TERRA LATINOAMERICANA

Effect of Hydraulic Retention Time on Municipal Wastewater Treatment and Energy Recovery at a Semi-Pilot Scale Using a Modular Bioelectrochemical System

Efecto del Tiempo de Retención Hidráulica en el Tratamiento de Agua Residual Municipal y Recuperación de Energía a Escala Semi-Piloto Utilizando un Sistema Bioelectroquímico Modular

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Recommended citation:

Pérez-Rodríguez, P., Alvarado-Camarillo, D., Borrego-Limón, B. V., Méndez-López, A., Rodríguez-De la Garza, J. A., Reyes-Acosta, A. V., & Martínez-Amador, S. Y. (2025). Effect of Hydraulic Retention Time on Municipal Wastewater Treatment and Energy Recovery at a Semi-Pilot Scale Using a Modular Bioelectrochemical System. *Terra Latinoamericana, 43*, 1-11. e2073. https://doi.org/10.28940/terra.v43i.2073

Received: August 30, 2024.
Accepted: May 20, 2025.
Article, Volume 43.
October 2025.

Section Editor:
Dra. Circe Aidín Aburto-González

Technical Editor:
M. C. Ayenia Carolina Rosales Nieblas

SUMMARY

Bioelectrochemical systems (BES) remove high concentrations of organic and inorganic contaminants and generate energy. It has been proven that they are more efficient when working with small-volume modules (such as stacks or cascades) than with a large reactor volume. In this work, the effect of hydraulic retention time (HRT) on the removal of chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), total solids (TS), total suspended solids (TSS), total coliforms (TC), fecal coliforms (FC), helminth eggs (HE), and power generation of a modular bioelectrochemical system consisting of ten dual-compartment microbial fuel cells (MFC) of 2000 mL each during the treatment of municipal wastewater (MWW) was assessed. The increase in HRT had a positive impact. In a 5-h HRT, the removal efficiency percentages of COD, BOD_5, TS, TSS, TC, FC, and HE were 66.46, 34.18, 36.76, 53.84, 99.99, 99.99, and 91.88%, respectively. Likewise, at this same HRT (5 h), the highest maximum power density of 5.709 mW m^{-3} and the coulombic efficiency of 0.0056% were obtained. Results showed that the modular BES increases energy generation and contaminant removal, especially pathogens (TC, FC, and HE).

Index words: *coulombic efficiency, microbial fuel cell, pathogens, power density, water quality.*

RESUMEN

Los sistemas bioelectroquímicos (BES) remueven altas concentraciones de contaminantes orgánicos e inorgánicos y generan energía eléctrica. Se ha demostrado que son más eficientes cuando operan en módulos de volumen reducido (apilados o en cascada) que en reactores de gran volumen. En este trabajo, se evaluó el efecto del tiempo de retención hidráulica (TRH) en la remoción de la demanda química de oxígeno (DQO), demanda bioquímica de oxígeno (DBO_5), sólidos totales (ST), sólidos suspendidos totales (SST), coliformes totales (CT), coliformes fecales (CF), huevos de helminto (HE), y la generación de potencia de un sistema bioelectroquímico modular compuesto por diez celdas de combustible microbianas (CCM) de 2000 mL cada una durante el tratamiento de agua residual municipal (ARM). El incremento en el TRH tuvo un impacto positivo. A un TRH de 5 h, los porcentajes de eficiencia de remoción de DQO, DBO_5, ST, SST, CT, CF y HE fueron de 66.46, 34.18, 36.76, 53.84, 99.99, 99.99 y 91.88%, respectivamente. Asimismo, a este mismo TRH (5 h), se obtuvo una densidad de potencia máxima de 5.709 mW m^{-3} y una eficiencia coulómbica de 0.0056%. Los resultados demostraron que el SBE modular incrementó la generación de energía eléctrica y la eliminación de contaminantes, especialmente patógenos (CT, CF y HE).

Palabras clave: *eficiencia coulómbica, celda de combustible microbiana, patógenos, densidad de potencia, calidad del agua*.

