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Characterization and proximate analysis of composite biochar briquette from oil palm frond and red clay

Saipin Suwankamnoed[1], Pattareewan Imsuwan[2], Worrathon Tilokkarn[3], Bunyarit Meksiriporn[4], Thanawat Sutjaritvorakul[1,2*]

[1] Department of Petrochemicals and Environmental Management, Faculty of Engineering, Pathumwan Institute of Technology, Bangkok, Thailand
[2] Faculty of Science and Technology, Pathumwan Institute of Technology, Bangkok, Thailand
[3] Department of Industrial Engineering, Faculty of Engineering, Pathumwan Institute of Technology, Bangkok, Thailand
[4] Department of Biology, School of Science, King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand
* Corresponding author's e-mail: thana5306@hotmail.com

ABSTRACT

The ultimate goal of this research is to create effective and environmentally friendly biochar briquettes from oil palm frond (OPF) waste. Red clay, a plentiful local resource, has been demonstrated to improve the properties of fuel briquettes. In this study, the tested composite briquettes (biochar:red clay:starch by weight) were prepared in the following ratios: 40:55:5 (T2), 50:45:5 (T3), 60:35:5 (T4), and 70:25:5 (T5), while the control sample (T1) consisted of 95:5 (biochar:starch). All of the different ratios were analyzed using proximate analysis. The results showed moisture content ranging from 7.35% to 8.47%, ash content from 29.75% to 49.82%, volatile matter from 15.63% to 17.47%, and fixed carbon from 25.31% to 46.48%. Among them, T4 exhibited the lowest moisture and ash content and the highest calorific value at 5,650 cal/g, making it the optimal candidate for further investigation. A comprehensive analysis of T4 was conducted using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and X-ray diffraction (XRD). The techniques revealed the morphological structure and inorganic components that contribute to the energy efficiency of T4. The study also examined air pollutant emissions. T4 produced lower emissions of pollutants such as carbon monoxide (CO), carbon dioxide (CO_2), total volatile organic compounds (TVOCs), formaldehyde (HCHO), and particulate matter ($PM_{1.0}$ and $PM_{2.5}$) compared to the OPF biochar briquette without red clay (T1). Composite biochar briquettes made from OPF and red clay (T4) are safe and suitable for use as a fuel source for household applications like cooking and heating. This research contributes to the development of the green bioeconomy and supports sustainable energy initiatives.

Keywords: agricultural waste, renewable energy, calorific value, carbon emission, air pollution

INTRODUCTION

These days, energy consumption is increasing due to economic development, industrialization, and population growth (Nikiforov et al., 2024). Historically, the use of fossil fuels has caused significant environmental impacts, including air pollution and carbon dioxide emissions – major contributors to global warming. Additionally, fossil fuels are non-renewable natural resources (Wang and Azam, 2024). In agriculture-based countries, carbonized biomass briquettes are considered to be alternative renewable energy sources because these countries can produce biochar from agricultural wastes, including fruit shells, rice husks, and sugar cane bagasse. The agricultural residue biochar was generated through the pyrolysis process and was compressed with binder to form briquettes (Lubwama et al., 2024). These biochar briquettes are widely used for domestic cooking and heating in developing countries (He et al., 2024). Although charcoal briquettes from biomass are an alternative energy source with less environmental impact, there are many research studies on how to increase the efficiency of charcoal and make it more environmentally friendly (Celestino et al., 2023; Tazebew et al., 2023; He et al., 2024). The binders are known to be the important composition of biomass briquettes. The briquette needs binder to hold the structure together because the biochar is totally lacking in plasticity, and they also have effects on strength and heating potential (Celestino et al., 2023). Nakhon Si Thammarat is a large province in southern Thailand where oil palm is cultivated commercially. Where oil palm is grown for commercial purposes. After oil palm harvesting and processing, oil palm wastes are generated in large amount such as palm empty fruit bunch, palm frond, palm truck, palm kernel shell and palm pressed fiber (Awoh et al., 2023). Previous research showed these oil palm wastes can be used to fabricate high potential biomass briquette. Agustiar et al. (2023) studied the possibility of replacing fossil fuels with renewable energy. They stated that the average calorific value of the oil palm empty fruit bunch, fiber, and shell mixture used to make bio-briquettes was 5000 cal/g. According to Maulina et al. (2019), the oil palm frond charcoal briquette also showed a high calorific value, and they observed that the calorific value rises when the quality of the binder material is improved.

