

Simultaneous electricity and biogas generation of vinasses and cattle manure

Producción simultanea de electricidad y biogás a partir de vinazas y estiércol de vaca

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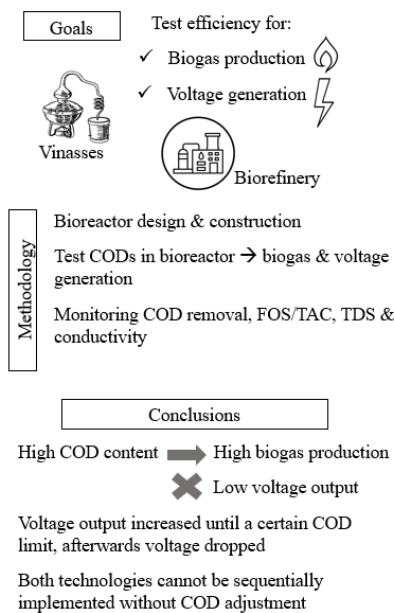
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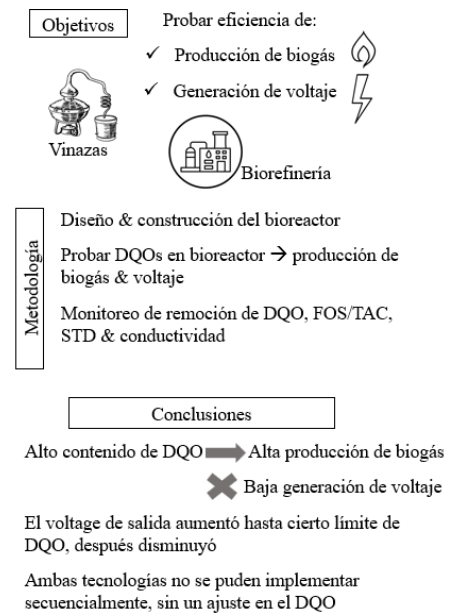
Abstract

A new design for the simultaneous generation of electricity and biogas was constructed. The voltage production in a microbial fuel cell and the biogas generation through anaerobic digestion were tested at different concentrations of mezcal vinasses and cattle manure. When comparing the control test with the concentrations using vinasses, these resulted in inhibition of voltage output. On the contrary, if no vinasses were used, no biogas production took place, revealing that the inoculum did not have activity itself. The concentration with the lowest organic matter content showed the poorest AD efficiency and lowest voltage output. By increasing the organic matter, power density increased until a certain limit. Contrary to the effect of organic matter content on the voltage production, biogas yield and methane content increased with increased organic matter. These results show that the combination of these technologies is not suitable for the simultaneous voltage production and biogas generation.



Resumen

Se probó simultáneamente la producción de voltaje en una celda de combustible microbiana y la generación de biogás mediante digestión anaerobia en diferentes concentraciones de vinazas de mezcal y estiércol de vaca. Al comparar la prueba de control con las concentraciones usando vinazas, estas resultaron en inhibición de la producción de voltaje. Cuando no se utilizaron vinazas no se produjo biogás. La concentración con el menor contenido de materia orgánica mostró la peor eficiencia de digestión anaerobia y la menor salida de voltaje. Al aumentar la materia orgánica, la densidad de potencia aumentó hasta cierto límite. Contrariamente al efecto del contenido de materia orgánica sobre la producción de voltaje, el rendimiento de biogás y el contenido de metano aumentaron con el aumento de materia orgánica. Estos resultados muestran que la combinación de estas tecnologías no es adecuada para la producción de voltaje y generación de biogás simultáneamente.



Bioenergy, Efficiency, Microbial

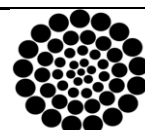
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Introduction

There are a broad variety of technologies either on use or on development, for the utilization of organic wastes to generate bioenergy. The concept of a biorefinery comprehends an integrative overall concept, where the biomass will be sustainable converted into biofuels, bioplastics or chemical intermediates (FNR 2012, Garcia and Ensinas 2024).

Conversion routes for bioenergy production are mostly thermal and biochemical technologies. Anaerobic digesters and microbial fuel cells (MFC) are both suited technologies for biomass treatment and energy production. Anaerobic digestion (AD) is one of the most common technologies of bioenergy production at industrial scales, while MFCs have not yet found significant practical applications.