INTRODUCTION

Constant population growth (estimated at 8 billion inhabitants on the planet) has begun to generate problems such as the depletion of natural resources, including water. The shortage of the vital liquid for human consumption is not only due to the demand for this product but is also caused by other factors, such as the irregular distribution of aquatic reserves, the waste of this resource by the upper social classes, unsustainable management due to excessive extraction, and contamination of aquifers (Salehi, 2022). The use of raw wastewater negatively modifies the physical and chemical properties and increases heavy metals in the soils (García-Carrillo *et al*., 2020). Because of this, water treatment and reuse have begun to play a fundamental role in managing this resource.

Within the different types of biological wastewater treatment, bioelectrochemical systems (BES) have generated interest due to their simultaneous capacity for the remediation of contaminated water and soil and the generation of electrical energy (Gude, 2018; Jung, Lee, Park, and Kwon, 2020; Li, Li, and Zhou, 2021; Lu *et al*., 2018; Wang and He, 2020). These systems stand out for their ability to operate efficiently at room temperature and to use a wide variety of organic waste as substrate (Gul and Ahmad, 2019). Despite this, some limitations make it difficult to scale this type of system to an industrial level, among which are: a) high production and operation costs, b) high complexity in their design and standardization, and c) poorly reproducible devices (Leicester, Amezaga, and Heidrich, 2020). Even so, several authors have successfully scaled this type of technology. For example, Mohamed, Zmuda, Ha, Coats, and Beyenal (2021) evaluated municipal wastewater treatment in 260 and 1200 L microbial fuel cells packaged with polarized anodes and cathodes. The results demonstrated that the potentiostatic modification of the electrodes considerably favors their microbial colonization, thus increasing the removal of organic matter in the substrate. On the other hand, Mohanakrishna, Al-Raoush, and Abu-Reesh (2020) used a single-compartment bioelectrochemical system (SC-BES, 1.2 L) fed with acetate and municipal wastewater as biostimulants for the remediation of soils contaminated with hydrocarbons. The authors verified that acetate and municipal wastewater promote energy generation and the removal of hydrocarbons in the system. Also, Lust, Nerut, Kasak, and Mander (2020) studied the biotic and abiotic processes that affect the capacity of a pilot plant scale bioelectrochemical system (5.86 L) to remove nitrates in environments with low organic matter content (groundwater). The authors highlighted the importance of inoculum in this research type, mentioning that specific bacteria could considerably increase cell performance. Similarly, San-Martín, Mateos, Carracedo, Escapa, and Morán (2018) characterized the performance of a pilot plant-scale bioelectrochemical system (150 L) fed with municipal wastewater to remove total nitrogen and total organic carbon. The results showed the capacity of this type of system to degrade highly complex substrates while generating directly usable electrical energy. Finally, Yang, Liu, and Yang (2022) studied the electrochemical performance, substrate contamination (zeolite), and microbial community of a microbial fuel cell coupled to a semi-pilot scale artificial wetland (316 L) fed with pond water as a substrate. The authors attributed the generation of electrical energy and the low accumulation of contaminants in the substrate to the high density and diversity of microorganisms that colonized the anode in the system.

In a previous study (Pérez-Rodríguez, Covarrubias, Rodríguez, Barrera, and Martínez, 2023), we focused on the synthesis of graphite and carbon nanofiber-based composites embedded in a polyurethane matrix and used these materials as anodes in a laboratory-scale microbial fuel cell for municipal wastewater treatment. In this work, a semi-pilot scale modular bioelectrochemical system (20 L) was developed to treat municipal effluents and generate electrical energy. The modular design of this system enhances its scalability, standardization, and reproducibility. The effect of hydraulic retention time (HRT) on the removal of COD, BOD, TS, TSS, TC, FC, HE, and power production was evaluated.

MATERIALS AND METHODS

Experimental Setup

Figure 1 shows the configuration of the modular semi-pilot scale bioelectrochemical system (MSP-BES) used in the experiments. This device comprised ten double-compartment microbial fuel cells (MFCs), with an approximate volume of 2000 mL per cell (1000 mL per compartment). The compartments were separated by a cation exchange membrane (CXM-200, International Membranes Inc., Ringwood, NJ, USA) previously treated in a 5% NaCl solution for 12 h. This membrane was chosen for its superior cost-effectiveness compared to competitors (Yuan *et al*., 2021). Polyurethane/graphite composites 0.5% were used as anodes, which were prepared according to Pérez-Rodríguez *et al*. (2023), using pieces of graphite felt (10 × 10 × 0.8 cm, SinBarreraS Servicios Industriales, Saltillo, Coahuila, México) as cathodes (considering their high electrical conductivity) in each cell. Stainless steel wire (316L, 0.64 mm diameter, Master Wire Supply, Beavercreek, OH, USA) was used as the electron collector. The cathode compartment of each MFC was externally oxygenated with an air pump (AquaJet101, Grupo Acuario Lomas, S.A. de C.V., Cuajimalpa de Morelos, Ciudad de México, México). The MFCs were connected in series to an external 1 kΩ resistor.

