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Influence of biochar mass on solids removal efficiency and biochar transformation during anaerobic digestion of palm oil mill effluent

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ABSTRACT

Palm oil mill effluent (POME) is a high-strength organic wastewater that poses significant environmental challenges, yet holds substantial potential for biogas generation through anaerobic digestion (AD). This study investigated the influence of biochar mass loading (0, 1, 1.5, and 2 g/L) on solids removal efficiency, biochar physicochemical transformation, and biogas production kinetics during POME anaerobic digestion. Solids of total (TS) and solids of suspended of total (TSS) were monitored throughout the digestion period, while biochar characterization was performed using scanning electron microscopy–energy dispersive X-ray spectroscopy (SEM-EDX) and Brunauer–Emmett–Teller (BET) analysis before and after the AD process. Results demonstrated that adding 2 g/L biochar yielded the highest TS and TSS degradation efficiencies of 76.2% and 72.22%, representing improvements of 24.92% and 35.42% over the control, respectively. SEM-EDX analysis revealed significant elemental transformation of biochar during AD, including a decrease in carbon content from 53.8% to 49.8%, a substantial increase in silicon from 1.5% to 5.0%, and the emergence of new elements (Na and P), confirming the active role of biochar beyond that of a passive microbial support medium. BET analysis indicated a reduction in surface area from 9.368 to 4.815 m²/g, consistent with pore occupation by organic compounds and microbial colonization. These findings confirm that biochar mass loading significantly enhances POME treatment performance and provides mechanistic evidence of biochar transformation during anaerobic digestion.

Keywords: palm oil mill effluent, anaerobic digestion, mass loading of biochar, SEM-EDX analysis, BET surface area analysis.

INTRODUCTION

The palm oil industry is among the most financially important agro-industrial industries , with Indonesia and Malaysia together accounting for approximately 85% of the world's crude palm oil (CPO) production (Varkkey et al., 2018). In Indonesia alone, the palm oil industry contributes approximately 3% of the national GDP and provides livelihoods for more than 9.7 million people (Nabila et al., 2023). However, this economic importance is accompanied by a severe environmental burden in the form of palm oil mill effluent (POME), a high-strength wastewater generated during the sterilization, clarification, and separation stages of crude palm oil extraction. Raw POME is characterized by chemical oxygen demand (COD) values of 40,000–100,000 mg/L, biochemical oxygen demand (BOD) of 20,000–60,000 mg/L, total solids (TS) in the range of 27,645–52,355 mg/L, as well as elevated levels of oil, grease, total suspended solids (TSS), and macro-nutrients (Poh & Chong, 2009). If discharged without adequate treatment, POME poses severe threats to aquatic ecosystems through oxygen depletion, eutrophication of receiving water bodies, and uncontrolled greenhouse gas emissions.

Among the available POME treatment technologies, anaerobic digestion (AD) is widely regarded as the most effective approach owing to the high biodegradable organic content of the effluent. Beyond pollutant removal, AD offers the dual advantage of recovering renewable biogas-based energy, which aligns with circular economy principles and cleaner production goals (Poh & Chong, 2009). Various reactor configurations have been investigated for the treatment of POME, including up-flow anaerobic sludge blanket (UASB) reactors, continuous stirred tank reactors (CSTR), and anaerobic baffled reactors, all seeking to optimize organic loading rates, hydraulic retention time, and microbial performance for maximum treatment efficiency and energy recovery (Choong et al., 2018). Despite these advances, the most widely applied treatment method remains the open anaerobic ponding system, which is environmentally unfavorable because a large proportion of the generated greenhouse gases escapes uncaptured into the atmosphere (Sodri & Septriana, 2022). Therefore, closed anaerobic digestion systems represent a critical pathway toward sustainable POME management. However, conventional AD of high-strength wastewaters, such as POME, is frequently limited by microbial inhibition caused by the buildup of ammonia, volatile fatty acids, and sulfur compounds, as well as pH instability and suboptimal conditions for methanogenic archaea, which collectively result in reduced solids removal efficiency and suppressed biogas yields (Chiappero et al., 2020; K. Zhang et al., 2023).