Through MFC technology electrogenic bacteria is used to oxidize a great amount of substrates such as glucose, acetate, organic acids or also inorganic substances like sulfates. MFCs transform chemical energy contained in the substrate into electricity by means of REDOX reactions. Therefore, through bacterial respiration, the reduction and oxidation of organic molecules take place (Higgins et al. 2013). Through AD almost all organic wastes can be anaerobically degraded for biogas and methane production. The nutrient-rich digestate remaining after AD is normally used as fertilizer. Nevertheless, it has been demonstrated, that an important amount of gases, such as methane NH_3 and N_2O remaining in the digestate, are released to the environment when using as a fertilizer. This is still an important and challenging topic since the use of biomass should be climate neutral (Lukehurst et al. 2010, Menardo et al. 2011, Rico et al. 2011 and Rosillo-Calle 2016). If the digestate is used before its release in the environment, gas emissions could be diminished.

Not only the efficient bioenergy generation but also the removal of organic matter is pursued in laboratory and industrial scales. A plenty amount of substrates, such as vinasses, consist of a high concentration of mineral salts and high recalcitrant organic matter in terms of chemical and biological oxygen demand (COD and BOD).

Besides bioenergy generation, the target is to reduce the degradable organic matter, convert major toxic organic substances to compounds that can be easily biodegraded and reach the permissible levels of contaminants in waste discharges.

Until now, the power generated by MFCs is low for large-scale wastewater treatment. According to Vilas Boas et al. (2022) power outputs of 4 W/m^2 could be achieved. A MFC design used on large scale produces electricity by embedding an anode in sediment and connecting it to the cathode, which is placed in the overlying aerobic seawater, through an electrical circuit (Pant et al. 2010).

The development of this technology is challenging, especially because of the cost of membrane and electrodes, potential of substrate-biofouling and high internal resistance that limits the power generation (Slate et al. 2019).

Some improvement on MFC design point out that the use of open air bio-cathodes and replacement of platinized with non-platinized cathodes, as well as the use of stainless steel and nickel or manganese dioxide cathodes are alternatives to be used (Pant et al. 2010). The research in regards to new alternative substrates for the efficient use of MFCs at large scale is necessary, especially regarding substrates with high organic loads which are produced in high amounts.

A variety of vinasses conversion technologies has been developed and implemented for biomass combustion and gasification, anaerobic digestion to biogas, fermentation to bioethanol, and direct electricity production using microbial fuel cells (Gbadeyan 2024).

Coupling AD and MFC could be one step of a biorefinery concept, where the effluent of AD is used in MFCs. Few research regarding coupling AD and MFC technologies has been carried out. Typically, AD has been used for COD reduction, especially when processing high strength wastewaters. MFC was proposed for AD effluents treatment and for enhancement of organic matter removal.

In the practice, total ammonia nitrogen hinders COD removal during AD, if AD effluent could be used in a MFC, ammonia nitrogen could be removed (Higgins et al. 2013, Kim et al. 2015). Also the diminution of toxic gases released to the environment could be goaled, when using digestate in MFC. No studies have described the approach of AD and MFC technologies operated with vinasses, for the simultaneous biogas and electricity production. This substrate is, due to the low pH-value and high organic content, very promising not only to produce electricity, but also to treat the vinasses before they are being discharged onto soils and water. On this account, aim of this work is to test a new design developed as a small biorefinery, where the digestate of vinasses AD could be used as input material for the MFC operation. The configurations of open air cathode and membrane-less MFCs will be studied to make this technology a low-cost alternative for green energy production at large scale.

This paper intends to present the research of a biorefinery concept, in which the AD digestate could be used as input material on a MFC.

Based on that, section 2 describes the methodology proposed and used to perform this research, including the bioreactor design and operation, characterization of substrate and inoculum, as well as a description of the measurement methods. Section 3 describes the results and discussions regarding voltage output, biomethane and biogas production, as well as COD removal, FOS/TAC, TDS and conductivity. Section 4 summarizes the conclusions obtained through this work.

Methodology

Reactor design

The reactor consisted of three chambers placed side by side. The first chamber (under anaerobic conditions) was a bioreactor, in which 400 cm³ vinasses and cattle manure were digested. The second chamber, anodic chamber, was designed also to guarantee anaerobic conditions and had a fluid volumetric capacity of 400 cm³. The last chamber, cathodic chamber, was designed so that one side of the cathode had direct air contact, to guarantee aerobic conditions. Thus, the open air cathode would make the costs of MFC construction and operation much cheaper.