In addition to being a major source of oil palm waste, southern Thailand is also rich in red clay. This clay, classified under the Krabi soil series, is moderately distributed across the Thai peninsula and southeastern coastal areas. It has traditionally been used for cultivating crops like oil palm, para rubber, and tropical fruits (Kheoruenromne et al., 2000), as well as in pottery and brick-making due to its high hardness, low expansion coefficient, strong structure, and chemical stability. Because of these properties, red clay is expected to perform well as a binder in the production of smokeless biochar briquettes (Mopoung and Udeye, 2017; Nawaukkaratharnant et al., 2022; Keawdee, 2023). Prior studies have shown that the addition of clay can improve the bulk density and compressive strength of coal briquettes. For instance, briquettes with 15% clay content were found to have high calorific values (Gebresas et al., 2015; Celestino et al., 2023). Furthermore, red clay is abundant and available at little to no cost in this region. Although clay does not contribute to the heating value of the briquette (Pei Chen and Ani, 2017), it may help reduce air pollutant emissions when incorporated into the fuel. Glalah et al. (2024) found that using clay as a binder in stool wood sawdust and cacao pod biochar briquettes reduced carbon monoxide emissions. Nevertheless, the proportion of clay utilized affects the characteristics of biochar briquette. Moreover, no prior research has been done on the utilization of red clay in Nakhon Si Thammarat province as a raw material for biochar briquettes. It's evident that utilizing red clay as a composite is a fascinating idea. Thus, the main objective of this research is focused on the characterization, proximate analysis, and assessment of the air pollution emission of composite biochar briquettes made from oil palm fronds (OPF) and red clay using tapioca starch as a binder. The findings of this study can offer empirical support for the production of biochar briquettes to address household and local energy requirements for environmental preservation and energy sustainability.

MATERIALS AND METHOD

Preparation of biochar briquettes and red clay

The OPF were gathered from nearby plantations in Nakhon Si Thammarat province and sun-dried for two days. A 200-liter kiln was filled with the dried biomass for the pyrolysis process. The procedure was conducted under anaerobic conditions at temperatures ranging from 450 to 500 °C for five hours, with a temperature increase of 10 °C per minute (Sanvong and Nathewet, 2014; Wijitkosum, 2022). Red clay was sourced from the Lum Thap district in Krabi province, Thailand. The oil palm frond biochar was crushed into small pieces and thoroughly mixed with the red clay. Tapioca starch was used as a binder in this study, and each treatment involved a different ratio of the composite briquettes (biochar : red clay : starch) (% w/w), including 40:55:5 (T2), 50:45:5 (T3), 60:35:5 (T4), and 70:25:5 (T5). The control group consisted of 95:5 (biochar : starch) (T1). The briquetting process was carried out at a pressure of 0.5 MPa using a screw briquetting machine (Saputro et al., 2021; Suwanhumnerd et al., 2025).

Proximate analysis of biochar briquettes

The proximate analysis of the biochar briquettes was conducted to determine moisture content, ash content, volatile matter, calorific value, and fixed carbon. These analyses were performed according to standards from the American Society for Testing and Materials (ASTM), including ASTM D3173, ASTM D3174, ASTM D3175, ASTM D5865, and ASTM D3172, respectively (Suryaningsih et al., 2019; Abineno et al., 2025).

