the impedance is the interfacial charge transfer resistance, which is potential dependent. Dependence on electrode potential is given by the Tafel equation ($V \sim \log Rp^{-1}$).

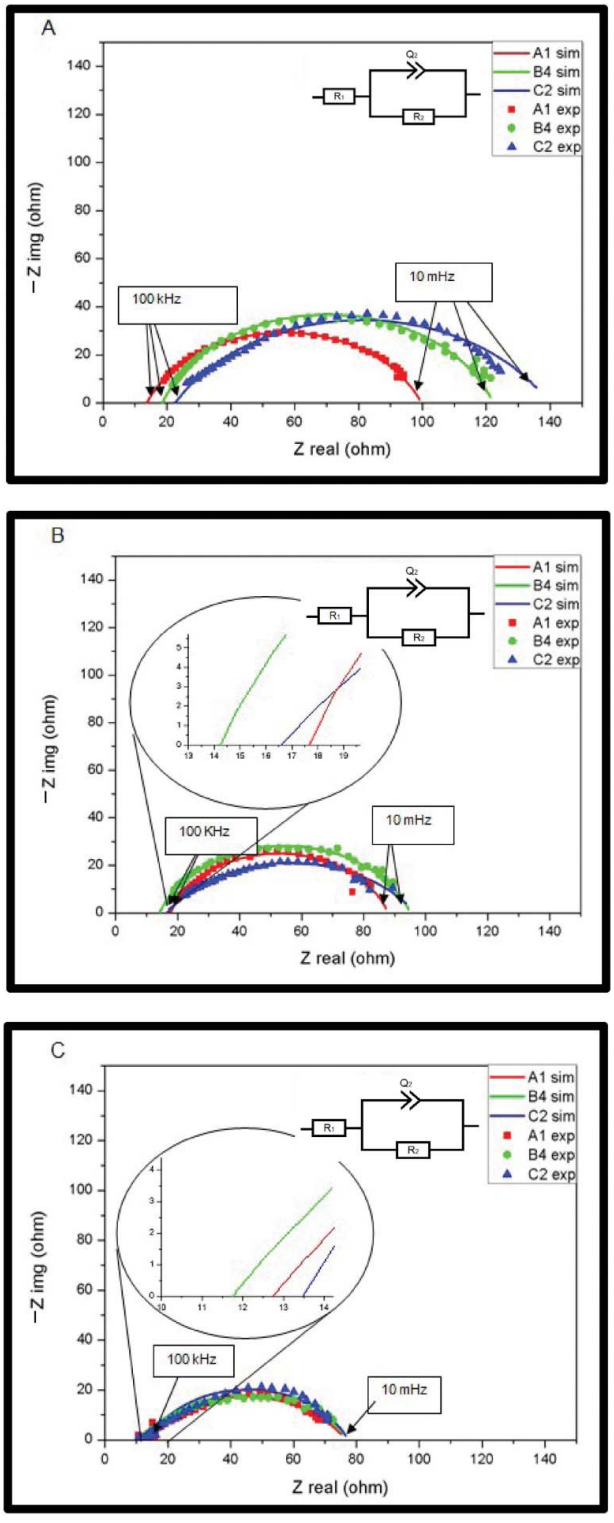

Figure 5. Nyquist plots and equivalent circuit (A) 200 ppm, (B) 500 and (C) 1000 ppm.

The simulated data in the Nyquist plot were generated from the experimental data at the frequency range between 0.01 to 100,000 Hz. A Nyquist plot anode showed that R_{int} of SMFC at the volumetric loading rate of 0.24 kg COD/m^3 day was 120.53 ± 20.05 Ω and significantly decreased R_{int} in 23% and 36% by 93.27 ± 7.68 Ω (0.52 kg COD/m^3) and 77.30 ± 2.05 Ω (1.05 kg COD/m^3), respectively, likely due to the fact that the biofilm facilitates charge accumulation at the electrode interface. Although the values may vary based on the experimental conditions (electrode material, inoculum type, etc.), the behavior is similar to that observed in previous works.[31,32]

Rabaey et al. [26] with an Upflow microbial fuel cell obtained a lower overall R_{int} of 4 Ω compared to this study. The lower electrolyte resistance was likely caused by higher anolyte conductivity with sodium acetate (780 mg/L) as compared to sucrose as an artificial wastewater and a lower volume to PEM surface area ratio that was compared.

The trend of ohmic resistance is decreased with increasing substrate concentration from 200 to 1000 mg/LCOD. This is likely due to the amount of ions that increase with the concentration of synthetic wastewater (SW). This favors the conductivity of the anolyte resulting in a reduced ohmic resistance (from 18.27 ± 4.43 Ω to 12.63 ± 0.87 Ω). The relationship between conductivity and ohmic resistance is consistent with that obtained in other studies [33,34] using higher buffer concentrations [35] enhances MFC performance through reduction of ohmic resistance. The buffer affects MFC performance in several ways due to its chemical composition and interaction with the electrodes, bacteria, and membrane (if present). In addition, the buffer helps to reduce changes in pH in the bulk solution and in the biofilm, and therefore it maintains the pH in the range suitable for the growth of microorganisms.[36] While the effect of solution conductivity on maximum power density is consistent with the literature.[34,36,37,38]

As shown in Table 1, when the volumetric loading rate was increased from 0.24 to 0.52 kg COD/m^3 d, R_{ct} decreased from 102.26 ± 15.62 to 77.13 ± 5.93 Ω. Likewise, a similar correlation between anode charge transfer resistance and power output was observed between 500 and 1000 mg/L, 77.13 ± 5.93 Ω for 131.58 ± 27.75 mW/m^3 and 64.67 ± 1.17 Ω corresponding to 124.01 ± 27.57 mW/m^3, respectively, thereby indicating the increased electron transfer ability through the selection of anodophilic microbes.[28]

Also, it was found that there is only one semicircle over the low frequency range in the cathode EIS results, which indicates that there was no obvious

diffusion effect. The diameter of the semicircle still represents the resistance. Thus, the cathode internal resistance is divided into ohmic resistance and charge transfer resistance in series.[39] The R_{int} represented the cathode with a slightly varying resistance $66.78 \pm 0.89\Omega$ over the operating period of the three different organic loading rates 0.24, 0.52 and 1.05 kg DQO/m^3 d (catholyte composition was constant).

Comparison of the anode and cathode impedance reveals that anode internal resistances are higher. The larger semicircle relates approximately to the larger reaction resistance due to the slow kinetics.[40] However, anode and cathode transfer resistances were similarly limited and performance can be further improved by optimizing both the anode and the cathode reaction rates.[41]

4. Conclusions

This research has demonstrated the great challenges in applying a stack of MFC in scale-up. However, it is very important to identify the potential application into a septic tank as polishing treatment. R_{int} showed that anode resistances are higher than cathode; however, the configuration seems to be indicated for this kind of system depuration in developing countries. The difference in electrochemical performance between laboratory and scale up exposed that factors as conductivity of the anode and cathode solutions, biofilm in the anode, and the high activation energy play an important role. An electrochemical model must be developed for scale up which explains performance and electrochemical data. It is necessary to develop experiments in scale up and to test the feasibility of real implementation.

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Disclosure statement

No potential conflict of interest was reported by the authors.

ORCID

Francisco Del Rio Jorge-Rivera http://orcid.org/0000-0002-6494-5885

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