The volumetric capacity of the cathodic chamber was 700 cm³. The total fluid volume contained in the reactor was 1500 cm³. Anode and cathode were connected through an external resistance of 1000 Ω and a stainless steel wire of 0.7 mm diameter. The distance between anode and cathode was approximately 7 cm. Anode and cathode were made of activated carbon felt, with volumes of 115 cm³ and 255 cm³, correspondingly. Anode and cathode were inoculated with cattle manure, one month before experiments startup. The reactor was kept under mesophilic temperatures around 32 – 33 °C, with a ceramic hotplate SP88857100 from Thermo Scientific. A 250 ml Erlenmayer flask was connected to the first chamber, in order to collect the daily biogas produced. An external feeding tank with the mixture mezcal vinasses and cattle manure was placed next to the reactor and was connected to it by a peristaltic pump TS7892K07 from Thomas Scientific. The effluent from the reactor was recirculated to the feeding tank. The reactor was designed with the solid modeling computer-aided design (CAD) Solidworks 23 and the simulation of the substrate flow was done with the computational fluid dynamics (CFD) software ANSYS Fluent 14.5. The CFD simulation was done in order to analyze the dynamics and fluid displacement in the reactor, according to the fluid density, anode-cathode porous mediums related to a loss of pressure and the minimal inlet velocity reached with the available peristaltic pump. With the CFD simulation, the correct dimension regarding the inlet diameter, could be found. Figure 1 shows the designed bioreactor and figure 2 shows the CFD analysis performed in ANSYS.

Box 1

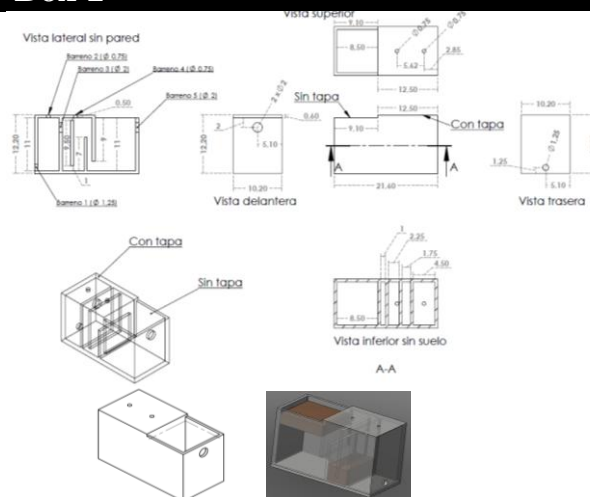


Figure 1
Scheme of the designed bioreactor

Source: Own elaboration

The streamlines show the path that a mass particle (substrate) would take through the current flow. The lowest speed achieved with the peristaltic pump was 0.347 cm³/min. According to [1] for flow rate estimation, the calculated flow speed, with the optimal inlet diameter of 0.0125 m, was 4.172 x 10⁻⁵ m/s, which corresponds to the velocity streamline in the green area of figure 2. With the parameters of inflow velocity and inlet diameter, no accumulation of sediments could be seen through the CFD simulation. After simulation, reactor was manufactured from an external supplier, according to figure 1.

$$V = \frac{V \pi \phi^2}{4}$$

[1]

