

Valorization of orange solid waste through pyrolysis: production of biochar and its potential as an enhancer of the anaerobic digestion

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ABSTRACT

Objective: To valorize orange solid waste through pyrolysis to obtain biochar and to analyze its potential application as an enhancer of anaerobic digestion.

Design/methodology/approach: Orange solid waste was conditioned and subjected to pyrolysis at 550 °C in an Auger-type reactor. The produced biochar was characterized by measuring pH, ash content, total solids, volatile solids, cation exchange capacity, electrical conductivity, and carbon content. Additionally, an anaerobic hybrid reactor was conditioned and monitored by measuring pH, total and soluble COD, TSS, VSS, and biogas production to subsequently evaluate the effect of the biochar on the reactor performance.

Results: Biochar exhibited alkaline properties pH (8.6), a carbon content of 60%, and an increase in cation exchange capacity (42.6 meq 100 g⁻¹), indicating the development of a porous and conductive structure favorable for microbial adhesion and the mitigation of inhibitory compounds. Meanwhile, the anaerobic hybrid reactor was stabilized, maintaining a pH between 7.1 and 7.4, achieving 90% removal of total and soluble COD, as well as 4.6 L biogas/d, favoring a balanced biological environment.

Limitations on study/implications: The effect of biochar addition in the anaerobic hybrid reactor will be evaluated to determine its influence on anaerobic digestion performance. However, further studies are required to confirm its long-term stability and scalability.

Findings/conclusions: Biochar derived from orange solid waste represents an environmentally sustainable alternative to optimize the anaerobic digestion process and valorize agro-industrial waste within the framework of a circular economy.

Keywords: Orange solid waste, biochar, pyrolysis, anaerobic digestion, anaerobic hybrid reactor.

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INTRODUCTION

Citrus production constitutes one of the most important agricultural and agro-industrial activities worldwide. These fruits especially oranges provide bioactive compounds such as vitamin C, flavonoids, folic acid, and essential minerals, in addition to exhibiting antioxidant



and anti-inflammatory properties that contribute to human health (SIAP, 2022). Mexico ranks among the world's leading citrus producers, with more than 4.6 million metric tons annually. The state of Veracruz accounts for more than 40% of national orange production, making it a strategic region for the development of waste recovery projects (Carreño-López *et al.*, 2024). However, industrial citrus processing, especially in juice and essential oil production, generates large amounts of solid and liquid residues, being between 40% and 60% of the processed mass (Fernández-Echeverría *et al.*, 2024). The inadequate management of waste derived from orange processing poses both environmental and economic problems, so its valorization presents itself as a sustainable alternative for generating energy and value-added materials. Among the emerging technologies aimed at transforming agro-industrial residues is pyrolysis, which has gained relevance due to its capacity to convert organic waste into high value-added products (Méndez *et al.*, 2023). This thermochemical process involves the thermal decomposition of organic materials under an inert oxygen atmosphere, generally at temperatures between 300 °C and 800 °C. As a result, three main fractions are obtained: a gaseous phase, a liquid fraction (bio-oil), and a solid residue known as biochar (Jerzak & Li, 2024). Biochar has attracted significant interest due to its physicochemical properties, such as its high specific surface area and porosity, which give it a great capacity for adsorption, nutrient retention, and microbial support. It also contributes to carbon sequestration and the recovery of degraded soils (Pérez-Vázquez *et al.*, 2021). On the other hand, anaerobic digestion (AD), widely used in the treatment of organic waste and wastewater, converts organic matter into a mixture mainly composed of methane (CH₄) and carbon dioxide (CO₂), called biogas, which can be used as a source of renewable energy (Akor *et al.*, 2021). However, the treatment of citrus waste through anaerobic digestion faces difficulties associated with the presence of inhibitory compounds, including D-limonene, which affects methanogenic microbial activity. Several authors have indicated that the addition of biochar in the anaerobic reactors can mitigate these effects by adsorbing toxic compounds, stabilizing pH, and promoting direct interspecies electron transfer (DIET), resulting in increased methane production and greater system stability (Liu *et al.*, 2022; Gao *et al.*, 2023). Biochar can act as an electrochemical mediator, promoting DIET. This mechanism improves energy conversion efficiency during AD by facilitating the formation of conductive bridges between fermentative bacteria, leading to a more stable and efficient process (Zhang *et al.*, 2024). In particular, biochar derived from citrus residues has been the subject of various studies due to its potential to generate carbonaceous materials with remarkable functional properties. Imran & Chew (2025) reported obtaining biochar from orange peels and pulp, with specific surface areas exceeding 500 m²/g, applicable in both environmental remediation and energy systems. Jiang *et al.* (2020) demonstrated that this type of biochar can significantly improve biogas production in the co-digestion of sludge and food waste by facilitating electron transfer. Likewise, Wang *et al.* (2025) highlight the application of biochar within circular economy strategies, where the integration of thermal and biological processes allows for the closure of material and energy cycles, promoting the sustainability of the agro-industrial sector. Recent meta-analyses and review studies (Velasquez-Pinas *et al.*, 2025; Zhao *et al.*, 2021) have further emphasized the central role of pyrolysis and biochar systems in advancing

