

Characterization of biogas and effluent produced during the anaerobic digestion of mezcal vinasse

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Abstract: The production of mezcal generates vinasse, a liquid waste with high concentrations of organic matter and acidic pH, which represents a potential source of pollution. The use of anaerobic biodigesters has emerged as an alternative for the treatment of this waste stream. This study characterizes the anaerobic digestion (AD) of mezcal vinasse in a 10 m³ tubular (bag-type) anaerobic biodigester. To this end, daily biogas production was measured and normalized to standard conditions. The methane content in the produced biogas was determined through carbon dioxide (CO₂) removal, and the thermal efficiency of a biogas stove was evaluated. The physicochemical properties of the liquid effluent resulting from the AD process were also assessed. The results demonstrated that the AD treatment of vinasse with an initial Chemical Oxygen Demand (COD) concentration of 44.93 g L⁻¹ yielded 15.61 L of biogas per liter of vinasse, with a methane concentration of 68.48%. Furthermore, the thermal efficiency of the biogas stove was 35.45%. The effluent obtained had a pH of 7.5 and a COD concentration of 17.00 g L⁻¹, which represents a 62.16% COD removal relative to the influent. Additionally, its organic carbon content reached 17%, while total nitrogen accounted for 19%. These results indicate a high potential for the proposed approach to be implemented as a strategy for waste valorization method, pollution mitigation, local clean energy production, and agroecological applications.

Keywords: anaerobic digestion, biogas, mezcal vinasses, biofertilizer, renewable energy

Introduction

In recent years, the mezcal industry has experienced rapid growth, reaching a production of 12.239 million liters in 2023. Mezcal is produced in nine Mexican states holding the designation of origin for its commercialization. Oaxaca is the largest producer, accounting for over 90% of the total national production (COMERCAM, 2024). The growth of this industry has brought significant environmental challenges, particularly as the generation of vinasse, a byproduct of the mezcal distillation process. This substance exhibits high concentrations of organic matter (biochemical oxygen demand, BOD₅: 22,500–35,000 mg L⁻¹ and COD: 35,000–122,000 mg L⁻¹) and high acidity (pH between 3 and 5) (Robles-González *et al.*, 2012). Vinasse generation can reach quantities of 10–12 L/L of final distillate, and the majority of this waste is not properly managed or treated; instead, it is dumped directly into drains, soil, rivers, or lakes at high temperatures, posing serious risks to aquatic and terrestrial ecosystems (Crespo-González *et al.*, 2018; Jiménez *et al.*, 2006). Alternatively, some producers choose to store it; however, due to the lack of adequate treatment systems, they are forced to hire services to empty these containers or tanks without regulatory oversight which merely relocates the waste management problem rather than resolving it.

Anaerobic digestion (AD) is an alternative method for treating organic waste from various sources, including vinasse (Silva *et al.*, 2021; Parsaei *et al.*, 2019). This process allows the conversion of organic substrates into a gaseous mixture known as biogas, which has a high methane content, making it suitable for clean energy production (Ullah Khan *et al.*, 2017). In addition, the digestate generated during AD comprises partially digested organic matter, micro- and macronutrients, and water. This effluent contains essential nutrients in highly bioavailable forms, making it a valuable biofertilizer and soil conditioner (Nayak & Ranade, 2025). Thus, the anaerobic digestion of organic residues is related to important socio-environmental co-benefits, including improved waste management, clean energy generation from local inputs, mitigation of greenhouse gas (GHG) emissions, support for the circular economy, contribution to energy independence, and more sustainable agricultural and livestock practices. In this context, the use of AD has gained popularity in various parts of the world, with more than 20 million systems in China, 4.3 million in India, and

approximately 7,000 in Germany; these three countries are reported to currently have the highest number of anaerobic digestion systems (Zelaya-Benavidez, 2022; Zelaya-Benavidez *et al.*, 2023).