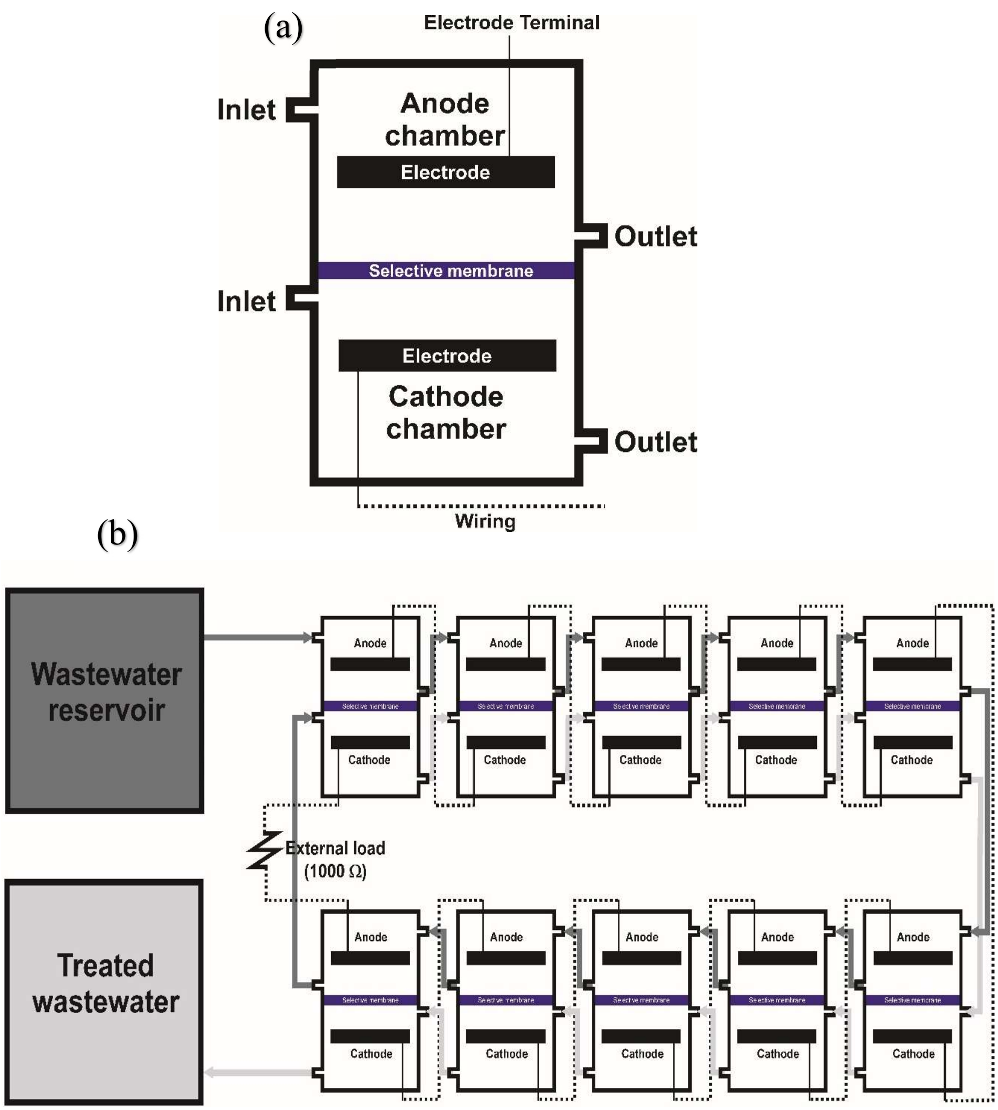

Figure 1. MSP-BES scheme = (a) MFC configuration; (b) BES arrangement.