circular bioeconomy models through carbon recycling, waste valorization, and renewable energy generation. Similarly, Zhao *et al.* (2023) reported that the addition of biochar on the AD of organic waste not only increases methanogenic efficiency but also improves pH stability and electrical conductivity, fostering a more resilient and active microbial community. In this context, the valorization of orange solid waste (OSW) through pyrolysis presents itself as a promising alternative to transform an environmental liability into an energy resource and value-added material. Obtaining biochar from these residues not only helps reduce the negative impacts of their inadequate disposal, but also offers an effective technical option for optimizing AD. Unlike previous studies that have primarily evaluated biochar derived from generic agricultural or food wastes, this research focuses on biochar produced specifically from orange solid waste at 550 °C and its performance within an anaerobic hybrid reactor configuration. This integration of thermal valorization and biological treatment using a citrus-derived feedstock under controlled operational conditions provides novel insights into the optimization of biochar properties for enhancing anaerobic digestion efficiency. Therefore, the objective of this work is to valorize OSW through pyrolysis, obtaining biochar and to analyze its potential application as an additive or enhancer of the AD. This work contributes to the development of sustainable strategies for the comprehensive use of agro-industrial waste within the framework of a circular economy.

MATERIALS AND METHODS

Preparation of the OSW

To carry out the pyrolysis process, the OSW underwent a pretreatment in order to ensure suitable conditions for operation (Figure 1). First, the OSW was fragmented into smaller pieces measuring 2-4 cm, to facilitate drying and homogenize material size. The OSW was subjected to an open-air drying process, utilizing solar radiation for 10-14 hours. Once a moisture content below 10% was achieved, the OSW was crushed and sieved to obtain a uniform particle size between 1.0 and 3.8 mm to be subjected to pyrolysis.

Pyrolysis process application

Pyrolysis process was conducted in an Auger-type laboratory reactor (Patent CA 2830968), with a nominal capacity of approximately 1 kg h⁻¹, located at the Institut de Recherche et de Développement en Agroenvironnement (IRDA) in Quebec, Canada. The reactor was operated at a controlled temperature of 550 °C, selected based on literature

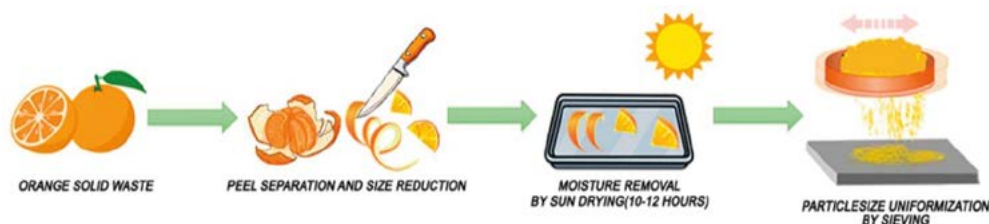


Figure 1. Pretreatment of the OSW (Prepared by the author).

