

Characterization and Analysis of Bottom Ash Derived from the Combustion of Empty Fruit Bunch (EFB) for Potential Fertilizer

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Abstract

The palm oil industry generates large quantities of Empty Fruit Bunch (EFB), creating challenges in waste management and environmental sustainability. Combustion is commonly used to reduce EFB volume but the resulting bottom ash is often discarded despite its potential nutrient. This study investigates the characteristics of bottom ash produced from the combustion of EFB pellets to assess its suitability for potential fertilizer applications. EFB pellets were combusted at 200°C, 300°C, and 400°C for 60 minutes inside a combustion chamber under controlled laboratory conditions. Thermogravimetric analysis (TGA) was conducted to evaluate the thermal decomposition behavior of raw EFB, while scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) and X-ray diffraction (XRD) were used to analyze ash morphology, elemental composition, and mineralogical phases. The results indicate that increasing combustion temperature enhances mineral crystallinity and reduces residual carbon content. The bottom ash contained essential plant nutrients such as potassium, calcium, magnesium, phosphorus, and silicon, with progressive formation of thermally stable mineral phases at higher temperatures. Among the conditions studied, combustion at 300 °C produced ash with balanced mineral composition and limited sintering, indicating favorable characteristics for agricultural application.

1. Introduction

As a major contributor to agricultural economy in Malaysia, the palm oil industry generates substantial quantities of biomass waste, particularly empty fruit bunches (EFB). The large volume of EFB produced presents significant challenges in waste management and disposal. The conventional practices such as open burning and natural decomposition contribute to environmental pollution and greenhouse gas emissions. Combustion is often applied as a volume-reduction method, resulting in the formation of bottom ash as a by-product [2]. However, this bottom ash is typically treated as industrial waste and disposed of in landfills, despite containing

agriculturally valuable nutrients such as potassium, calcium, and magnesium [1,3]. Such disposal practices not only pose environmental concerns but also limit opportunities for nutrient recovery and sustainable utilization of biomass residues.

Growing interest in circular economy frameworks has driven research into converting agro-industrial residues into value-added materials. Biomass-derived products such as EFB-based biochar have demonstrated potential to improve soil structure, enhance nutrient retention, and increase crop productivity [4]. In a similar context, EFB bottom ash presents promise as a fertilizer or soil amendment. However, its application remains limited due to insufficient understanding of its physicochemical properties, compositional variability, and potential environmental implications when applied to soil systems.

The characteristics of bottom ash are strongly influenced by combustion parameters, particularly temperature and residence time, which govern mineral transformation, crystallinity, elemental distribution, and thermal stability [8]. Inadequate control or understanding of these parameters may result in incomplete combustion, unstable mineral phases, or undesirable sintering effects, which affecting ash quality and usability. The absence of standardized characterization data has contributed to uncertainty regarding the safe and effective reuse of EFB bottom ash in agricultural applications.

Accordingly, detailed characterization of EFB bottom ash produced under controlled combustion conditions is essential. Analytical techniques such as thermogravimetric analysis (TGA), X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) enable systematic evaluation of thermal behavior, mineralogical composition, morphology, and elemental content. Establishing the relationship between combustion temperature and ash properties is critical for identifying optimal conditions that maximize nutrient availability while maintaining structural and mineral stability.

This study aims to systematically investigate the influence of combustion parameters on the formation and characteristics of bottom ash derived from EFB. Specifically, the work focuses on analyzing the effects of controlled combustion temperatures on bottom ash generation and evaluating its physical and chemical properties, with particular emphasis on elemental composition, mineral phases, and thermal behaviour. Through comprehensive characterization using TGA, XRD, and SEM-EDX, this research seeks to identify combustion conditions that yield stable, nutrient-rich ash suitable for potential fertilizer applications. The outcomes are expected to support sustainable biomass waste management, enhance resource recovery within the palm oil industry, and promote environmentally responsible practices consistent with circular economy principles.