Inoculum and Substrate Characteristics

Raw municipal wastewater was used as inoculum and substrate. The sample was collected from a local wastewater treatment plant (Saltillo, Mexico). Table 1 shows the physicochemical and microbiological characteristics of the wastewater used in the experiments, which, according to the contained concentration of COD (712.5 mg L^{-1}), BOD (433.33 mg L^{-1}), TSS (400.2 mg L^{-1}), and fecal coliforms (*E. coli* 1×10^7 MPN 100 mL^{-1}) corresponds to a MWW of medium composition (Henze and Comeau, 2008).

BES Operation

Initially, the system was fed with raw municipal wastewater, and the medium was recirculated with a peristaltic pump (Kcp200, Kamoer, Shanghai, CN) for 30 days to promote microbial growth on the supports used (electrodes). During this process, the anode and cathode chambers (biotic/biotic) were kept separate when considering their operating conditions (anode/anoxic, cathode/aerobic). The residual water was replaced when reaching a value less than 100 mg COD L^{-1}. Subsequently, three hydraulic retention times (HRTs) of 2, 3.5 and 5 h (values selected based on the operational limitations of the peristaltic pump used) were established to evaluate the removal of organic matter (as chemical oxygen demand) and generation of electrical energy (as voltage, volumetric power density and coulombic efficiency) during the sequential treatment (anoxic-aerobic) of raw municipal wastewater in the MSP-BES. The system was configured so the wastewater passed through all anodic compartments and subsequently through all cathodic compartments, as shown in Figure 1. All experiments were conducted in triplicate at room temperature.

The pH and electrical conductivity (EC) were determined using a multiparameter meter (Orion Star A215, Thermo Scientific, Waltham, MA, USA). The chemical oxygen demand (COD) was determined by the small-scale sealed tube method by NMX-AA-030/2-SCFI-2011 (SE, 2011). MSP-BES's output voltage (U) was monitored using a digital multimeter (Fluke 289, Fluke Corporation, Everett, WA, USA). The volumetric power density (mW m^{-3}) was calculated based on what was proposed by Luo *et al*. (2010). The coulombic efficiency (%) was calculated as:

$$CE = \frac{MI}{Fqb\Delta COD} \times 100\ \% \quad (1)$$

Where M is the molecular weight of oxygen (32 g mol^{-1}), I is the electric current (A), F is Faraday's constant (96.485 C mol e^{-1}), q is the inlet volumetric flow (L/s), b is the number of electrons exchanged per mole of oxygen ($4e^-$ mol/mol), and ΔCOD is the difference in COD over time (mg L^{-1}) (Logan *et al*., 2006). All measurements were performed in triplicate.

Table 1. Physicochemical and microbiological analysis of raw municipal wastewater.

Parameters	Raw Municipal Wastewater
Color	Dark grey
Odor	Strong pungent
Temperature	25 °C
pH	9.36
Electrical conductivity	1.29 mS cm^{-1}
Chemical oxygen demand	712.5 mg L^{-1}
Total solids	1091.1 mg L^{-1}
Total suspended solids	433.33 mg L^{-1}
Biochemical oxygen demand	400.2 mg L^{-1}
Total coliforms	≥ 2400 MPN † 100 mL^{-1}
Fecal coliforms	≥ 2400 MPN 100 mL^{-1}
Helminth eggs	2464 HE L^{-1}

† MPN = Most probable number.

Water Quality Analysis

The quality of municipal wastewater treated in the modular bioelectrochemical system was determined. Total solids (TS) and total suspended solids (TSS), biochemical oxygen demand (BOD_5), total (TC) and fecal coliforms (FC), and helminth eggs (HE) were evaluated based on the provisions of the Norma Oficial Mexicana (Mexican Oficial Standard) NMX-AA-034-SCFI-2015 (SE, 2015a), NMX-AA-028-SCFI-2021 (SE, 2021), NMX-AA-042-SCFI-2015 (SE, 2015b), and NMX-AA-113-SCFI-2012 (SE, 2012), respectively.

Electrochemical Characterization

The variable resistance method calculated the polarization and power density curves (Bose, Dhawan, Kandpal, Vijay, and Gopinath, 2018), applying external resistances ranging from 50 Ω to 100 kΩ once the system's voltage was stabilized. The power density obtained was normalized to the volume of the anodic compartment in cubic meters, (m^3).