A variety of anaerobic digester designs are commercially available; however, some of these systems tend to be more expensive and complex. Nevertheless, the selection of a system should consider the available resources, user characteristics, and specific features of the installation region (Parsaee *et al.*, 2019). Zelaya-Benavidez (2022) proposed the use of tubular anaerobic digesters made of polyethylene to treat mezcal vinasse, as they are less expensive and easier to handle than concrete digesters (Zelaya-Benavidez, 2022). In comparison, digesters made of PVC geomembranes, although more expensive, offer longer service life and greater mechanical resistance during installation and operation. In this context, the objective of this study was to evaluate the applicability of PVC tubular anaerobic digesters for the treatment and valorization of mezcal vinasse. To this end, the products resulting from the anaerobic digestion of mezcal vinasse—biogas and liquid effluent (digestate)—were characterized, and the methane content and thermal efficiency of biogas were measured. The results of this study highlight the effectiveness and benefits of treating vinasse using anaerobic biodigesters as an appropriate and sustainable technology for the mezcal industry.

Materials and Methods

Study location

The study was conducted at the “Mezcal Capotlan” palenque located in the Arrazola neighborhood of Santa Cruz Xoxocotlán, Oaxaca, Mexico (17° 2' 27.7" N, 96° 46' 59.8" W). The site was equipped with a functioning tubular biodigester, also known as a bag-type, with a liquid capacity of 10 m³, manufactured by ARQUEA®, using a PVC geomembrane (Figure 1).

Biodigester management

The system was started with fresh bovine manure and mezcal vinasse (VM) at a 1:3 ratio (manure: vinasse, V/V), and the mixture was stabilized with commercial calcium hydroxide (Ca (OH)₂) from the brand CALHIDRA®. During the data collection period, the biodigester system was continuously fed 166 L of vinasse per day. The pH of the vinasse was adjusted between 6.3 and 9.9 by adding calcium hydroxide. The vinasse used for the experiment was obtained from an underground cistern on-site, which collected vinasse generated during mezcal distillation in the alembics.



Figure 1. A tubular or bag-type biodigester with a liquid volume of 10 m³ was used in the present study

The substrate and effluent were characterized through physicochemical analysis performed by a laboratory accredited by the Mexican Accreditation Entity (EMA). The effluent sample was collected from the outlet of the biodigester on day 21 of operation. The tests performed included microwave digestion/inductively coupled plasma optical emission spectroscopy (ICP-OES) for the determination of phosphorus, K, Ca, Mg, Na, sulfur, Fe, Cu, Mn, Zn, and B; and loss-on-ignition (calcination) for the quantification of organic matter, ash, and organic carbon. The pH levels were measured using a Yieryi® C-600 pH meter in liquid samples collected from both the inlet and outlet streams of the biodigester.

Analytical methods for determining biogas production

Biogas production was measured over a 21-day period, with a 24 h measurement frequency, using a KEUK DONG KI JEON® G-2 model low-pressure analog diaphragm gas meter. The ambient temperature of the site was recorded throughout the experiment, taking readings, every 10 min using an Elitech® RC-5 model temperature sensor.

The daily biogas production was normalized to standard conditions (27.15 K and 1013 hPa) as presented in Equation 1 (Dinuccio *et al.*, 2010):

$$V_0^{NPT} = V \frac{(P - P_v)T_0}{P_0T} \quad (1)$$

where V_0^{NPT} represents the volume (L) of dry biogas normalized to standard pressure and temperature, V is the recorded biogas volume (L), P is the biogas pressure at the time of measurement (hPa), T is the ambient temperature (K), T_0 is the standard temperature (K), and P_v is the water vapor pressure in mmHg as a function of the temperature of the measurement site in hPa, as obtained by Equation 2:

$$P_v = 10\left(A - \frac{B}{T + C}\right) \quad (2)$$

where T is ambient temperature ($^{\circ}\text{C}$). A , B , and C are parameters of the Antoine Equation constants for water. Under the experimental conditions, their values were 8.0713, 1730.6300, and 24.6900, respectively.

Methane content in biogas

The methane content in the produced biogas was determined by CO_2 absorption using a 20% KOH solution combined with the liquid water displacement method (Córdova *et al.*, 2022) (Figure 2). The methane percentage was calculated using Equation 3 (Abdel-Hadi, 2008):

$$\% \text{CH}_4 = 100\% - \left[\left(\frac{V_1 - V_2}{V_1} * 100 \right) + 3\% \right] \quad (3)$$

where $\% \text{CH}_4$ represents the methane concentration in the biogas, V_1 is the initial biogas volume (mL), and V_2 is the remaining gas volume after CO_2 removal (mL).