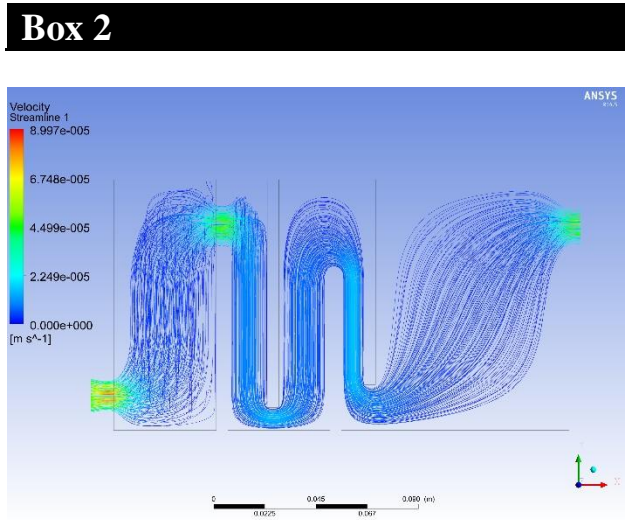


Figure 2
Streamlines velocity analysis in reactor in m/s
Source: Own elaboration

Reactor operation

Experiments were carried out for 92 days. Four different concentrations of mezcal vinasses digested with cattle manure, diluted with deionized water, were tested. Concentration were tested one after the other. Each concentration test was interrupted, when whether voltage nor biogas were produced. CA was tested for nine days, CB for 24, CC for 26 and CD for 23. A control test, with only cattle manure was performed for 12 days at the beginning of the assays. Table 1 shows the tested concentrations CA, CB, CC and CD, chemical oxygen demand (COD) in mg/L related to each concentration, as well as substrate to inoculum ratio (SI-ratio).

Box 3

Table 1

Tested concentrations CA, CB, CC and CD, chemical oxygen demand COD (mg/L) and substrate to inoculum ratio (SI-ratio)

	Control	CA	CB	CC	CD
Mezcal vinasses (L)	0	0.4	0.6	0.8	1
Cattle manure (L)	3.5	1	1	1	1
Deionized water (L)	3.5	4	4	4	4
COD (g/L)	12.20	8.95	11.90	14.85	17.80
SI-ratio	N/A	0.26	0.40	0.53	0.66

Source: Own elaboration

Inoculum and substrate

Vinasses generated from the mezcal production from Agave Salmiana were collected from the mezcal factory Laguna Seca in San Luis Potosí, Mexico and were stored in the refrigerator at 4 °C prior to use. Cattle manure was collected from a local pasture-raised dairy and was left at room temperature, in order to eliminate the microbial activity of the inoculum itself (VDI 2016). Table 2 shows the characteristics measured in both, mezcal vinasses and cattle manure, before the experiment started.

Box 4

Table 2

Characteristics of cattle manure and mezcal vinasses

	Cattle manure	Mezcal vinasses
pH @ 27 °C	7.95	4.41
Chemical oxygen demand COD (g/L)	12.20	63.73
Total solids TS (% FM)	3.70	5.26
Volatile solids VS (% FM)	1.80	2.88
Total dissolved solids TDS (g/L)	8.11	5.87
Conductivity (µS/cm)	12.24	11.75
REDOX (mV)	-211.00	-142.00
Volatile organic acids (gHAc/L)	1585.00	N/A
Total inorganic carbonate (gCaCO3/L)	9525.00	N/A
FOS/TAC (volatile organic acids/total inorganic carbonate)	0.17	N/A

Source: Own elaboration

Measurements

The amount of biogas was determined according to the water displacement principle. The water displaced from the Erlenmeyer flask connected to the first chamber, was weighed with a digital scale from Media Data PS-5 and converted to volume biogas, according to the biogas density 1.2 kg/m³ (Uni Bremen 2009). The biogas quality, regarding CH₄, CO₂, O₂, H₂S and CO contents, were measured with a biogas analyzer Multitec 540 from Sewerin GmbH.

The voltage produced between the anode and cathode was daily recorded with a Fluke 115/EFSP Digital Multimeter. Power density P was estimated according to [2] and current I according to [3], where R means resistance, V means voltage and V_{anode} means the volume of the anodic chamber. Polarization curves were calculated with Excel 2013 and plotted with the software Minitab 17.

$$P = \frac{V \times I}{V_{anode}} \quad [2]$$

$$I = \frac{V}{R} \quad [3]$$

Each concentration was characterized at the beginning and end of every test, regarding pH, REDOX, FOS/TAC, TDS (ppm) and conductivity ($\mu\text{S}/\text{cm}$) with a waterproof tester from HANNA Instruments HI-98311 and a pH-meter VWR-110. FOS/TAC, the ratio of volatile organic acids and total inorganic carbonate, was measured throughout the titration of sulfuric acid 0.05 M (H_2SO_4) to pH 5 and 4.4 (Lossie and Pütz 2008). FOS indicates the amount of volatile organic acids, mostly acetic acid (mgHAc/L) and TAC indicates the total inorganic carbonate or buffer capacity (mgCaCO_3/L) (Mézes et al. 2011, Moerschner 2015). COD, total solids (TS) and volatile solids (VS) were measured according to the norms DIN 38414-9:1986-09 (DIN 1986) and VDI 4630 (VDI 2016).