RESULTS AND DISCUSSION

pH, EC, COD Removal, and Power Generation in MSP-BES

The pH and EC of the treated wastewater in the MSP-BES are presented in Table 2 and Figure 2. The results show an increase in the pH of the effluent (exiting the cathode chamber) with the increase in HRT in the system. Işık, Altaş, Kurmaç, Özcan, and Oruç (2010) studied the influence of HRT on the performance of a biocatalytic calcification reactor (BCR) fed with synthetic wastewater. The authors observed an increase in the pH of the system with the increase in HRTs. They argue that the production of ammonium and CO_2 due to urea hydrolysis causes an increase in pH. On the other hand, Baâti, Benyoucef, Makan, El Bouadili, and El Ghmari (2018) investigated the influence of HRT on biogas production during urban waste leachate treatment using an upflow anaerobic sludge blanket (UASB) reactor, observing the decrease in EC as the HRT of the system increased. The authors attribute this behavior to the precipitation of mineral salts due to the increased pH in the reactor used.

Table 3 and Figure 3 display the COD removal efficiency (along with the standard error for each value) in the MSP-BES. At HRTs of 2, 3.5, and 5 h, removal efficiencies of 49.24, 58.54, and 66.46% were obtained, respectively. The values obtained indicate a linear increase in COD removal by proportionally decreasing the feed flow of the influent to the system. This behavior may be due to two factors. On the one hand, under low HRT, the contact time between microorganisms and substrate is limited, significantly reducing the metabolization of organic matter in the sample (Cai *et al*., 2019). On the other hand, longer HRT favors microbial colonization of the supports used (Fazli, Mutamim, Jafri, and Ramli, 2018; Jiang *et al*., 2018). The above agrees with what is described by other authors (Al-Rubaye *et al*., 2018; Cruz-Salomón *et al*., 2017; Musa and Idrus, 2020) and demonstrates the capacity of the MSP-BES to remediate municipal effluents to the HRTs evaluated. Nevertheless, future strategies to enhance the scale-up of the modular BES, with a particular emphasis on increasing pollutant removal rates while reducing the required HRT, must be contemplated. These strategies may include enhancing the electrical conductivity, surface area, and biocompatibility of the electrodes and integrating pretreatment systems to lower the organic load before the influent enters the BES.

The changes in the HRT had a subtle but perceivable effect on the generation of electrical energy in the MSP-BES. During system operation, the maximum voltage reached at HRTs of 2, 3.5, and 5 h was 0.306, 0.322, and 0.337 U, respectively (Table 4). The maximum power density was 5.709 mW m^{-3}, obtained at an HRT of 5 h. Coulombic efficiency increased from 0.0028% to the highest value of 0.0056% when HRT was increased from 2 to 5 h.

Table 2. pH and EC in the MSP-BES at different HRTs.

HRT (h)	pH	EC (mS cm^{-1})
2	8.64 ± 0.02	1.84 ± 0.01
3.5	8.71 ± 0.03	1.73 ± 0.03
5	9.15 ± 0.03	1.66 ± 0.01