Figure 2. Mechanism of CO_2 removal from biogas

Thermal efficiency

The thermal efficiency of the burner was determined using the “Water Boiling Test” method, which relates the energy transferred to water and the energy released by biogas combustion, as expressed in Equation 4 (Clean Cooking Alliance, 2014):

$$h_c = \frac{\left[\left(\frac{M_a * \Delta T * c}{100} \right) + (M_{av} * H) \right]}{V_{biogas} * \% \text{CH}_4 * PPC_{\text{CH}_4}} \quad (4)$$

where h_c is the thermal efficiency (%), M_a is the initial mass of water (kg), ΔT is the temperature increase until boiling point (K), c is the specific heat capacity of water (4.2 kJ kg^{-1}), M_{av} is the mass of evaporated water (kg), H is latent heat of vaporization (2.26 kJ kg^{-1}), V_{biogas} is the volume of biogas consumed (m^3), $\% \text{CH}_4$ is the methane content of the biogas (calculated by Equation 2), and PPC_{CH_4} is the lower calorific value of methane (34 MJ m^{-3}).

Results and Discussion

Site temperature

Figure 3 shows the site temperature records, with a minimum recorded temperature of $9.2 \text{ }^{\circ}\text{C}$ on day 1 and a maximum temperature of $38.0 \text{ }^{\circ}\text{C}$ on day 10. The average temperature during the measurement period was $24.78 \pm 1.16 \text{ }^{\circ}\text{C}$. These

temperature conditions fall within the growth ranges of both psychrophilic (4–20 °C) and mesophilic (20–40 °C) microbial communities, which are commonly involved in anaerobic digestion processes.

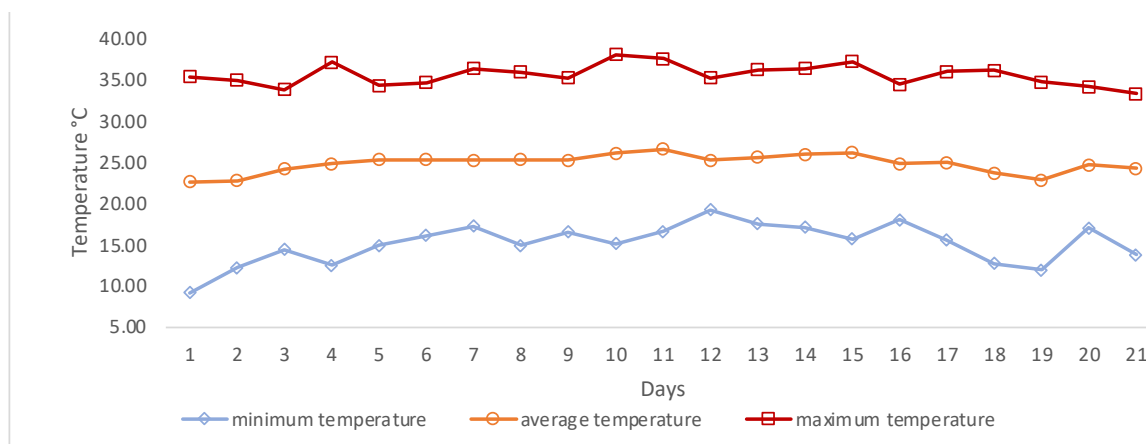


Figure 3. Temperature records of the site where the study was conducted

Physicochemical characterization of substrate and effluent

Table 1 summarizes the physicochemical properties of the mezcal vinasse used as feedstock for the biodigester, as well as those of the digestate (effluent) obtained after anaerobic treatment. The pH of the vinasse indicates a partial degradation of the substrate during storage prior to digestion, as evidenced by a reduction in acidity from fresh vinasse produced at the stills (on average pH=3.5) to a pH of 5.38 at the biodigester inlet.