In recent years, biochar has attracted considerable attention as a promising and cost-effective additive to address these limitations and enhance AD performance. Biochar has a lot of carbon. solid material generated by pyrolyzing biomass in low oxygen environments, and its unique physicochemical properties — including groups of functional surface, a well-developed structure of pore, and a large specific surface area, buffering capacity, and electrical conductivity — make it a multifunctional additive in AD systems (Hoang et al., 2022). Its porous structure provides a favorable habitat for microbial colonization and biofilm formation, while its adsorption capacity removes inhibitory compounds such as ammonia and sulfur, thereby stabilizing the digestion environment. Furthermore, biochar has been demonstrated to facilitate direct interspecies electron transfer (DIET) between syntrophic bacteria and methanogenic archaea, a mechanism that accelerates methane production and improves overall process stability (K. Zhang et al., 2023; Zhao et al., 2020). Dose-dependent studies have confirmed these benefits across various substrates; the biochar derived from pine sawdust and corncob ash enhanced biogas yield by 47% and 37%, respectively, while biochar addition to AD, food wastewater raised the cumulative methane yield by up to 46.90% (Sugiarto et al., 2021; Wang et al., 2018).

Several previous studies have investigated the application of biochar in anaerobic digestion of various organic substrates, providing important insights that motivate the present work. Yan et al. (2025) investigated biochar-assisted AD of POME using response surface methodology (RSM), reporting that biochar addition at an optimal dosage of 2 g/L resulted 45.0% more biogas was produced, 73.3% more methane was produced, and 98.2% more solids of volatile (VS) removal compared to POME mono-digestion, with the model of transference function identified as the most accurate kinetic model for predicting methane yield (Yan et al., 2025). Building on this, Chang et al. (2025) applied artificial neural network (ANN) modeling combined with RSM to optimize the biochar-augmented AD of POME, achieving an ANN prediction accuracy of $R^2 = 0.9999$ for cumulative biogas volume and confirming the non-linear relationship between biochar dosage and methane yield (Chang et al., 2025). In the studies using non-POME substrates, Li et al. (2023) demonstrated that biochar addition to AD of piggery wastewater increased the COD removal rate by 37.5–44.0% and enhanced maximum methane yield by up to 89.52% (Li et al., 2023), while Jiang et al., (2023) showed magnetic bio-char addition in sewage sludge AD improved the TS removal efficiency by 28.99–46.13% and reduced the lag phase by 43.78% (Jiang et al., 2023). Similarly, BET as well as SEM-EDS characterization of biochar before and after AD — reported in studies, including Chiappero et al. (2024) who used municipal solid waste substrates — confirmed that reductions in pore volume and specific surface area after AD reflect pore occupation by microbial cells and adsorbed organic compounds, while elemental composition changes reveal the degree of biochar interaction with the process environment (Chiappero et al., 2020).

Despite these advances, critical knowledge gaps remain. First, no study to date has simultaneously examined the effects of biochar mass variation on the TS and TSS removal profiles in POME-specific AD systems, even though total solids and suspended solids are the primary indicators of organic matter degradation efficiency and regulatory compliance in palm oil mill wastewater discharge. Second, while SEM-EDS and BET characterization of biochar transformation have been reported for sludge and municipal solid waste substrates, such physicochemical analysis has not been systematically coupled with POME anaerobic digestion studies across multiple biochar dosages. These gaps limit the mechanistic understanding needed to design and scale up biochar-enhanced AD systems for palm oil mill wastewater treatment.