2. Methodology

This study details the methodological framework included for this investigation of combustion behavior and the subsequent characterization of bottom ash derived from palm oil Empty Fruit Bunch (EFB). The methodology is systematically structured to fulfill the research objectives, encompassing sample preparation, controlled combustion procedures, and comprehensive analytical characterization. A rigorous experimental design is implemented to ensure the acquisition of reproducible and reliable results, thereby contributing to a deeper understanding of EFB combustion performance and the properties of bottom ash produced under varying combustion conditions.

2.1 Sample Preparation

The EFB was processed through drying, size reduction, and densification to produce uniform cylindrical pellets, as shown in Fig. 1. When combustion is conducted, 1 kg of prepared EFB pellets is used to achieve complete combustion and to produce enough bottom ash for detailed analysis. The pellets were first ground and sieved through a stainless-steel mesh to remove smaller particles. Following this, the sample is arranged in accordance with the requirements of the specific experimental setup. Prior to every combustion conducted, the combustion chamber was cleaned thoroughly and preheated to remove residual matter from other experiments. This procedure is necessary to ensure stable and consistent combusting conditions.

The EFB pellets were gradually and carefully fed into the combustion chamber through a fuel feeder to prevent any unforeseen incidents that could disrupt the combustion process. For temperature monitoring while undergo the combustion, thermocouples were placed at the exhaust and inside the combustion chamber, specifically at the flame, where the combustion of the EFB pellets occurs. This arrangement ensured uniform thermal conditions throughout the process of combustion.

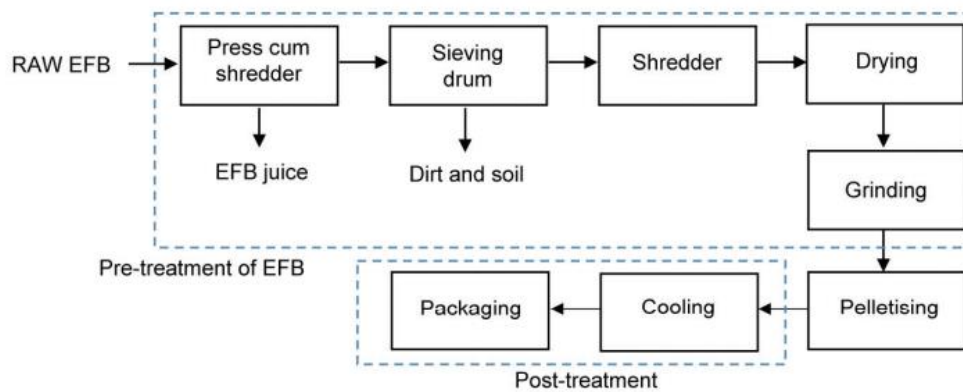


Fig. 1 Flowchart of the production process for EFB pellets [5]

2.2 Combustion Process

EFB pellets were combusted in a laboratory-scale combustion chamber, as shown in Fig. 2. The combustion chamber was fully equipped, including a blower to support the combustion process. Each experiment was conducted for a duration of 60 minutes per sample. Three samples were obtained from the combustion process, corresponding to three different combustion temperatures: 200, 300, and 400 °C.