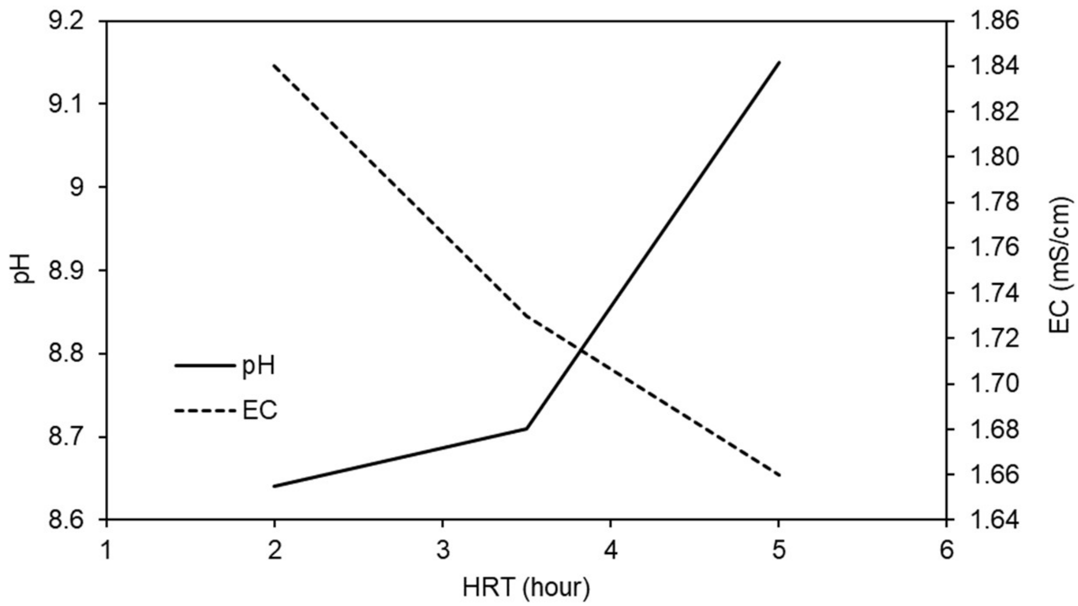

Figure 2. Evolution of pH and EC in function of HRT.

These results are compatible with what was reported by Liu, Cheng, Huang, and Logan (2008), who pointed out that in MFCs operated at low HRTs the generation of electrical energy decreases due to the increase in the concentration of dissolved oxygen in the medium. Furthermore, Ye *et al*. (2020) noted that high flow rates of the feed solution (synthetic municipal wastewater) worsen the mixing conditions in an MFC reactor, thereby negatively impacting the development of electrically active microorganisms (exoelectrogens) within the system. In its current form, the application of this technology is thus limited to processes with low energy demands (biosensors, low-power microcontrollers, and LED indicators), particularly in the context of on-site wastewater treatment (integrated into larger hybrid systems) and in resource-limited environments.

Treated Wastewater Quality at HRT of 5 h

As shown in Table 5, the removal rate of TS, TSS, BOD_5, total coliforms, fecal coliforms, and helminth eggs in the MSP-BES at an optimized HRT of 5 h was 36.76, 53.84, 34.18, 99.99, 99.99, and 91.88%, respectively. In a study where they used the organic matter recovered from municipal wastewater as a substrate for an MFC operated at an HRT of 2 h, they detected a removal of 56.9% of the TSS (Ma, Wang, Li, Wang, and Wu, 2014), a percentage close to that obtained in this study (53.84%). Even in wastewater with low COD content (≤ 130 mg L^{-1}), such as the effluent of a primary clarifier that treated municipal wastewater with which a pilot scale MFC of 45 liters, divided into 4 modules operated at an HRT of 22 h obtained a removal of 40% of the TSS (Hiegemann *et al*., 2016). In a complete domestic wastewater treatment system consisting of a septic tank primary treatment, a secondary treatment containing 18 MFCs, coupled to an energy-harvesting circuit that stored the

Table 3. COD removal efficiency and standard error for each value in the MSP-BES at different HRTs.

HRT (h)	COD removal efficiency (%)	Standard error
2	49.24	2.78
3.5	58.54	3.02
5	66.46	4.2

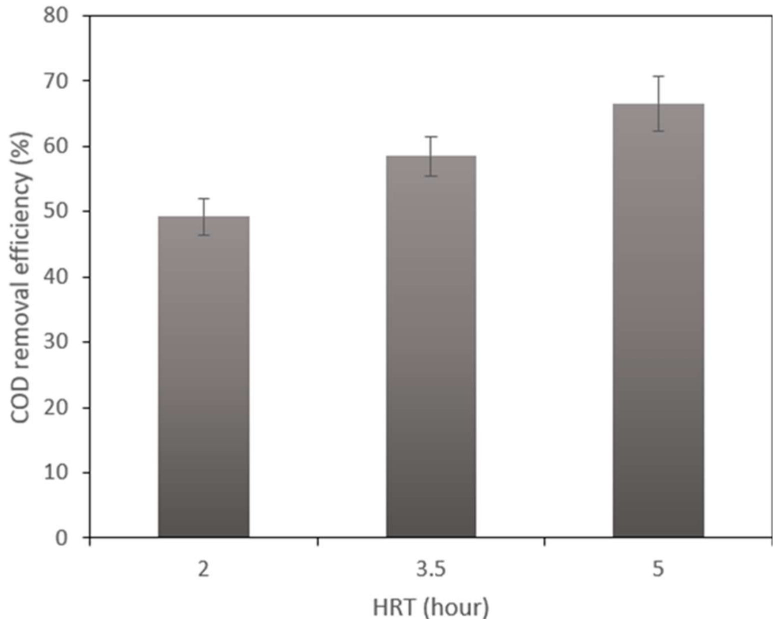

Figure 3. COD removal efficiency in the MSP-BES at different HRTs.