Therefore, this study presented a novel integrated investigation of the influence of biochar mass (0, 1, 1.5, and 2 g/L) on two interconnected aspects of POME anaerobic digestion: (1) the degradation profiles and removal efficiencies of total solids (TS) and total suspended solids (TSS); and (2) the morphological and elemental transformation of biochar before and after the AD process, as characterized by SEM-EDS and BET analysis. The novelty of this study lies in the integrated evaluation of solids degradation, biochar physicochemical transformation, and biogas production during POME anaerobic digestion, providing mechanistic insight into the role of biochar mass in enhancing AD performance. The findings are expected to provide a comprehensive, evidence-based rationale for biochar application in industrial POME anaerobic digestion, contributing to more sustainable and efficient palm oil industrial wastewater management.

MATERIALS AND METHOD

Materials

Palm oil mill effluent (POME) used as the primary substrate in this study was collected from PTPN III, North Sumatra, Indonesia. The inoculum was obtained from the methanogenic stage sludge of a previously operated anaerobic digester. Biochar was incorporated into the anaerobic digestion system to enhance reactor stability and improve biogas generation efficiency.

The chemical reagents employed during the analyses included hydrochloric acid (HCl, 0.1 N), sulfuric acid (H_2SO_4), distilled water, silver sulfate (Ag_2SO_4), mercury (II) sulfate ($HgSO_4$), potassium dichromate ($K_2Cr_2O_4$), sodium bicarbonate ($NaHCO_3$), ferroin indicator ($C_{36}H_{24}FeN_6O_4S$), and ferrous ammonium sulfate ($Fe(SO_4)_2(NH_4)_2 \cdot 6H_2O$). All reagents were commercially purchased from Merck KGaA® (Darmstadt, Germany).

Experimental configuration

The anaerobic digestion research was carried out at the Laboratory of Ecology, Department of Chemical Engineering, Universitas Sumatera Utara, Indonesia. A stirred batch reactor with a total capacity of 7 L and an effective working volume of 6 L was employed in this study. The reactor was equipped with a paddle-type agitator and connected to a temperature controller to maintain the operating temperature at 45°C throughout the digestion process.

A sampling port was installed to facilitate periodic sample collection, while biogas production was monitored using a gas collection system connected to the reactor. Thermal stability was maintained using a water bath system measuring 2 m × 0.6 m × 0.6 m equipped with an ATN water heater. Biochar was added into the reactor at concentrations of 0, 1, 1.5, and 2 g/L.

Raw material preparation

Approximately 150 L of fresh POME collected from PTPN III was temporarily stored at −4°C prior to use to suppress microbial activity and preserve substrate characteristics before the anaerobic digestion process. Before experimentation, the raw material was described in order to ascertain its initial properties. Analytical parameters and methods used for raw material characterization are summarized in Table 1.

Biochar preparation

The biochar utilized in this study was produced from oil palm empty fruit bunch (EFB) bio-pellets through a pyrolysis process. Initially,

Table 1. Raw material analysis parameters as well as methods

Parameters	Unit	Methods
Total solid	mg/L	APHA 2540B
Total suspended solid	mg/L	APHA 2540D

the EFB material was crushed and milled into powder using a ball mill until reaching a particle size of approximately 100–150 µm (100 mesh). The powdered material was subsequently oven drying 110°C prior to pelletization.

The prepared pellets were pyrolyzed using a microwave-assisted reactor under oxygen-limited conditions. Nitrogen gas was continuously supplied at a flow rate of 0.5 L/min to maintain an inert atmosphere inside the reactor. Pyrolysis was conducted using a Samsung microwave system operated at 450 W for 40 min with a feed mass of 100 g. After completion of the process, the produced biochar was cooled and weighed after 30 minutes in a desiccator using a digital balance. The schematic diagram of biochar preparation is shown in Figure 1. Based on previous characterization following SNI 06-4369-1996, the biochar exhibited a pH of 7.8, a moisture content of 3.43%, volatile matter of 20.81%, an ash content of 7.41%, and fixed carbon of 68.35% (Sidabutar et al., 2024).