Characterization of selected biochar briquettes

The surface morphology and structural characteristics of the selected biochar briquette were examined using a scanning electron microscope equipped with energy-dispersive X-ray spectrometry (SEM-EDS, JEOL JSM-IT300, Oxford X-Max 20). Carbon tape was used to mount the briquette samples, which were then immediately scanned using the SEM. Observations were performed at an acceleration voltage of 10 kV. For elemental analysis, the composition was identified and quantified using EDS, and the elemental distribution on the biochar briquette sample was also examined. Additionally, X-ray powder diffraction (XRD) analysis was conducted to determine the chemical constituents. Biochar briquette powder samples were scanned at a 2θ range of 5° to 80°, using a voltage of 40 kV and a current of 30 mA (Adeleke et al., 2021; Shah et al., 2023; Waheed et al., 2023). The bulk density of the selected biochar briquette and the control was measured and calculated according to the method described in Sanka et al. (2024).

Evaluation of air pollutants emission

To evaluate air pollution emissions, 500 g each of T1 and T4 were tested using a locally available cooking stove under typical cooking conditions. A portable air quality detector was used to measure emissions of carbon dioxide (CO_2), formaldehyde (HCHO), total volatile organic compounds (TVOCs), and particulate matter ($PM_{1.0}$ and $PM_{2.5}$). Combustion time was also recorded (Senila et al., 2022; Sanka et al., 2024).

Statistical analysis

All experiments were carried out in triplicate. GraphPad Prism software version 10.0 (GraphPad Software, San Diego, CA, USA) was used to perform a one-way analysis of variance (ANOVA) on the collected data. Tukey's test was used to analyze significant differences among treatment means at $P \leq 0.05$.

RESULTS AND DISCCUSSION

Proximate analysis was performed to evaluate the energy potential of composite bio-briquettes made from oil palm frond biochar and red clay at different ratios (biochar : red clay : starch), as illustrated in Figure 1. The moisture content of the briquettes ranged from 7.45% to 8.47%. The highest moisture content was found in T5 (70:25:5) at 8.47%, while the lowest was observed in T2 (40:55:5), at 7.40%. Ash content represents the incombustible residue remaining after the combustion of a bio-briquette. T2 exhibited the highest ash content at 49.82%, whereas T4 (60:35:5) showed the lowest at 32.20%. The proportion of red clay to biochar influenced the volatile matter content. A lower red clay content corresponded with a lower percentage of volatile matter. The highest volatile matter content was recorded in T2 (17.47%), and the lowest in T5 (15.63%), both of which were not significantly different from the control (T1). For fixed carbon, the highest percentage was found in T1 at 46.48%, followed closely by T4 at 44.6%. However, when considering calorific value, T4 demonstrated the highest energy content at 5.650 cal/g. Therefore, T4 was chosen for further research, as its combination of high heating value and low ash content indicates strong performance for hybrid biochar briquettes.

The microstructure of the selected composite OPF biochar briquettes (T4) was investigated using a scanning electron microscope (SEM). The surface morphology of T4 is shown in Figure 2A. The SEM micrograph reveals a rough and uneven surface, with clay particles clearly visible within the briquette matrix. These particles are distributed and bonded to the surface of the briquette. The addition of clay also blocks and reduces the porous structure of the OPF biochar embedded in the briquette (Figure 2B), which may affect its combustion potential. Energy-dispersive X-ray spectroscopy (EDS) was used to analyze the elemental composition of the composite biochar briquette, and the results are shown in Figure 3. The two major elements present are carbon (C) and oxygen (O), comprising 44.06% and 43.19% of the total, respectively. Minor elements detected include aluminum (Al, 4.74%), silicon (Si, 3.74%), and iron (Fe, 1.09%). The spatial distribution of these elements was further examined using SEM-EDS mapping, with results displayed in Figure 4. Only spectral images of the most significant and abundant elements are shown. In