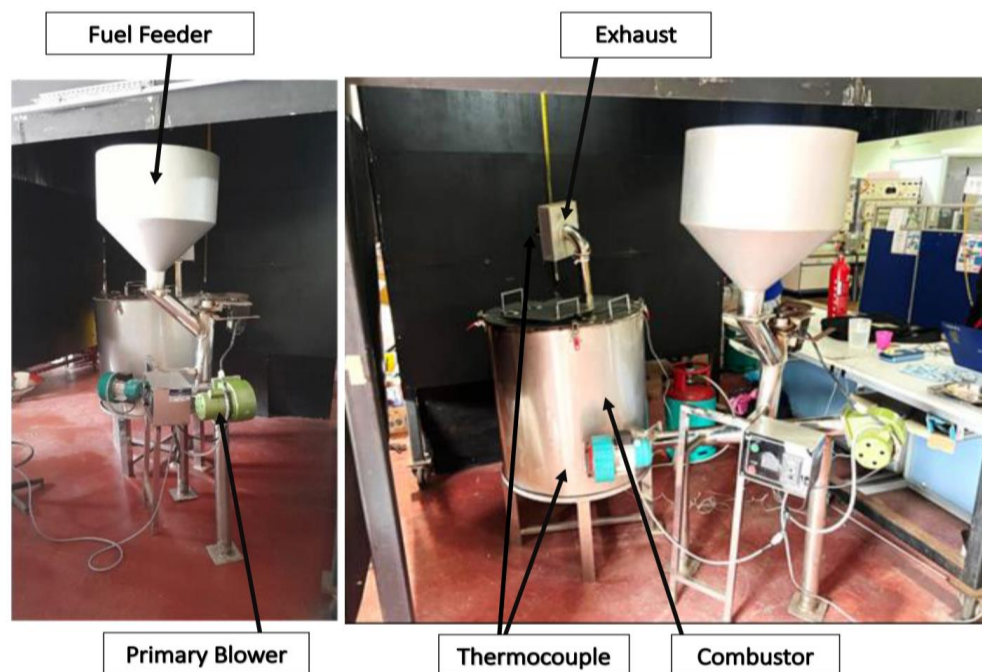


Fig. 2 Schematic diagram of the combustion chamber

2.3 Ash Collection and Storage

Bottom ash was collected immediately after cooling phase to reduce exposure to atmospheric moisture and avoid contamination. The ash samples were then carefully separated from any residual unburned material by mechanical sieving to a particle size of 71 μm , ensuring sample purity for subsequent characterization. Fig. 3 illustrates the ash samples following the sieving process.



Fig. 3 Sample collection

2.4 Analytical Techniques

This study applied various analytical methods to thoroughly characterize the bottom ash resulting from the combustion of palm oil EFB. Table 1 presents an overview of all the methods applied for this research. Thermogravimetric Analysis (TGA) was performed to assess the thermal decomposition behavior of the raw EFB pellet, including its mass loss pattern and thermal stability. Scanning Electron Microscopy (SEM) was conducted to examine the surface morphology of the ash, offering insight into particle structure, shape, and surface features while Energy Dispersive X-ray Spectroscopy (EDX) was used to assess the overall elemental composition of the sample. X-ray Diffraction (XRD) analysis was carried out to identify the mineral phases present and to differentiate between crystalline and amorphous structures that affect ash behavior and potential utilization. Collectively, all these techniques offer a comprehensive understanding of the physical, chemical, and structural characteristics of EFB bottom ash, as well as insights into optimal combustion conditions.

Table 1 Analytical techniques for ash characterization [10]

Technique	Purpose	Information provided
Thermogravimetric analysis (TGA)	To study the thermal behavior and stability of the sample under controlled heating	Mass changes, decomposition patterns, thermal stability, moisture and residual ash content
Scanning Electron Microscopy (SEM)	To examine the surface morphology and microstructure of the sample	Detailed images of particle shape, size, surface texture, and microstructural features
Energy Dispersive X-ray Spectroscopy (EDX)	To determine the elemental composition of the sample	Qualitative and semi-quantitative elemental analysis
X-ray Diffraction (XRD)	To identify crystalline phases and analyze the structural properties of the sample	Mineralogical composition; crystalline vs. amorphous phases and crystallinity information.

3. Results and Discussion

In this study, EFB pellets were combusted in a controlled laboratory combustor for a fixed duration of 60 minutes at three different temperatures which are 200°C, 300°C, and 400°C. For each experiment, 1 kg of pellets was used, and the resulting bottom ash was collected for further analysis. Prior to combustion, Thermogravimetric Analysis (TGA) was performed on the raw EFB pellets to assess their thermal decomposition behavior under controlled heating conditions. The ash samples were further characterized using Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDX) and X-ray Diffraction (XRD) to examine their morphology and elemental composition. This chapter presents the observed variations in ash structure, chemical makeup, and thermal stability with respect to combustion temperature, providing insights into the decomposition characteristics of EFB pellets and potential applications of the resulting bottom ash.