Biogas production

Anaerobic digestion was performed using a paddle-stirred batch reactor system for biogas production. Initially, POME and inoculum were mixed at a volumetric ratio of 80:20 prior to being introduced into the reactor. The digestion process was operated at 45°C using a 7 L reactor with a 6 L working volume.

Biochar was supplemented into the reactor at different concentrations of 0, 1, 1.5, and 2 g/L substrate. During the digestion period, total solids (TS) and total suspended solids (TSS) were monitored daily using the samples collected from the reactor sampling port. The generated biogas was collected and quantified through a gas collection balloon connected directly to the reactor system.

RESULTS AND DISCUSSION

The impact of biochar mass on the total solids profile

TS is the amount of solids in organic matter during the process in the digester. TS indicates the level of destruction or decay of organic matter, which indicates decomposition (Warisaura & Kurniawati, 2024). The impact of biochar mass on the total solids profile is shown in Figure 2. It 2 illustrates the gradual decrease in TS during the anaerobic fermentation process. Prior to fermentation, the TS concentration ranges between 36,000 and 42,000 mg/L. After fermentation, this value decreases significantly to a range of 10,000–14,000 mg/L. The highest TS degradation efficiency is observed with the addition of 2 g/L biochar, achieving a reduction of 76.2%, compared to only 61% in the absence of biochar. This represents an increase in degradation efficiency of 24.92% when 2 g/L biochar is added.

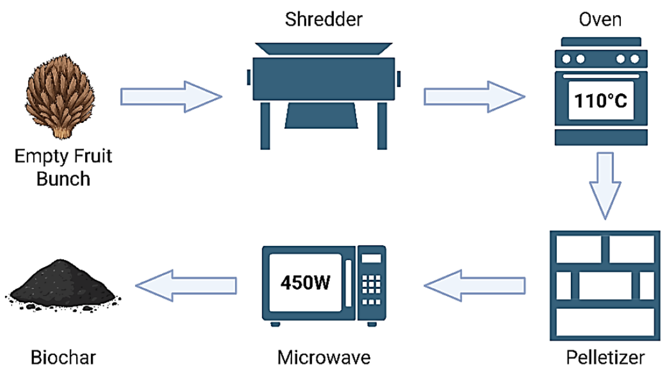


Figure 1. Schematic diagram of biochar preparation

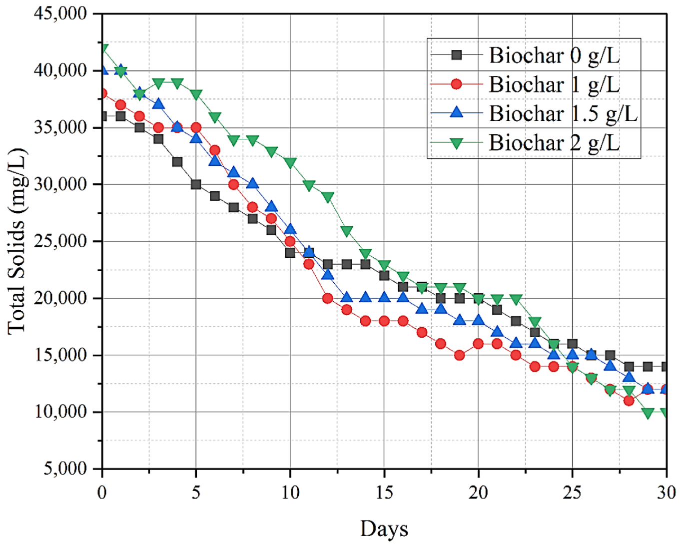


Figure 2. The impact of biochar mass on the total solids profile

The reduction in TS is primarily attributed to the metabolic activity of microorganisms, which effectively break down organic matter into simpler compounds and convert them into methane gas as part of the anaerobic digestion process (Chen et al., 2023). The presence of biochar enhances this process by providing a favorable environment for microbial activity, thereby improving the overall efficiency of organic matter degradation.