Table 1. Physicochemical characteristics of vinasse and biodigester effluent (Biol)

Parameter	Units	Vinasse	Effluent
pH		5.38	7.50
Electrical conductivity	dS m ⁻¹	5.20	5.01
COD	g L ⁻¹	44.930	17.008
BOD ₅	g L ⁻¹	29.610	3.405
Organic matter	%wt	0.96	0.29
Ashes	%wt	0.41	0.29
Organic carbon	%wt	0.56	0.17
Total nitrogen	%wt	0.80	0.19
Phosphorus	mg L ⁻¹	56	400
Potassium	mg L ⁻¹	400	5700
Calcium	mg L ⁻¹	1300	10200
Magnesium	mg L ⁻¹	200	2300
Sodium	mg L ⁻¹	81	1000
Sulfur	mg L ⁻¹	82	500
Iron	mg L ⁻¹	114	294
Copper	mg L ⁻¹	6.76	36.30
Manganese	mg L ⁻¹	2.56	14.50
Zinc	mg L ⁻¹	2.04	20.90
Boro	mg L ⁻¹	1.36	11.40

U/M: unit of measurement

Vinasse entered the biodigester with an initial chemical oxygen demand (COD) concentration of 44.930 g/L. After anaerobic digestion, the effluent COD decreased to 17.008 g/L, representing a 62.15% reduction. This reduction

demonstrates the effectiveness of the biodigester in degrading the organic load present in vinasse and confirms its potential as a technically viable alternative for mitigating the environmental impact associated with mezcal production.

The COD removal efficiency obtained in this study (62.15%) fell within the range reported for the anaerobic digestion of vinasse and other distillery effluents. Díaz-Barajas *et al.* (2024) reported COD removal efficiencies close to 81% for mezcal vinasse under high dilution conditions, while removal efficiencies ranging from 49 to 82% have been reported for sugarcane vinasse treated in upflow anaerobic sludge blanket (UASB) reactors (Gomes de Barros *et al.*, 2016). In this context, the COD removal achieved in the present study suggests that the implemented tubular biodigester represents a robust and scalable option for the treatment of high strength mezcal vinasse.

Although the pH values of the incoming vinasse were below the optimal range for anaerobic digestion, the substrate was stabilized prior to feeding by the addition of commercial calcium hydroxide adjusting the pH to values between 7.3 and 9.9. This procedure ensured suitable pH operating conditions within the biodigester, where pH values remained between 6.7 and 7.5, which is considered optimal for anaerobic digestion (Rajendran *et al.*, 2012). Furthermore, the COD concentration of the vinasse used in this study (44.930 g L⁻¹) was lower than the values reported by Ibarra-Camacho *et al.* (2019) for vinasse samples produced in Cuban distilleries, where COD concentrations ranged from 51.53 to 54.25 g L⁻¹. Comparison between influent and effluent indicated a 62.14% COD removal and an 88.5% BOD₅ removal after anaerobic digestion, with a hydraulic retention time of 60 days.

Inlet and outlet pH

The pH of the liquid substrate within the biodigester remained within the optimal range for anaerobic digestion (6.7–7.5) throughout the operational period. The average inlet pH was 7.37 ±0.63, while the outlet pH was 7.38 (±0.15). During the initial 24 h of operation, the outlet pH temporally shifted toward alkaline values following substrate feeding, after 48 h of operation, the outlet pH reached a neutral value (Figure 4).

Vinasse load and biogas yield

During the evaluation period, the biodigester was fed with an average of 200 L of mezcal vinasse with an influent COD of 44.93 mg L⁻¹, BOD₅ of 29.62 mg L⁻¹, and pH of 8.2 (± 1.12), which is slightly alkaline for anaerobic digestion. The biogas yield obtained in this study was 15.61 L of biogas per liter of vinasse fed, which is 3.27% higher than values reported in previous studies. However, the average methane content was 2.17% lower than that reported by Lorenzo-Acosta *et al.* (2015). The methane concentration ranged from 63.83 % to 74.25%, with an average of 68.48% (±2.9%), which is lower than those reported in the literature (Ferrer *et al.*, 2011; Lorenzo-Acosta *et al.*, 2015; Rivera *et al.*, 2020) (Figure 4). The biogas yield was 15.61 L/L of vinasse fed to the biodigester.