3.1 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was performed on raw Empty Fruit Bunch (EFB) pellets to examine their thermal decomposition behaviour under controlled heating conditions. The resulting TGA curve, together with the derivative thermogravimetric (DTG) and heat flow data, offers valuable insight into the mass loss stages and the thermal stability of the raw biomass prior to combustion. The results of the TGA performed are shown in Fig. 4 below.

The EFB pellet sample, initially weighing approximately 9.3 mg, experiences a mass reduction of about 7.23 mg when heated to 1000 °C, reflecting extensive breakdown of organic constituents and ash formation. A minor mass loss observed below 150 °C is attributed to the evaporation of moisture present in the sample. The most significant mass loss occurs between 200 °C and 380 °C, with a major reaction step identified at around 310 °C resulting in a mass loss of around 4.77 mg. This temperature range corresponds to the thermal degradation of hemicellulose and cellulose, which represent the thermally labile fraction of lignocellulosic biomass. At temperatures above 400 °C, the mass loss rate decreases significantly, reflecting the gradual decomposition of lignin and the stabilization of char and inorganic residues.

The derivative thermogravimetric (DTG) curve supports the TGA results by indicating a well-defined endothermic peak during the main degradation stage. The thermal event is characterized by an onset temperature of approximately 205 °C, a peak maximum at around 310 °C, and an offset temperature near 377 °C defining the active decomposition interval of the EFB pellet. The DTG peak confirms that the most intense devolatilization process takes place within this temperature range, consistent with the rapid breakdown of cellulose and hemicellulose structures.

A clear correlation is observed between the TGA and DTG results, as the maximum mass loss rate corresponds with the DTG peak temperature at approximately 310 °C. This confirms that the principal decomposition mechanism of the EFB pellet is thermally activated and involves simultaneous mass loss and endothermic energy consumption. The absence of significant DTG transitions beyond 400 °C suggests that no major phase or chemical transformations occur at elevated temperatures, indicating enhanced thermal stability of the residual char.

Overall, the combined TGA-DTG results demonstrate that the thermal decomposition of EFB pellets is dominated by hemicellulose and cellulose degradation within the temperature range of 200–380 °C, while lignin contributes to thermal stability at higher temperatures. These findings provide critical insight into the thermal behaviour and decomposition kinetics of EFB pellets, which are essential for optimizing their performance in thermochemical conversion processes.