The impact of biochar mass on the profile of total suspended solids

TSS refers to all solid substances or particles suspended in water (Nasution & Andriani, 2023). Figure 3 illustrates the reduction in TSS over time as it is converted into biogas during anaerobic fermentation. Initially, TSS ranges between 30,000–36,000 mg/L and decreases to 10,000–14,000 mg/L after fermentation. The highest degradation efficiency, reaching 72.22%, is achieved with the addition of 2 g/L biochar, mean 35.42% rise in comparison to the condition without biochar. This improvement highlights the role of biochar in enhancing microbial activity and facilitating the breakdown of suspended organic matter into biogas. The reduction in TSS indicates that the solids in the waste can be naturally filtered and settled (Direstiyani, 2016). The greater the degradation, the lower the TSS and the more biogas is produced by the waste-degrading bacteria (Liliasari, 2017). The

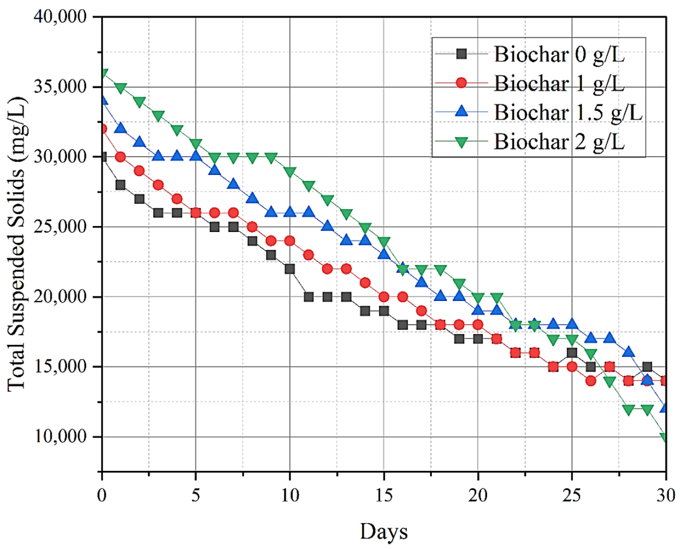


Figure 3. Effect of biochar mass on TSS profile

impact of biochar mass on the profile of total suspended solids is shown in the Figure 3.

Morphological characteristics and elemental composition

The morphology and elemental composition of the biochar were analyzed using Scanning Electron Microscope-Energy Dispersive X-Ray Spectroscopy (SEM-EDS) with a SEM JEOL JCM-7000 device, Germany. The analysis was conducted at the SEM Laboratory, Department of Physics, Faculty of Mathematics and Natural Sciences, Syiah Kuala University, Banda Aceh, Indonesia. Table 2 presents the morphology of the biochar before and after treatment in the anaerobic digestion (AD) process at 1,000x, 5,000x, and 10,000x magnifications. The rough and porous surface of the biochar, formed due to the loss of volatiles during pyrolysis, allows microorganisms to adhere and proliferate effectively (Zheng et al., 2022). This structure supports bacterial colonization and the adsorption of toxic compounds such as ammonia and sulfur, which can inhibit microbial activity, thereby enhancing the efficiency of anaerobic digestion (AD). Following the AD process, the biochar undergoes changes in surface structure, becoming more irregular, with pores clogged by residues resulting from the absorption of organic compounds or microbial activity.