electrons produced by anaerobic respiration, and gravity-driven disinfection, the removal of 95% of the TSS and 87% of the BOD at an HRT of 43 h. They measured the removal of FC before and after the chlorine disinfection process, obtaining 77.9 and 100%, respectively (Valladares-Linares *et al*., 2019), while in this research, removal of 99.99% of FC was achieved (from 1×10^7 to 1100 MPN 100 mL^{-1}). Pérez-Rodríguez *et al*. (2018) detected a 94, 90 and 64% removal of BOD, FC and HE, respectively, when treating municipal wastewater in a BES at an HRT of 24 h; in comparison in this study, at an HRT of 5 h, a 34.18, 99.99 and 91.88% of the BOD, FC, and HE were removed, respectively. Some mechanisms that promote contaminant removal in BES are well documented. For example, microbial metabolization of organic matter decreases the concentration of biochemical oxygen demand (BOD) in the sample through the breakdown of complex organic compounds by microorganisms, resulting in simpler, less harmful substances (Abrevaya, Sacco, Bonetto, Hilding, and Cortón, 2015). Also, anodic microorganisms (particularly those belonging to the genera *Geobacter* and *Shewanella*) involved in pollutant degradation and energy generation can outcompete (for space and nutrients) pathogenic microbes, reducing coliforms and helminth eggs in the wastewater (Ieropoulos, Obata, Pasternak, and Greenman, 2019). Finally, the prevalence of anaerobic conditions (required for exoelectrogens to efficiently transfer electrons to the anode instead of using oxygen or other compounds as final electron acceptors) in the anodic chamber (counting the cathode chamber with completely opposite conditions) decreases the system's pH (acidogenesis), which can result in the solubilization of total and suspended solids in the effluent (Subha *et al*., 2019). These arguments support the results obtained, increasing the efficiency of contaminant removal by decreasing the feed flow of the substrate to the MSP-BES to a greater extent.

Table 4. Voltage, power density, and coulombic efficiency in the MSP-BES at different HRTs.

HRT (h)	Voltage	Power density	Coulombic efficiency
	U	mW m^{-3}	%
2	0.306	4.706	0.0028
3.5	0.322	5.193	0.0043
5	0.337	5.709	0.0056

Table 5. Pollutant removal in the MSP-BES at a 5 h HRT.

Parameters	Effluent	Removal efficiency (%)
Chemical oxygen demand	407.5 mg L^{-1}	42.80
Total solids	690 mg L^{-1}	36.76
Total suspended solids	200 mg L^{-1}	53.84
Biochemical oxygen demand	263.4 mg L^{-1}	34.18
Total coliforms	1100 MPN100 mL^{-1}	99.99
Fecal coliforms	1100 MPN 100 mL^{-1}	99.99
Helminth eggs	200 HE L^{-1}	91.88

Polarization and Power Density Curves

Figure 4 shows the MSP-BES polarization and power density curves at an optimized HRT of 5 h. The maximum power density achieved was 6.23 mW m^{-3} at a current density of 14.41 mA m^{-3}. The internal resistance measured was 1414.49 Ω, a value higher than that commonly reported in the literature (Dekker, Heijne, Saakes, Hamelers, and Buisman, 2009; Ewing *et al*., 2014; Samsudeen, Sharma, Radhakrishnan, and Matheswaran, 2015). This elevated resistance is likely due to factors such as; a) the low electrical conductivity of the synthesized electrodes (1.54×10^{-9} S/m), b) the high feed flow of the influent to the system (as discussed earlier), c) the inherent resistance of the cation exchange membrane employed in this research, and d) increased ohmic losses due to the large distance between the electrodes (Leong, Daud, Ghasemi, Liew, and Ismail, 2013; Moreno-Cervera, Aguilar, Domínguez, Cámara, and Alzate, 2019). These findings suggest the need for more detailed research to fully understand the factors limiting MSP-BES performance.