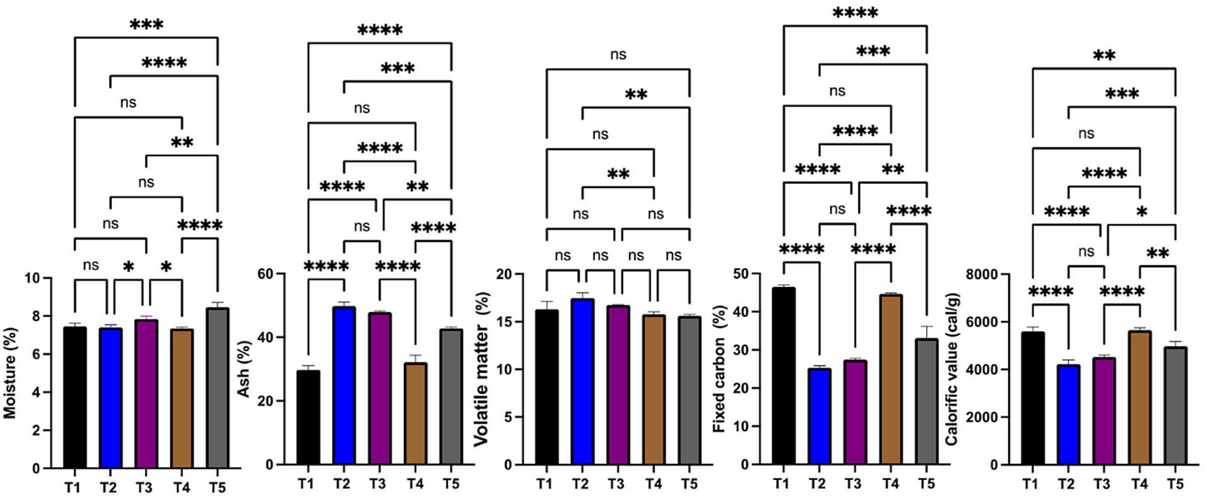


Figure 1. Proximate analysis and calorific value of composite briquettes from OPF biochar and red clay, T1: 95:5 (biochar: starch), T2: 40:55:5 (biochar: red clay: starch), T3: 50:45:5 (biochar: red clay: starch), T4: 60:35:5 (biochar: red clay: starch), and T5: 70:25:5 (biochar: red clay: starch), *, ***, and **** show the significance, and "ns" indicates not significant at $P \leq 0.05$

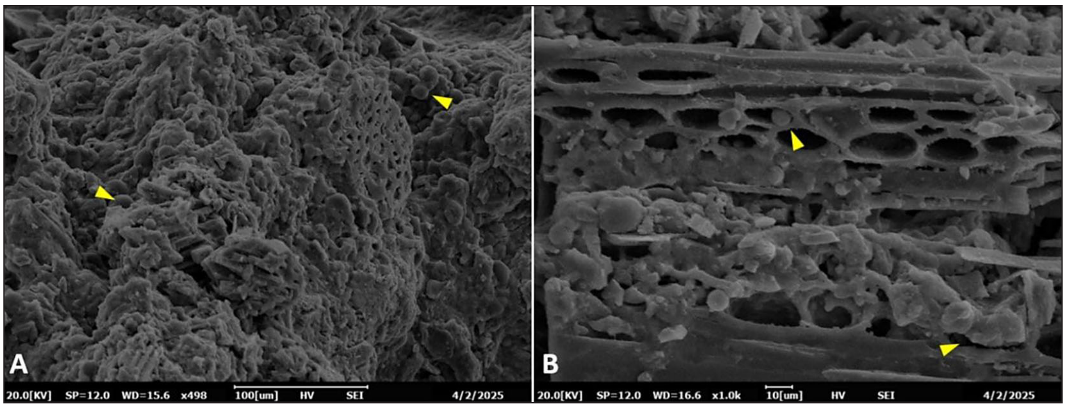
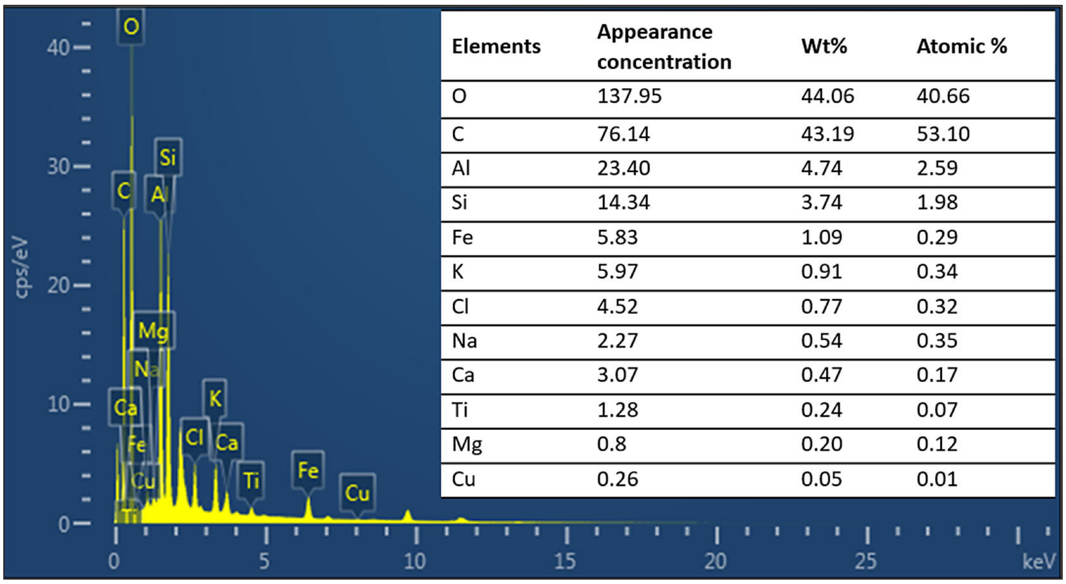