Table 3 compares the elemental composition of biochar before and after the AD process treatment. Before treatment, carbon (C) was the dominant element at 53.8%, followed by oxygen

Table 2. SEM images of biochar before and after anaerobic digestion

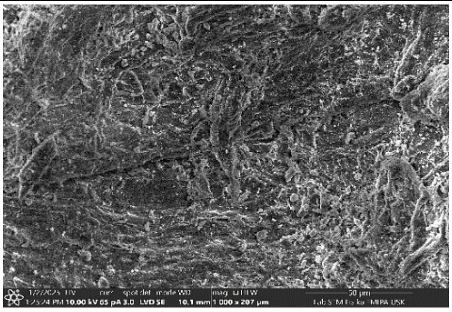
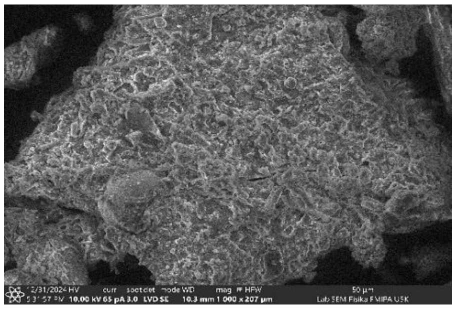
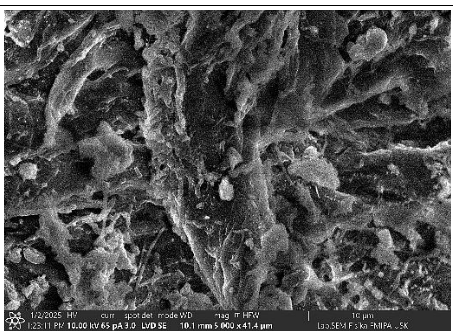
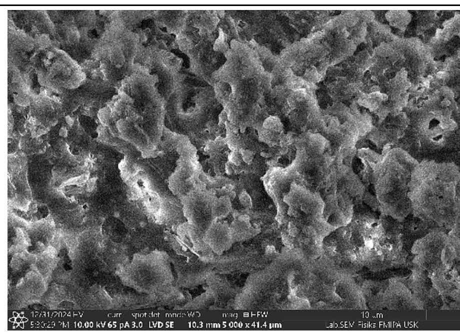
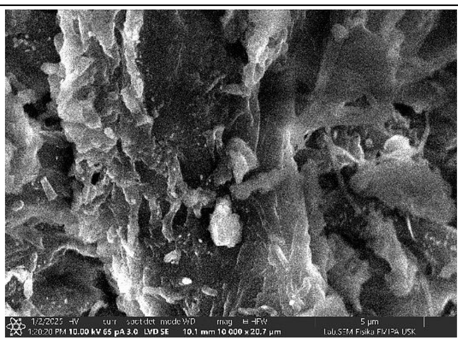
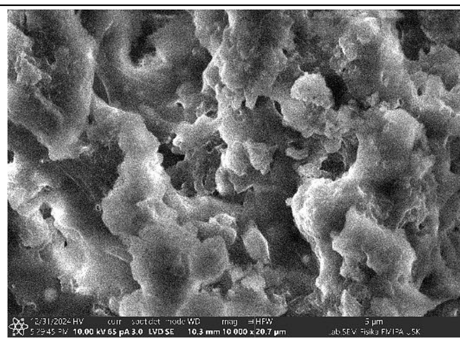
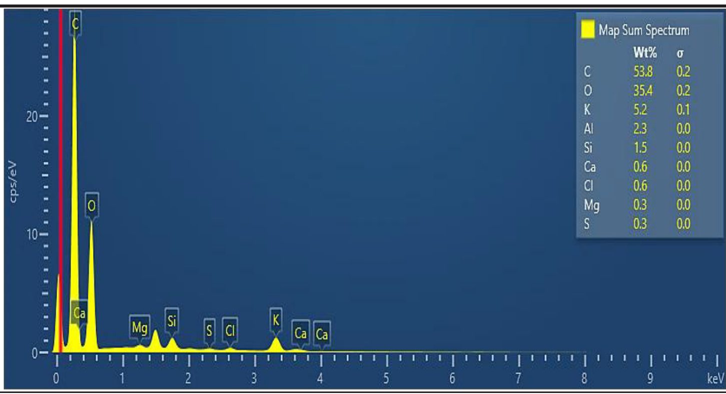
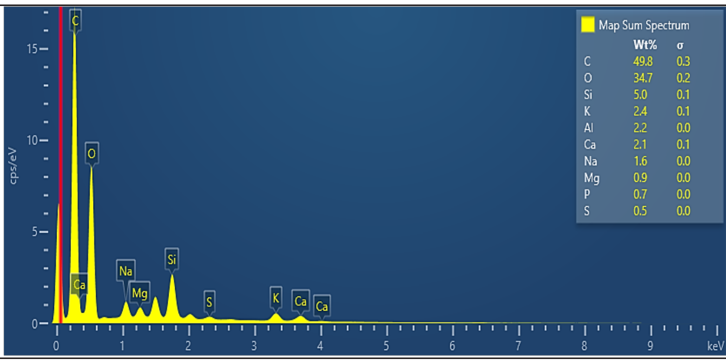
Biochar before AD process treatment	Biochar after AD process treatment
	