reporting that mid-range temperatures (500-600 °C) favor optimal biochar stability, carbon retention, and surface functionality, which are desirable for applications in anaerobic systems (Zhao *et al.*, 2021). Previous studies involving orange-peel biochar, including studies focused on subsequent activation and characterization, also employed at 550 °C (Rizzioli *et al.*, 2024). Typical biochar yields reported for orange peels under slow pyrolysis conditions usually range from 20-35% (Rizzioli *et al.*, 2024) which is comparable to the 22% yield reported by Kordoghli *et al.*, (2023) and serves as a useful benchmark for the present study. Furthermore, the residence time of 104 seconds was established according to reactor design constraints and prior operational experience to ensure uniform heating and adequate volatile release without excessive degradation of the carbon matrix. A continuous nitrogen flow (3 L min^{-1}) was maintained as an inert gas to prevent combustion. The energy required was supplied by two 1.5 kW electric resistors installed in a copper block surrounding the main screw, ensuring homogeneous heat transfer during treatment. A total of 1.7 kg of dried OSW was subjected to pyrolysis.

Characterization of the OSW and biochar

Determinations of pH, moisture, ash, total solids (TS), volatile solids (VS), cation exchange capacity (CEC), and electrical conductivity (EC) were carried out in duplicate for both the OSW and the biochar, which was previously ground with a mortar and pestle until a homogeneous texture was achieved. All analyses were conducted following their corresponding standard methodologies. pH was measured according to AOAC (2016), moisture content following ASTM D2216-19, ash content according to ASTM D3174-12, and TS and VS according to APHA (2017) standard methods for water-quality parameters. Electrical conductivity was determined following ASTM D1125-14. The CEC was evaluated using the ammonium acetate saturation method (1 N, pH 7) as described by Sumner and Miller (1996). pH was determined using a representative sample of 1 g, which was suspended in 10 mL of distilled water, establishing a mass/volume ratio of 1:10 (w/v). The mixture was stirred for 30 minutes. Afterwards, it was left to settle for 10 minutes to allow coarse particles to settle and then the pH was measured using a pre-calibrated potentiometer. The moisture content was determined gravimetrically, by evaluating mass loss after drying the sample in an oven at 105 °C for 24 hours, until constant weight was reached. The TS was calculated as the residual weight of the material after drying at 105 °C, representing the total fraction of matter present in the biochar, while VS was estimated as the difference between total solids and ash content. The ash content was determined from the previously dried material, which was subjected to calcination in a muffle furnace at 550 °C for 2 hours, until complete elimination of organic matter. The CEC was determined according to the AS-12 method, using 1 N ammonium acetate as the exchange agent. To do this, 5 g of sieved biochar was weighed, and 25 mL of ammonium acetate solution was added. The mixture was stirred for 30 minutes to ensure contact between the biochar and the exchange agent, and then left to settle for 15 minutes. The system was vacuum filtered and the filtrate collected in a 100 mL volumetric flask, completing the volume with the same solution. Excess ammonium was removed by successive washes with 95% ethanol, distilled water, and a 10% NaCl solution. Then, 10 mL of 40% formaldehyde was added

and titration using 0.1 N NaOH was performed. Similarly, the EC of the biochar was determined to evaluate the presence of soluble salts and their possible influence on the behavior of the material in biological systems. The measurement was performed using the method described in ASTM D1125-14. To do this a 1:5 (w/v) suspension of biochar in distilled water was prepared, agitated for 30 minutes at 25 °C, allowed to settle for 1 hour, and then the conductivity of the supernatant was recorded using a conductivity meter. Organic carbon (OC) and total carbon (TC) of the biochar were also determined.

Conditioning of an anaerobic hybrid reactor (AHR)

A continuous type of AHR was monitored and operated during 65 days at a temperature of 35 °C (Figure 2). The AHR has a useful volume of 2 L and was fed with an organic substrate composed of the liquid fraction of OSW at an organic loading rate of 5 gCOD/L·d. The feeding pH was consistently controlled and maintained within a range of 7-7.5, a condition typically sought to favor hydrolysis and acidogenesis for optimal biogas production (Ali *et al.*, 2021). A control reactor without biochar (blank condition) was included in the experimental setup to serve as a reference for assessing the efficiency of the biochar.