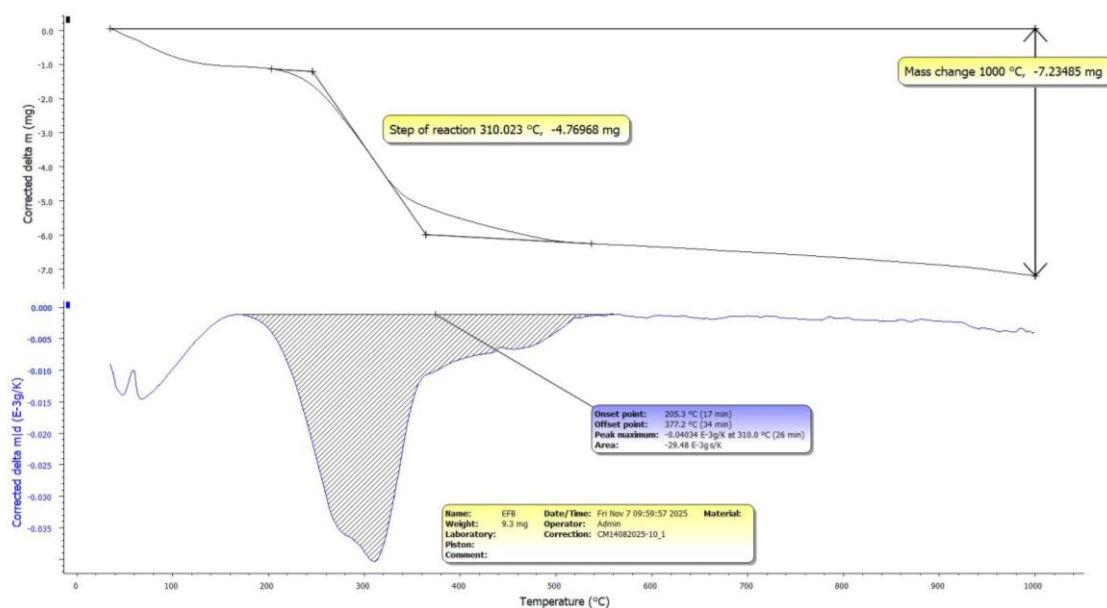


Fig. 4 TGA and DTG curves of raw Empty Fruit Bunch (EFB) pellets

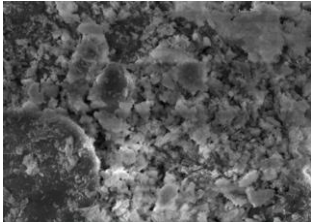
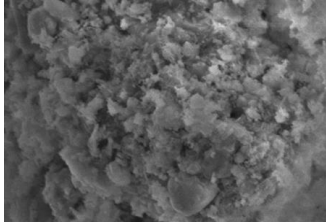
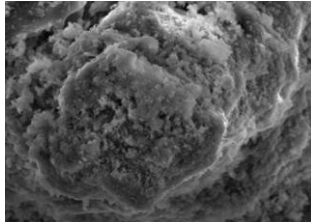
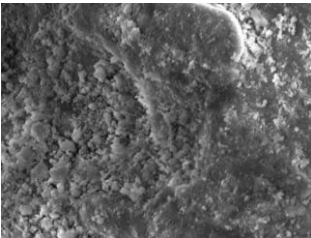
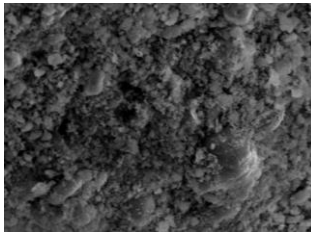
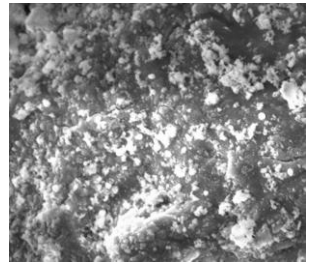
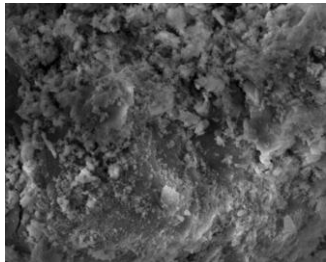
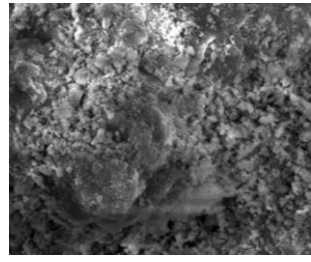
Table 2 Summary of TGA and DTG Analysis

Parameter	Value	Description
Initial weight	9.3 mg	Sample mass for TGA
Total mass loss	7.23 mg	Mass loss observed up to 1000 °C
Peak maximum temperature	310.0 °C	Main thermal decomposition stage
Onset temperature	205.3 °C	Temperature where decomposition begins
Offset temperature	377.2 °C	Temperature where decomposition ends
Mass loss at major step	4.76 mg	Decomposition of hemicellulose and cellulose

3.2 SEM-EDX Analysis

SEM-EDX analysis was conducted to examine the microstructural characteristics and elemental composition of the bottom ash samples. SEM imaging reveals the surface morphology and particle structure, while EDX identifies the elemental constituents, allowing correlation between thermal treatment conditions and material composition. The SEM images for each sample are presented in Table 3. For each combustion temperature, images were captured at three different spots to ensure a representative observation of the surface morphology. All images were obtained at a magnification of 2000×. This analysis provides insights into the microstructural features, particle morphology, and elemental distribution, which are essential for understanding the thermal and chemical behavior of the samples.