magnification 1,000x	
	
Magnification 5,000x	
	
magnification 10,000x	

Table 3. Comparison of the elemental composition of biochar before and after treatment

Sample	Composition (%)
	C = 53.8 O = 35.4 K = 5.2 Al = 2.3 Si = 1.5 Ca = 0.6 Cl = 0.6 Mg = 0.3 S = 0.3
a. Biochar before AD process treatment	
	C = 49.8 O = 34.7 Si = 5.0 K = 2.4 Al = 2.2 Ca = 2.1 Na = 1.6 Mg = 0.9 P = 0.7 S = 0.5
b. Biochar after AD process treatment	

(O) at 35.4%, and potassium (K) at 5.2%. After the AD process, the carbon content decreased to 49.8%, while oxygen remained relatively stable at 34.7%. Silicon (Si) increased from 1.5% to 5.0%, whereas potassium (K) decreased to 2.4%. Additionally, new elements such as sodium (Na) at 1.6% and phosphorus (P) at 0.7% were detected after treatment, which were not present before. These changes indicate that the AD process alters the chemical composition of biochar (Zhou et al., 2020), likely due to the degradation of organic material or interactions with the process environment. Furthermore, the results suggest that biochar can adsorb and bind certain compounds during anaerobic digestion.

Textural characteristics

The texture characteristics of biochar were analyzed using the Brunauer-Emmett-Teller (BET) instrument with a Quanthacrome Nova 2000. The analysis was conducted at the Instrumental Analysis Laboratory, Gadjah Mada University, Yogyakarta, Indonesia. The analysis included measuring the specific surface area of the biochar based on the volume of nitrogen gas (N_2) adsorbed in its pores at a temperature of 77 K (Tsubota et al., 2023). BET analysis is a method for determining the specific surface area of solid materials, which is crucial for gas adsorption performance (Sidabutar et al., 2025).

Table 4 shows the changes in the characteristics of biochar before and after its use as an additive in biogas production. During the anaerobic digestion process, biochar experiences decreased pore capacity and surface area as a result of the adsorption of organic compounds. Initially, the biochar had a surface area of 9.368 m^2/g, a volume of pore of 0.02562 cc/g, and a radius of pore of 2.6833 nm (d = 5.3666 nm). After the process, the area of surface decreased to 4.815 m^2/g, the volume of pore reduced 0.02043 cc/g, and the radius of pore slightly decreased to 2.6542 nm (d = 5.3084 nm). This indicates that biochar not only serves as a habitat for microbes but also adsorbs compounds that influence biogas production. The use of biochar results in a more uniform and concentrated distribution of pore sizes, particularly in medium-sized pores. As the biochar content increases, the radius of micropores gradually becomes larger (Chen et al., 2023).