Figure 2. SEM micrograph of surface structure of T4, the clay particles (arrows) distributed on surface structure of the briquette at 498 magnifications, scale bar: 100 µm (A). The clay particles block the porous structure of OPF biochar at 1000 magnifications, scale bar: 10 µm (B)

the elemental maps, carbon (C) is represented in green, aluminum (Al) in red, iron (Fe) in blue, and silicon (Si) in purple. The chemical composition was also investigated by X-ray diffraction (XRD), and the XRD pattern is shown in Figure 5. Broad peaks commonly observed in biochar samples were detected at around 20° and 30°, indicating the presence of amorphous or disordered carbon structures. The pattern also revealed peaks corresponding to quartz or silicon dioxide (SiO_2). Diffraction peaks observed at approximately 33°, 35°, 37°, and 41° may be attributed to magnetite (Fe_3O_4) or hematite (Fe_2O_3). Additionally, small continuous peaks in the 65°–70° range suggest the presence of multiple crystalline phases, such as alumina (Al_2O_3), anorthite, or gehlenite – minerals typically found in clay. The presence of Si, Fe, and Al was clearly confirmed by SEM-EDS analysis (Figures 3 and 4). Bulk density, a crucial physical characteristic of briquetting, was 1.43 g/cm³ for T4 and 1.12 g/cm³ for T1.

The determination of air pollutant emissions from the combustion of the selected composite biochar briquettes (T4), compared to the control (T1), is presented in Table 1. The results show that all air pollutants emitted by T4 were significantly lower than those from the control, except for formaldehyde (HCHO), which did not differ significantly. The carbon monoxide (CO) emission from T4 was 1.33 ppm, compared to 2.33 ppm from T1. For carbon dioxide (CO_2), a significant difference was observed between T1 (2,444 ppm)



Elements	Appearance concentration	Wt%	Atomic %
O	137.95	44.06	40.66
C	76.14	43.19	53.10
Al	23.40	4.74	2.59
Si	14.34	3.74	1.98
Fe	5.83	1.09	0.29
K	5.97	0.91	0.34
Cl	4.52	0.77	0.32
Na	2.27	0.54	0.35
Ca	3.07	0.47	0.17
Ti	1.28	0.24	0.07
Mg	0.8	0.20	0.12
Cu	0.26	0.05	0.01

Figure 3. EDS spectrum of composite briquette from OPF biochar and red clay (T4)