Results and discussion

Voltage output

The results of the voltage produced are shown in figure 3. It can be said, that vinasses content in substrate inhibit the voltage production. Control test was tested for 12 days and achieved the highest voltage of 0.436 V already the third day. CA, with the lowest COD and vinasses content, showed the worst results, producing only 0.032 V the second day of tests and was carried out only nine days. CB produced 0.202 V by day 14 and was carried out for 22 days, while CC generated the highest voltage output 0.317 V by day 15 and was carried out for 26 days. CD was carried out for 23 days and achieved 0.25 V by day 19.

Few values for Agave vinasses were found for comparison. López-Velarde S. et al. (2017) used vinasses diluted with deionized water for the electricity production in aerated-cathode with proton exchange membrane (PEM) at batch conditions. For short term operation (10 days), using a COD of 4060 mg/L, the highest voltage output of 0.12 V was achieved. The highest vinasses content with a COD of 17143 mg/L produced the lowest voltage output of 0.04 V. Results of the presents study showed a higher voltage and power output, using an open air cathode with no PEM at continuous operation. When comparing the results of the present work with the results of the long term operation (70 days) reported by López-Velarde S. et al. (2017), results in this assay show lower values.

Box 5

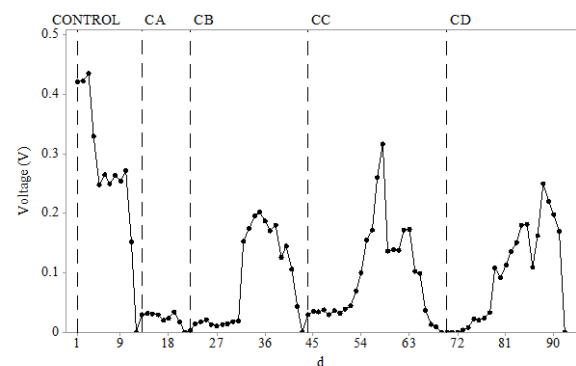


Figure 3
Voltage produced by control test, CA concentration, CB concentration, CC concentration and CD concentration

Source: Own elaboration

Another finding in this study was that the highest the vinasses content, the longest the time to achieve the highest voltage output. This suggests that the microorganisms in the anode took more time to oxidize substrate with high organic matter content. This fact can be confirmed by Vogl et al. (2016), who reported that easily degradable substrates produce higher power densities. In this study the voltage output and power density increased with increased vinasses content (CODs of 8950, 11900 and 14850 mg/L), until a certain point (COD of 17800 mg/L). Afterwards a voltage drop took place, when the saturated state of the electrolyte inhibit the oxidation mechanisms. Figure 4 shows the polarization curves of the concentrations tested.

When considering only the assays with vinasses, the highest power and current densities were generated with CC, 251 mW/m³ and 792 mA/m³, correspondingly. The worst results were obtained with CA, which contained also the lowest vinasses content and COD.

Results of this assays are comparable to Belafi-Bako et al. (2014). AD effluent, from a sugar factor wastewater plant was used in a MFC for electricity production. With a COD of 7150 mg/L, the highest power density of 8652 mW/m² was achieved. The lowest power density was generated with the highest COD of 19800 mg/L. Nam et al. (2010) found similar results. The highest power density of almost 3 W/m² was obtained with an organic loading rate (OLR) of 3840 mg/L*d, whilst an increase of OLR to 4800 m/L*d resulted in a decrease of the power density. The reduction and oxidation reactions of the microorganisms adhered in the anode determines the power output of a MFC. If the electrolyte solution has a high organic matter content, a difficult electron and proton transfer takes place (Nam et al. 2010). Schievano et al. (2016) reported that MFC performance decreased at higher COD concentrations, which were tested to avoid high dilutions of the AD effluent.

CA produced 63 L_{biogas}/kgVS_{vinasses} with a highest methane content of 45 %. CB produced 257 L_{biogas}/kgVS_{vinasses} with a highest methane content of 57 %. CC generated 127 L_{biogas}/kgVS_{vinasses} and achieved a methane production of 35 %, and CD produced 347 L_{biogas}/kgVS_{vinasses} with the highest methane content of the concentrations tested (59 %). The highest biogas production was generated by CD. Similar results were reported by Alkhrissat (2023), who achieved the highest cumulative methane production, with the highest substrate concentration tested, whilst the minimum concentration tested resulted in a methane production reduction. CB showed the highest methane production of 117 L_{CH₄}/kgVS_{vinasses}, one of the highest methane content of 57 % and showed also the highest COD removal of 93 %, suggesting a successfully conversion of organic matter into methane. CC, which produced the highest voltage output, as well as power and current densities, did not show significant biogas and methane yields, and showed the lowest methane content of 35 %. CA showed the worst results regarding not only electricity production, but also AD efficiency. This indicates that the lack of organic matter available was insufficient for both electricity and biogas production.