Table 4. Textural characteristics of biochar before and after the AD process treatment

Sample	Surface area (m^2/g)	Pore volume (cc/g)	Pore radius (nm)
Biochar before treatment	9.368	0.02562	2.6833
Biochar after treatment	4.815	0.02043	2.6542

Figure 4 shows an increase in the volume of N_2 adsorbed on the biochar surface, both before and after treatment, as the relative pressure (P/Po) increases up to 1. After that, the volume of adsorbed N_2 decreases, indicating the occurrence of N_2 desorption from the biochar surface, both before and after treatment. This graph represents the nitrogen adsorption/desorption isotherm used to analyze the surface area and porosity of the material. The difference between the adsorption and desorption curves is called the hysteresis loop (Viisanen et al., 2024), which provides information about the pore structure. The loop deviates and reconnects at a relative pressure (P/Po) of around 1.0 (J. Zhang et al., 2025), thus classifying it as a H3 of the Type of loop of hysteresis. This indicates that adsorption continues to increase until the pressure approaches saturation, suggesting that the material has macropores without internal pores. The initial slope of the adsorption curve reflects the material's surface area, where a steeper slope indicates a larger surface area. Additionally, the shape of the curve and the hysteresis loop help ascertain the distribution and size of the material's pores.

CONCLUSIONS

The present study demonstrated that biochar addition significantly enhanced the anaerobic digestion performance of palm oil mill effluent (POME), particularly in terms of solids degradation and biogas production behavior. Among the investigated conditions, the addition of 2 g/L biochar exhibited the best performance, achieving TS and TSS degradation efficiencies of 76.2% and 72.22%, respectively, which were substantially greater than the system of control without biochar. These findings confirm the important role of biochar in promoting microbial activity and improving the conversion of organic matter during anaerobic digestion. Characterization analyses further revealed that biochar underwent considerable physicochemical transformation throughout the digestion process. SEM-EDX analysis showed changes in surface morphology and elemental composition, including the reduction of carbon content and the enrichment of silicon, sodium, and phosphorus after treatment, indicating that biochar actively participated in adsorption and interaction processes within the reactor system. BET analysis also demonstrated a decrease

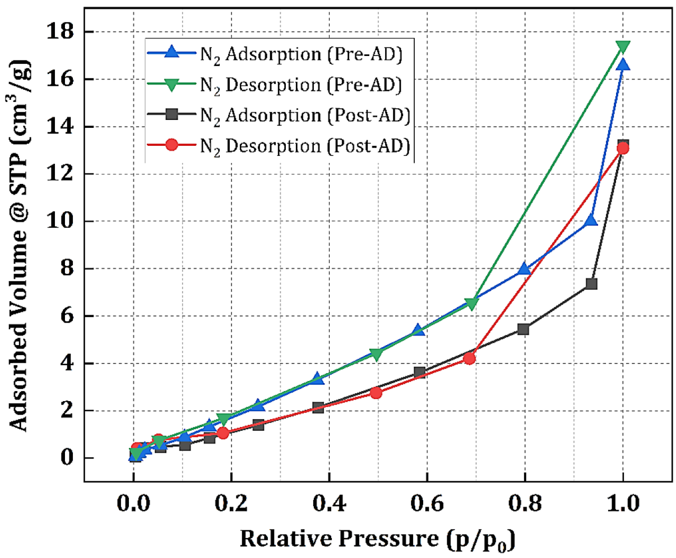


Figure 4. Adsorption-desorption isotherm of biochar after treatment

in surface area and pore volume after anaerobic digestion, suggesting pore occupation by microbial colonies and adsorbed organic compounds. These results provide mechanistic evidence that biochar functions not only as a microbial support medium but also as an active adsorbent influencing digestion performance. Overall, this study highlights the strong potential of biochar as an effective additive for improving POME treatment efficiency and biogas generation, while also providing important insights into the transformation behavior of biochar during anaerobic digestion processes. Future research is recommended to investigate the long-term stability and reusability of biochar, as well as to evaluate the performance of different biochar feedstocks and reactor configurations under continuous anaerobic digestion systems for industrial-scale applications.

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