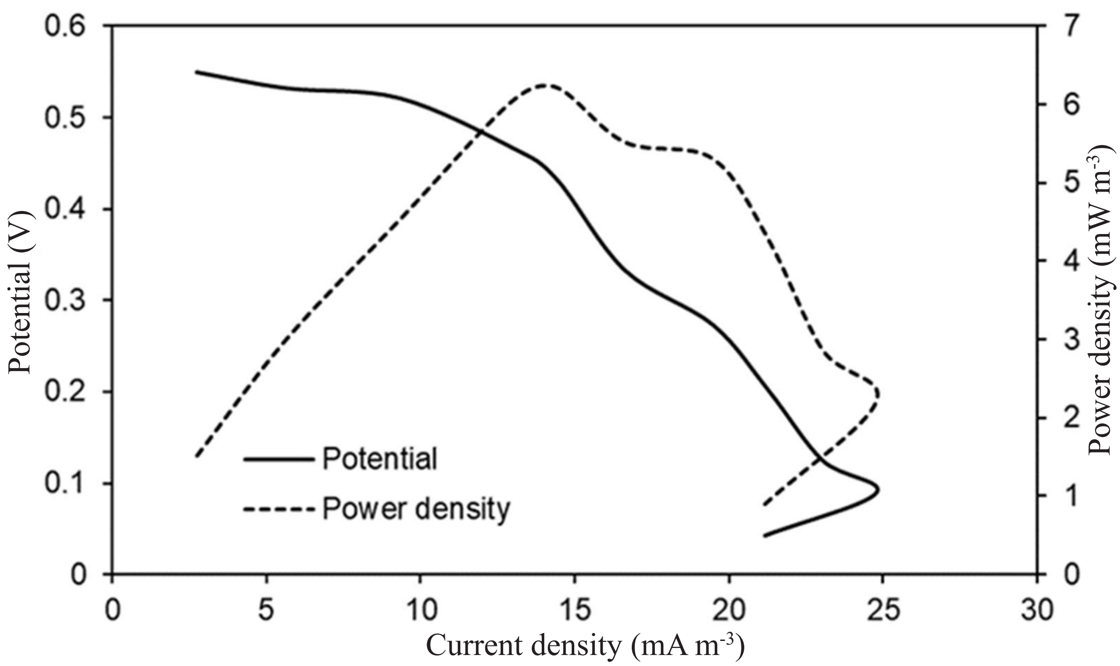

Figure 4. Polarization and power density curves of MSP-BES at a 5 h HRT.

CONCLUSIONS

The semi-pilot scale modular bioelectrochemical system maintained good performance proportional to the increase in HRT. In a 5-h HRT, energy generation in terms of maximum power density and coulombic efficiency was superior, as was removing contaminants, especially high efficiency in eliminating pathogens (TC, FC, and HE). Studies of this modular BES with an HRT greater than 5 h should be carried out to increase the percentages of COD, BOD, TS, and TSS removal efficiencies. Likewise, strategies (for example, isolation of exoelectrogenic microorganisms from the anodes for bioaugmentation purposes) should be established to increase energy production so that the operation of the BES is more economically sustainable.

ETHICS STATEMENT

Not applicable.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF SUPPORTING DATA

The datasets used or analyzed during the current study are available from the corresponding author on reasonable request.

COMPETING INTERESTS

The authors declare that they have no competing interests.

FINANCING

This research was funded by the Fondo Destinado a Promover el Desarrollo de la Ciencia y la Tecnología, COECYT-Gobierno del Estado de Coahuila, grant number COAH-2022-C19-C037.

AUTHORS' CONTRIBUTIONS

Conceptualization: P.P.R., and S.Y.M.A. Investigation: P.P.R., A.M.L., S.Y.M.A., and J.A.R.G. Methodology: P.P.R., A.M.L., D.A.C., and B.V.B.L. Design and construction: A.V.R.A. Validation, B.V.B.L., and A.V.R.A.; writing-original draft preparation, P.P.R., and D.A.C.; Writing-review and editing, S.Y.M.A., and J.A.R.G.

ACKNOWLEDGMENTS

Not applicable.

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