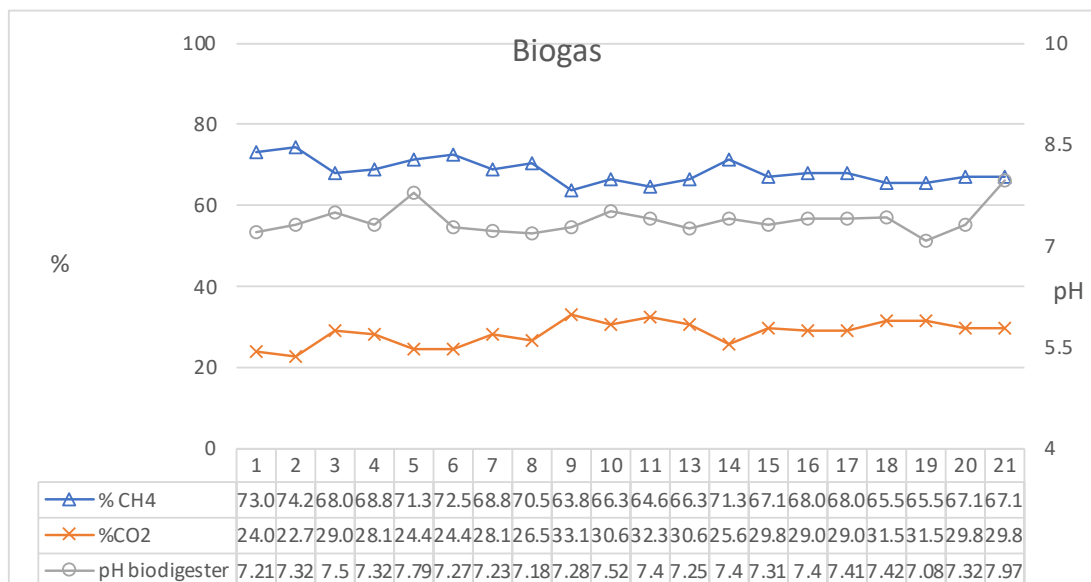


Figure 4. Amount of methane, carbon dioxide in the biogas and operating pH of the biodigester

To evaluate the potential social impact of biogas adoption, the biogas produced during the study was used for food preparation at the site and the volume of biogas consumed was recorded for each activity (Table 2). The results indicate that the daily biogas production is sufficient to meet the cooking energy requirements of approximately 15 people, using common food preparations such as beans, eggs, tortillas, and coffee.

Table 2. Food cooked with biogas and the amount of biofuel used

Food	Amount of biogas needed (m ³)
1 kg beans	2.350
30 eggs, 2.5 kg sausage	0.505
19 L of fish stock (10 kg fish)	2.460
2.5 kg stewed meat	1.581
1 kg rice	
5 L of coffee brewed in a pot	0.918
2.5 kg of fried tortillas	
5 L of coffee	0.257

Thermal efficiency of the biogas burner

The thermal efficiency of the biogas burner is a key parameter affecting overall energy conversion efficiency. In this study, thermal efficiency decreased with increasing valve opening and unfavorable wind conditions. The maximum thermal efficiency achieved was 35.45% corresponding to a 25% valve opening, while the minimum efficiency was 17.9% at a 100% valve opening, which is similar to the values reported by Zelaya-Benavidez (2023). These results emphasize the need for improved biogas stove designs to enhance energy utilization efficiency, particularly when compared with liquefied petroleum gas (LP) stoves, which can reach thermal efficiencies of up to 53% (Viquez-Arias *et al.*, 2018).

Conclusions

The rapid growth of mezcal production in Mexico has been accompanied by significant environmental challenges, particularly related to the inadequate management of organic residues such as vinasse. The results of this study demonstrate that anaerobic digestion is a technically and environmentally viable alternative for the treatment and valorization of mezcal vinasse enabling the simultaneous generation of renewable energy and nutrient-rich effluent with potential applications in soil improvement.

Specifically, the anaerobic treatment of 200 liters per day of mezcal vinasse in a 10 m³ tubular biodigester operating under psychrophilic-mesophilic conditions produced 15.61 L of biogas per liter of mezcal vinasse, with an average methane content of 68%. The biodigester a 62.16% COD removal efficiency while maintaining stable pH conditions near 7.38. The resulting effluent exhibited a high nutrient content, indicating its potential use as a biofertilizer or soil amendment. Biogas utilization in a conventional burner resulted in a maximum thermal efficiency of 35.45% highlighting both the feasibility of local energy recovery and the opportunity for further improvements in burner design.

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Author contributions: E. H.-S.: drafted the first version of the manuscript; J. S.-U.: study supervision; D. P.-M.: critical review of the manuscript, contributed to the final draft; E. Z.-B.: data analysis and interpretation.

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