During the operation, the AHR was monitored to evaluate performance parameters, including pH, total chemical oxygen demand (COD_T), soluble chemical oxygen demand (COD_S), total suspended solids (TS_S), and volatile suspended solids (VS_S), both in the influent and effluent. These parameters allowed for estimation of removal efficiency and the initial and subsequent stabilization behavior. The pH was measured with a potentiometer, while COD_T and COD_S were determined by the 5220 D colorimetric method of Standard Methods. TSS and VSS were determined gravimetrically following

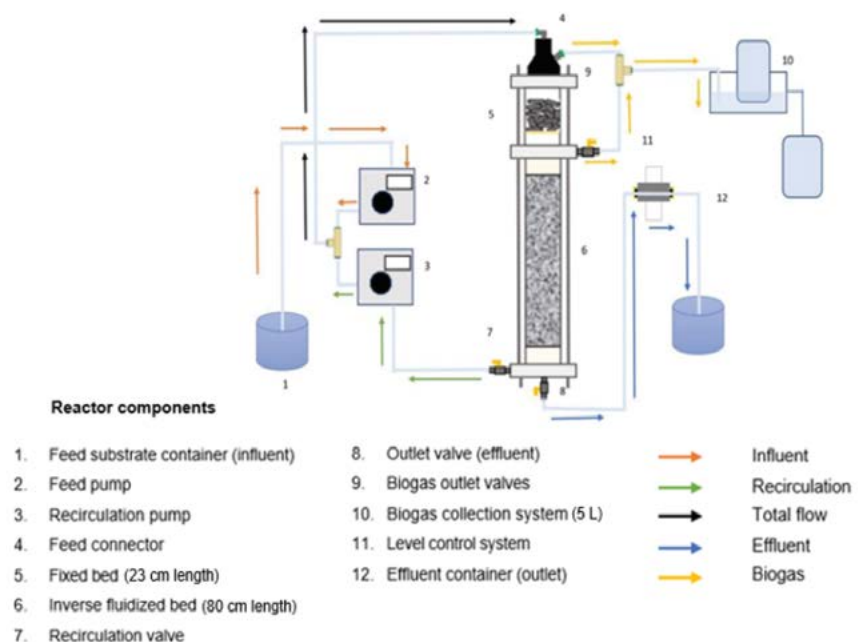


Figure 2. Diagram of the AHR (prepared by the author).

the standard procedure 2540 of APHA (2017). Daily biogas production was monitored using a gas-liquid displacement system.

Statistical analysis

The duplicated measurements of the physicochemical properties of OSW and biochar were statistically compared using a one-way analysis of variance (ANOVA). For the AHR operation and performance dataset, all analytical measurements were conducted in triplicate, except for biogas volume, which was recorded as a single operational value. A one-way ANOVA was applied to evaluate temporal variations during the transition from start-up to stabilization phases for key operational and performance parameters. All statistical analyses were performed using OriginPro 2025b (OriginLab, USA).

RESULTS AND DISCUSSION

Characterization of the OSW and biochar

The main results in duplicate of the physicochemical characterization of the dried OSW and biochar are shown in the Table 1.

Pyrolysis significantly transformed the properties of the OSW. For example, the pH of the biochar notably increased from 4.3 to 8.6, demonstrating its alkaline nature. This change is associated with the release of carbonates, metallic oxides, and basic compounds during pyrolysis, which gives the material the capacity to neutralize volatile fatty acids (Méndez *et al.*, 2023). The pH of the biochar is a valuable property in anaerobic reactors, where it helps maintain the stability of the system against acidification (Amoah *et al.*, 2022). The biochar showed a reduction in moisture content from 7.8% to less than 5%, indicating the near total removal of free and bound water, characteristic of pyrolysis processes at intermediate temperatures (Jiang *et al.*, 2020). The ash content increased up to 21%, evidencing the concentration of inorganic minerals after the thermal degradation of organic components. These minerals, especially Ca^{2+} , K^+ , and Mg^{2+} , can act as catalytic cofactors or pH buffers during biological processes (Jiang *et al.*, 2020). Similarly, the total and volatile solids content greater than 79% demonstrates the high structural stability of biochar, properties

Table 1. Physicochemical properties of the OSW and biochar.