Box 6

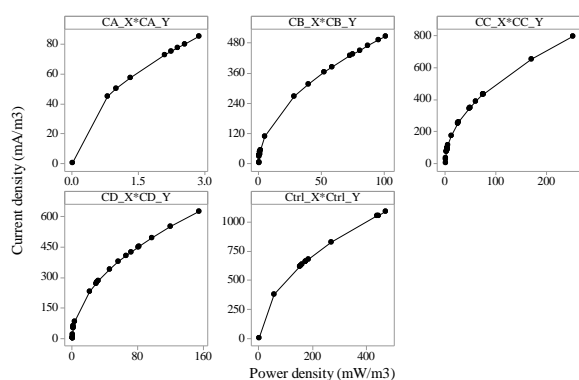


Figure 4
Polarization curves of control test, CA concentration, CB concentration, CC concentration and CD concentration

Source: Own elaboration

Biogas and methane production

The bar charts regarding the cumulative biogas and methane production generated by CA, CB, CC and CD are shown in figure 5.

An important finding was that CC produced less biogas and methane in comparison to CB and CA, but generated the highest power density and voltage output recorded. The conversion of organic acids into biogas was not successfully, but the microorganisms in anode could oxidize the organic acids to generate more voltage. Zhao et al. (2012) carried out experiments using the anode chamber as anaerobic digester. The results suggested that fermentation, more precisely the methanogenesis, compete with the electricity generation, what resulted on a power output and biogas yield. The coulombic efficiency was 2.79 % and the biogas yield persisted only 8 days with a maximum production of 0.285 L/d on a 15 L biodigester. Contrasting results were found by Li et al. (2015), who reported a decrement of methane production at higher organic loading rates, attributed to the high amount of Total Ammonia Nitrogen (TAN) levels in bioreactor when increasing substrate concentration, causing AD inhibition.

An adequate organic loading rate and thus TAN content should be done in order to ensure the process stability, so that operational failures, as well as TAN and FOS decrease could be avoided (Alkhrissat 2023).

Box 7

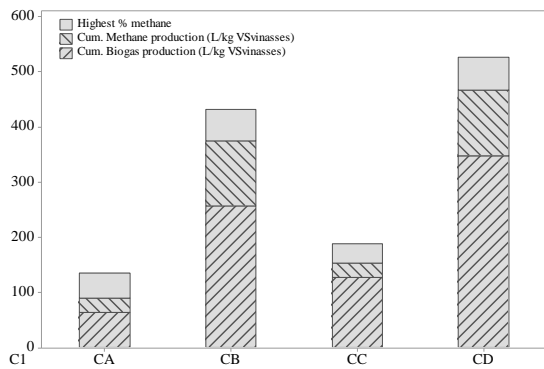


Figure 5

Cumulative biogas and methane production, as well as methane content generated by CA, CB, CC and CD

Source: Own elaboration

COD removal, FOS/TAC, TDS and conductivity

Table 3 shows the results regarding COD removal in CA, CB, CC and CD. CC showed the best removal of 93 % and CD the lowest. Results are comparable to López-Velarde S. et al. (2017), who obtained the worst COD removal with the highest COD tested. The high amount of organic matter results in a saturated state of the electrolyte inhibiting the oxidation mechanisms for COD removal.

Box 8

Table 3

Characteristics of cattle manure and mezcal vinasses

	CA	CB	CC	CD
Initial COD (mg/L)	8950	11900	14850	17800
Final COD (mg/L)	1343	833	2079	9434
COD removal (%)	85	93	86	47

Source: Own elaboration

The amount of TDS and conductivity were higher with a higher vinasses content and COD. Table 4 indicates the amount of volatile organic acids (FOS) and total inorganic carbonate (TAC), as well as the quotient FOS/TAC. With the increase of vinasses content, the volatile organic acids as well as the FOS/TAC increased.