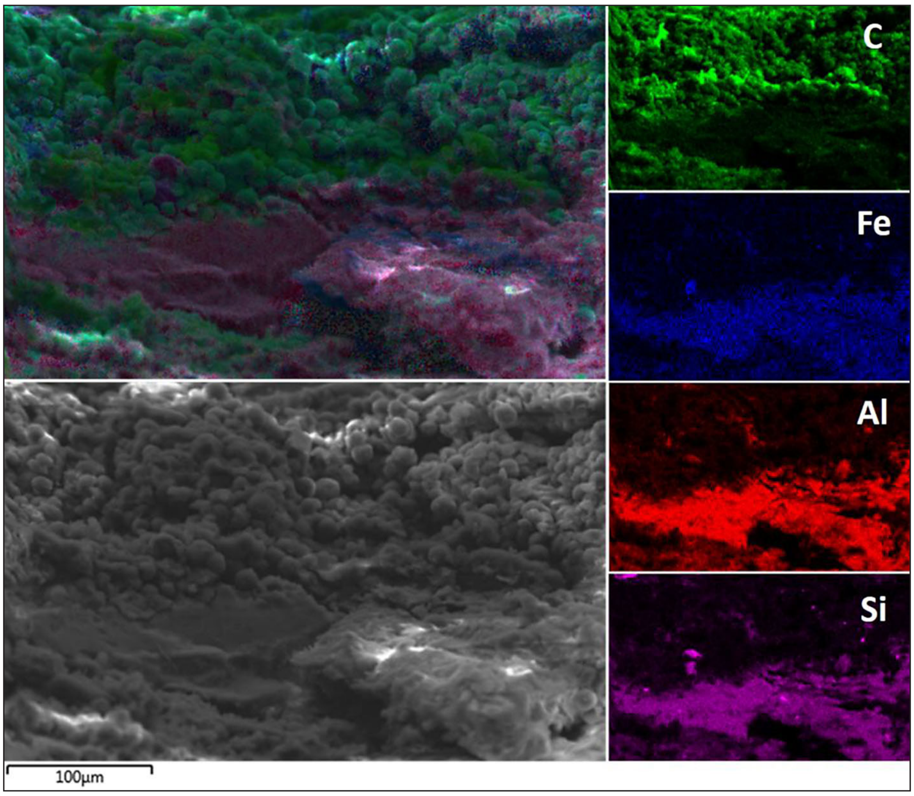


Figure 4. EDS elemental mapping (C, Fe, Al, and Si) of composite briquette from OPF biochar and red clay surface (T4), scale bar: 100 µm

and T4 (556.30 ppm). In addition, T1 exhibited higher levels of total volatile organic compounds (TVOCs), particulate matter 2.5 ($PM_{2.5}$), and particulate matter 1.0 ($PM_{1.0}$), while T4 showed lower levels of these pollutants 0.004 mg/m³, 30 µg/m³, and 38.33 µg/m³, respectively. In the combustion time test of 500 g biochar briquettes, T4 demonstrated a longer burning duration than T1, at 80 and 54 minutes, respectively. After more than 12 months of storage in plastic bags at room temperature (30 °C), both T4 and T1 biochar remained in normal condition.

The moisture content of the composite OPF biochar briquettes varied between 7.48% and

8.47%, depending on the mixing ratios. According to Celestino et al. (2023), briquettes should have a moisture level between 5% and 20% for optimal performance. In this study, the moisture levels were within the acceptable range, indicating no significant energy loss due to water evaporation during combustion. Additionally, biochar with moisture content exceeding 10% tends to become brittle when heated (Carnaje et al., 2018). According to the XRD profile (Figure 5), the presence of silicon dioxide (SiO_2) caused the ash content to rise as the fraction of clay increased. In addition to other negative impacts such reactor incrustation and slag formation, a high ash content generally has a negative impact on energy-yielding qualities (Celestino et al., 2023; Shah et al.,2023). Volatile matter is the term used to describe the parts of the biochar that are released in a gaseous phase when the biochar briquette burns. Volatile matter is an important determinant of the properties of the briquette since it affects its heating value, combustion behavior, and environmental impact. For this study, the composite OPF biochar briquettes showed the same tendency to have less volatile matter as clay levels decreased. The lower volatile matter from briquettes with clay may be explained by the fact that clay is an inorganic substance, meaning that the contribution of clay to the volatile matter concentration is negligible (Onchieku et al., 2012; Carnaje et al., 2018). Moreover, when the briquette has a high volatile matter concentration, it is more likely to ignite during combustion with a high proportional flame. On the other hand, high volatile matter briquettes burn quickly and emit a lot of smoke as they burn (Ajimotokan et al., 2019; Glalah et al., 2024). The most significant factor that directly affects the energy potential is fixed carbon (Akam et al., 2024). T4 exhibited the highest content of fixed carbon when compared with other composite OPF biochar briquettes with clay. Demirbas (2003) clarified that there is a highly substantial correlation between the percentage of fixed carbon in biomass and the amount of lignocellulosic material, where oil palm was found to contain high levels of cellulose, hemicellulose and lignin (Mohtar et al., 2015). A fuel's energy content is commonly measured by its calorific value, also