Parameter	Dried OSW	Biochar
pH*	4.3±0.2	8.6±0.2
Moisture (%)*	7.8±0.5	4.2±0.3
Ashes (%)*	4.7±0.4	21.8±1.2
TS (%)*	91.1±0.5	95.3±0.6
VS (%)*	86.5±0.7	79.2±0.9
CEC (meq·100 g ⁻¹)*	21.6±1	42.6±1.5
EC (dS·m ⁻¹)*	0.34±0.04	1.12±0.03
OC (%)**	–	46.2
TC (%)**	–	60

* The analyses were performed in duplicate.

** The analysis performed was simple.

that favor its use as a microbial support in anaerobic systems, allowing the establishment of electroactive consortia and the promotion of DIET. The CEC increased to more than doubled compared to the OSW, reaching $42.6 \text{ meq} \cdot 100 \text{ g}^{-1}$, which indicates the formation of a microporous network with oxygenated functional groups available for adsorption and ion exchange. This behavior suggests a high potential for retaining nutrients (NH_4^+ , K^+ , Ca^{2+}) or adsorbing inhibitors present in anaerobic digestion (Zhang *et al.*, 2020). Likewise, the increase in the EC (1.12) compared to the OSW (0.34) indicates the formation of basic compounds and soluble salts during pyrolysis (Wang *et al.*, 2025). Finally, the TC 60% and OC 46.2% values show a material with a high carbon content and low volatile matter content, indicating a stable and conductive carbon structure (Gao *et al.*, 2023).

Discussion on the potential of biochar as an enhancer in the anaerobic digestion processes

Based on its characterization, the biochar obtained from the OSW possesses various properties that make it a promising material for improving the anaerobic digestion process and other environmental applications. Firstly, its alkaline pH and mineral content act as regulators of medium acidity, helping to maintain optimal conditions for methanogenic communities. In addition, the high CEC and the porosity of the material favor the adsorption of ions and organic molecules, reducing the concentration of inhibitory compounds such as volatile fatty acids or free ammonia (Liu *et al.*, 2022). Furthermore, the conductive carbonaceous structure of the biochar, together with its thermal stability and high proportion of fixed carbon, make it an ideal support for electroactive microorganisms, facilitating biofilm formation and DIET, mechanisms that can increase methane production and the overall efficiency of the process (Chen *et al.*, 2023). Taken together, these results confirm that the biochar produced from the OSW at $550 \text{ }^\circ\text{C}$ is not only a valuable byproduct of the thermal valorization of agro-industrial waste, but also a functional coadjuvant for improving the stability, performance, and sustainability of biological energy conversion systems (Wang *et al.*, 2025).

Performance of the anaerobic hybrid reactor

Monitoring the stability of the AHR revealed notable differences between the start-up and stabilization stages (Table 2). Each value represents the standard deviation of three replicates, except for biogas, for which only a single measurement.

During the 65 days of the operation of the AHR, despite the absence of noticeable changes in the pH values, it showed a positive response. The pH remained within the optimal range of the AD (7.1-7.4) without any sudden drops attributable to the accumulation of volatile fatty acids, which coincides with the values reported by Liu *et al.* (2022). Likewise, an increase in the removal of COD_T and COD_S was observed, reaching values above 90%, which may indicate better utilization of soluble organic matter in the early stages of the process, a behavior also described by Jiang *et al.* (2020). The values of the TSS and VSS showed a notable increase in removal up to 84%, probably associated with increased retention of active biomass on the surface (Zhang *et al.*, 2020). Finally, biogas production increased significantly, reaching 4.6 L d^{-1} under standard temperature

Table 2. Start-up and stabilization of the AHR.