The buffer capacity of the system decreased with a high COD. When comparing the beginning and end of each concentration test, the amount of volatile organic acids decreased dramatically, what indicates that they were quickly adhered to the anode. When comparing beginning and end of every concentration tested, the buffer capacity of the system achieved always higher values than at the beginning. A lowest REDOX was shown, when the vinasses content, COD and SI-ratio diminished. Although FOS/TAC values were optimal for CA and CB according to values proposed by Lossie and Pütz (2008) of 0.3-0.4, the separated FOS and TAC values were much lower than recommended. FOS values should be higher than 10000 mg/L and TAC values should lie between 8500 and 13000 mg/L (Mézes et al. 2011, Moerschner 2015). This was the reason of the low AD efficiency of the system. For concentration CC, the FOS/TAC value of 1.26 was higher than recommended by Moerschner (2015) and Lossie and Putz (2008). This resulted in a drop of the biogas and methane yields.

According to Kretzschmar et al. (2016), volatile fatty acids are correlated to the current production. Inhibitions in MFC were found out when the amount of organic acids increased above 4000 mg/L. In the present study, the best voltage output was obtained when the amount of FOS were 2166 mg/L, which showed also the highest amount of organic acid concentration.

Box 9

Table 4

FOS/TAC values measured at the beginning and end of each assay

	FOS mgHAc/L	TAC mgCaCO3/L	FOS/TAC
CA_I	713.5	1750	0.41
CA_F	174	2400	0.07
CB_I	838	1950	0.43
CB_F	49.5	2312.5	0.02
CC_I	2166	1725	1.26
CC_F	49.5	2625	0.02
CD_I	1253	1425	0.88
CD_F	215.5	2687.5	0.08

Source: Own elaboration

Conclusions

The combination of bioenergy technologies offers a wide range of both sustainable energy generation and byproducts further utilization.

Substrates consisting of high amounts of organic and inorganic matter should stay in focus for treatment before discharge in soil and water. Through this study, the consecutive implementation of AD and MFC technologies was tested with mezcal vinasses and cattle manure as inoculum source for AD. It was found out that the use of vinasses in MFC inhibits the process of voltage generation. Besides, MFCs do not tolerate substrates with high amounts of COD. In the present study, voltage output increased with increasing vinasses content, until a certain limit of 14850 mg/L.

The highest COD tested was 17800 mg/L, at which the voltage dropped. In regards to biogas, the higher the COD content, the higher the biogas yield and methane content. It can be concluded that both technologies cannot be sequentially implemented without COD adjustment. This study presents an alternative for the further and deeper investigation of the use of both technologies consecutively, so that the organic acid content in substrate could result in a successfully MFC operation. Computational modeling could be used to simulate and study the whole biorefinery system using mathematics, physics and computer science. Through this tool the combination of alternatives could be analyzed regarding not only to the technological processes involved, but also economic feasibility

Declarations

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

López-Velarde S., Mónica: Contributed to the project idea, methodology and development.

Rodríguez-Morales, José Alberto: Contributed to the development and revision.

Mendoza Burguete, Yesenia: Contributed to the project idea and revision.

Hensel, Oliver: Contributed to the project idea and revision.

Availability of data and materials

Data related to this research is available at the Kassel University Library in Campus Witzenhausen.

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Abbreviations

AD	Anaerobic digestion
BOD	Biological oxygen demand
CaCO ₃	Total inorganic carbonate
CAD	Computer aided design
CFD	Computational fluid dynamics
CH ₄	Methane content
CO	Carbon monoxide
CO ₂	Carbon dioxide
COD	Chemical oxygen demand
FOS/TAC	Flüchtige organische Säuren / Total anorganic carbonate
H ₂ S	Hydrogen sulfide
H ₂ SO ₄	Sulfuric acid
HAc	Acetic acid
MFC	Microbial fuel cell
N ₂ O	Nitrous oxide
NH ₃	Ammonia
O ₂	Oxygen
PEM	Proton exchange membrane
REDOX	Reduction-oxidation reaction
SI-ratio	Substrate to inoculum ratio
TAN	Total Ammonia Nitrogen
TDS	Total dissolved solids
TS	Total solids
VS	Volatile solids

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Antecedents

Garcia V.F. and Ensinas, A.V. (2024). [Simultaneous Optimization and Integration of Multiple Process Heat Cascade and Site Utility Selection for the Design of a New Generation of Sugarcane Biorefinery](#). *Entropy*, 26(6), 501.

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