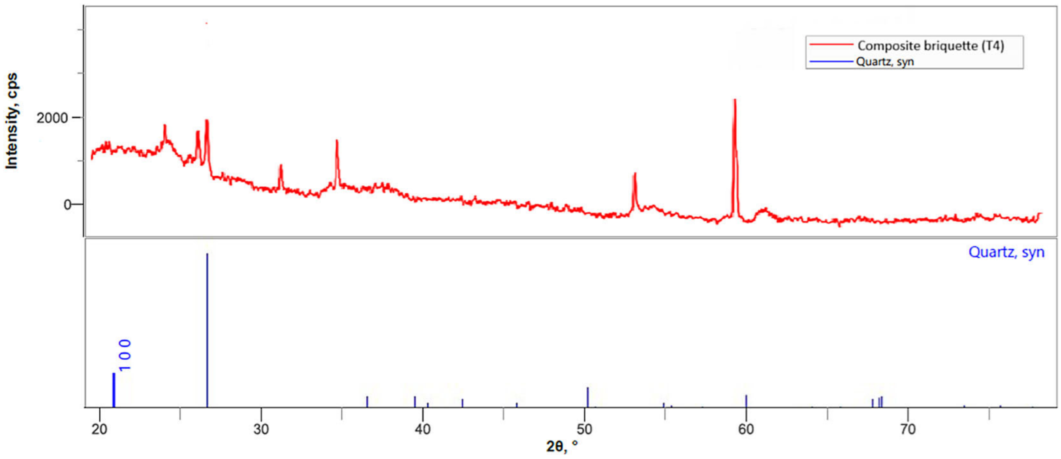


Figure 5. XRD diffracttogram of composite briquette from OPF biochar and red clay (T4)

Table 1. The air pollution emissions detected from selected composite briquette from OPF biochar and red clay (T4) and control (T1)

Air pollutants emitted	Biochar : starch (95:5) (T1)	Biochar : red clay : starch (60:35:5) (T4)
Carbon monoxide (CO) (ppm)	2.33	1.33 ns
Carbon dioxide (CO_2) (ppm)	2,444.00	556.30 ****
Formaldehyde (HCOH) (mg/m^3)	0.009	0.007 ns
Total volatile organic compounds (TVOC) (mg/m^3)	0.014	0.004 ***
Particulate matter 2.5 ($PM_{2.5}$) (µg/m^3)	50.00	30.00 *
Particulate matter 1.0 ($PM_{1.0}$) (µg/m^3)	58.00	38.33 ns

Note: Pairwise comparison, *, *** and **** show the significance, and "ns" means not significant at $P \leq 0.05$.