Parameter	Star-up stage			Stabilization stage		
Operational day	1	15	30	45	60	65
pH (inlet)*	7.0±0.3	7.1±0.6	7.1±0.4	6.9±0.2	7.0±0.1	7.1±0.4
pH (internal)*	7.1±0.5	7.3±0.2	7.7±0.2	7.2±0.3	7.3±0.3	7.1±0.2
pH (outlet)*	8.0±0.9	8.1±0.5	7.8±0.8	8.5±0.6	8.2±0.4	8.1±0.2
COD _T (% removal efficiency)*	35.2±3.4	72.4±2.5	84.6±2.9	91.5±2.1	90.3±3.9	93.3±4.2
COD _S (% removal efficiency)*	18.3±1.5	70.1±2.6	81.4±2.0	87.8±1.8	90.1±2.3	91.2±1.9
TSS (% removal efficiency)*	24.8±2.3	38.2±1.4	71.7±2.4	79.9±1.7	82.5±2.1	81.3±1.9
VSS (% removal efficiency)*	43.2±2.2	49.8±1.7	56.3±1.6	67.9±2.5	85.5±2.4	84.7±1.8
Biogas (L)**	0	3.6	4.0	4.3	4.2	4.6
Y _{biogas} (LgCOD _{rem})**	0	0.45	0.47	0.42	0.46	0.49

* The analyses were performed in triplicate.

** The analysis performed was simple.

and pressure conditions. The experimental biogas yield of the AHR reached $0.49 \text{ L g}^{-1} \text{ COD}_{\text{removed}}$, corresponding to approximately 92% of the theoretical biogas potential ($0.54 \text{ L g}^{-1} \text{ COD}_{\text{removed}}$) reported by Siciliano *et al.* (2019). This result indicates a high conversion efficiency of the biodegradable organic matter and a well-stabilized anaerobic process. This high yield indicates an efficient conversion of biodegradable organic matter and a well-stabilized anaerobic digestion process. When Compared the AHR which achieved an experimental biogas yield of $0.49 \text{ L g}^{-1} \text{ COD}_{\text{removed}}$, Guerrero-Martín *et al.* (2024) reported a biogas yield of 0.51 L g^{-1} for a two-stage system treating citrus residues, with a biogas CH₄ content around 54.9%. This comparison underscores the effectiveness of our reactor configuration and biochar amendment in achieving comparable or slightly better yields in a single-stage or hybrid reactor system.

Statistical analysis

The statistical comparison of the physicochemical properties of OSW and biochar revealed significant differences ($p < 0.05$) for all evaluated parameters, including pH, moisture, ash content, TS, VS, CEC, and EC. These results confirm that pyrolysis at $550 \text{ }^\circ\text{C}$ substantially altered the material's physicochemical characteristics. Regarding the AHR operational and performance dataset, the one-way ANOVA indicated statistically significant temporal changes ($p < 0.05$) during the transition from start-up to stabilization for several key parameters. Remarkably, COD_T and COD_S removal efficiencies increased consistently throughout the operational period. TSS and VSS removal efficiencies also exhibited significant improvements over time, reflecting the progressive establishment and activity of the biomass within the AHR. In contrast, pH values showed no statistically significant differences among sampling points ($p > 0.05$), indicating stable buffering conditions throughout the experimental period.

CONCLUSIONS

The valorization of the OSW through pyrolysis allowed the production of biochar with physicochemical properties that support its potential as a functional additive in biological processes, suggesting the effectiveness of pyrolysis in converting agro-industrial waste into a value-added product. These attributes favor the use of biochar as an enhancer in the anaerobic digestion, acting as an adsorbent of inhibitory compounds and a support for microbial communities, promoting DIET. During the operation of the AHR, the biogas production increased significantly, and the reactor showed greater operational stability. Overall, the results demonstrate that the OSW biochar represents a technically and environmentally viable alternative for the comprehensive use of agro-industrial waste. Its application in the AD can strengthen the energy conversion of organic matter and contribute to the sustainability of agro-industrial systems within the framework of a circular economy.

ETHICAL AND SAFETY CONSIDERATIONS

All experimental procedures were conducted in accordance with institutional biosafety and environmental management guidelines.

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