known as its heating value. T4 had the highest calorific value for this investigation at 5,650 cal/g, but it did not differ substantially from the control, which had a calorific value of 5,601 cal/g. However, T4 exhibited a higher heating value than the study of Amrullah et al. (2020). They studied the calorific value of mixed biomass briquettes between oil palm empty fruit bunches and oil palm kernel shells by using tapioca starch as a binder, and they reported that 5,475 cal/g was the highest calorific value. It was found at a ratio of 25:27 (oil palm empty bunches and oil palm shells). Chumsang and Upan (2014) also investigated the production of fuel briquettes from the waste of the Borassus or Palmyra palm (*Borassus flabellifer*). The findings demonstrated that the temperature characteristics of the fruit calyx, shells, and palm husks were examined. All six mixing ratios exhibited thermal values ranging from 5,281.60 to 6,702.00 cal/g in an experiment to examine the qualities of blends of palm wastes (husks and fruit calyx: shells) in six different mixing ratios using starch paste as a binding agent. Notably, T4 does not show sufficient porous structure (Figure 2). The mixing with red clay in the briquettes and clay particles that obstruct the porous structure of the biochar may be the reason why T4 does not offer a high heating value. Because biochar pores offer more airflow channels, more oxygen may move through the briquette during combustion, improving the briquette's combustion potential to increase energy release and burn more thoroughly (Sanka et al., 2024; Ayaa et al., 2025). However, mixing red clay into biochar briquettes has the advantage of increasing bulk density, resulting in a longer burning time. Briquettes fabricated using clay as a binder show increased density and hardness. Briquettes with high bulk density are ideal for handling, storage, and transportation (Deshannavar et al., 2018; Celestino et al.,2023).

EDX analysis confirmed that the primary elements in the composite biochar briquettes were carbon (C) and oxygen (O), with minor elements including aluminum (Al), silicon (Si), iron (Fe), sodium (Na), calcium (Ca), titanium (Ti), magnesium (Mg), and copper (Cu). A high carbon content is generally associated with higher heating values, while inorganic elements affect combustion in various ways. For example, calcium may enhance combustion, while silicon lowers the ash melting point, increasing the risk of fouling. Elements like potassium (K), sodium (Na), and magnesium (Mg) may also contribute to combustion challenges such as corrosion and slagging (Moreno et al., 2016; Senila et al., 2022; Yiga et al., 2023). The chemical composition of T4 was further validated by XRD analysis. The pattern was consistent with Rehali et al. (2025), who observed broad diffraction peaks between 20° and 30° (2θ), indicating the presence of amorphous carbon. The incorporation of red clay introduced additional inorganic minerals, as confirmed by the similarity with results from Nawaukkaratharnant et al. (2022), who reported that red clay in Thailand typically contains quartz, hematite, and alumina.

During combustion, composite OPF biochar briquettes release particulate matter and harmful gases such as CO, CO_2, TVOCs, and HCHO. Although T4 did not exhibit the highest calorific value, it significantly reduced air pollutant emissions compared to T1 (Table 1). Because of the absence of red clay, T1 emits more CO and CO_2 than T4. T1 has a high carbon content due to the biochar, and starch helped to increase CO and CO_2 emissions. This outcome is consistent with the VOC emission from T4. Briquettes containing inorganic minerals may lower VOC emissions. When hydrated lime ($Ca(OH)_2$) was added to charcoal briquettes, Cong et al. (2021) demonstrated that the VOC emissions were greatly decreased. $PM_{1.0}$ and $PM_{2.5}$ particulate matter emissions declined in comparison to T1, particularly $PM_{2.5}$ emissions of T4, which were within the WHO Air Quality Guidelines (AQGs) standard safety threshold (≤ 50 μg/m³) (Glalah et al., 2024). Otieno and Otieno (2020) created a clay-charcoal blend briquette for domestic usage and discovered that it didn't emit smoke during combustion, potentially lowering $PM_{2.5}$ emissions.

CONCLUSIONS

In conclusion, this research characterized and analyzed five different ratios of composite briquettes made from OPF biochar and red clay. According to proximate analysis, T4 exhibited the highest energy potential, with a calorific value of 5,650 cal/g, and was therefore selected for further investigation. A comprehensive examination of the morphological and chemical characteristics was performed using SEM, EDX, and XRD. SEM revealed an uneven and rough surface, with clay particles obstructing the biochar's pore structure, which may affect the heating value. EDX analysis identified carbon, aluminum, silicon, and iron as significant elemental components of the selected composite briquette, while XRD showed a high carbonaceous content along with crystalline inorganic minerals such as silicon dioxide. Furthermore, T4 significantly reduced air pollution emissions, indicating its suitability for domestic use. However, further research on improving calorific value and exploring alternative binder types is necessary to develop briquettes with both high energy content and low environmental impact for sustainable energy applications.

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