Table 3 SEM images of EFB bottom ash at 200°C, 300°C and 400 °C at three different locations

Temperature (°C)	Location 1	Location 2	Location 3
200			
300			
400			

The SEM images for 200°C show that the bottom ash particles exhibit an irregular and non-uniform morphology, with particles of varying sizes and rough surface textures. Fine particles appear to be adhered to larger ash clusters, suggesting possible partial melting or sintering during combustion. The porous and uneven

surfaces observed across different spots may indicate incomplete decomposition of the original biomass structure and the presence of mineral-rich phases. Overall, similar morphological features are observed at different locations, suggesting that the images are representative of the sample.

For the temperature of 300°C, the ash particles show irregular shapes with rough and uneven surfaces, with fine particles agglomerated on larger fragments. This morphology indicates incomplete combustion, where thermal decomposition and devolatilization occur rather than complete oxidation of the biomass. The ash structure is highly porous, with small pores and micro-voids formed due to the release of moisture and volatile compounds during the early stage of combustion, resulting in a loosely bonded ash matrix. No smooth, spherical, or melted particles are observed, confirming that mineral melting or fusion did not occur under these combustion conditions. Minor differences in particle texture suggest non-uniform heat and oxygen distribution during combustion, which led to variations in burnout efficiency.

Upon increasing the temperature to 400°C, the ash particles displayed irregular and clustered structures, with fine particles adhering to larger fragments. The surfaces appear rough and compact in some areas, indicating partial combustion and progressive decomposition of the biomass. Compared to very loose ash structures, the denser regions suggest that prolonged heating allowed some particles to bond together. The presence of fine particles filling surface pores indicates ongoing release of volatile matter and incomplete burnout of organic components. No smooth or spherical particles are observed, confirming that ash fusion or sintering did not occur under these combustion conditions. Variations in texture and compactness indicate uneven heat and oxygen distribution, which influenced the degree of combustion and ash formation. Overall, the ash morphology reflects incomplete combustion with limited mineral restructuring, typical of low to moderate combustion temperatures.

3.3 Elemental Composition

The elemental composition of the bottom ash was studied using Scanning Electron Microscopy combined with Energy Dispersive X-ray Spectroscopy (SEM-EDX). The results show that combustion temperature has a clear effect on the distribution of elements in the ash, indicating changes that occur during the burning of EFB pellets. At all temperatures, the ash mainly contained carbon (C) and oxygen (O), together with measurable amounts of magnesium (Mg), aluminum (Al), silicon (Si), phosphorus (P), sulfur (S), potassium (K), and calcium (Ca). The presence of these elements is mainly attributed to the natural mineral composition of EFB and the transformations that occur under combustion conditions.

The EDX analysis in Table 4 shows that combustion temperature influences the elemental composition of the bottom ash derived from EFB pellets. Carbon content decreases at 400 °C compared to lower temperatures, indicating enhanced thermal decomposition and reduced residual organic matter. Oxygen remains the dominant element across all conditions, reflecting the formation of oxide-based mineral phases in the ash.

Silicon exhibits a clear increasing trend with temperature, suggesting the relative enrichment of silica as volatile components are removed during combustion. Potassium and calcium are present in high concentrations at all temperatures, with potassium showing slight enrichment at elevated temperatures, which may contribute to ash-related operational issues such as slagging. Calcium plays an important role in stabilizing the ash through the formation of calcium-based compounds.

Magnesium and aluminium are detected in moderate amounts with minimal variation, indicating their stable presence in the ash matrix. Phosphorus and sulphur appear in smaller quantities and generally decrease with increasing temperature, likely due to volatilization or transformation during combustion. Overall, the results indicate a shift from organic-rich material toward mineral-dominated ash with increasing combustion temperature, which has implications for ash behaviour and potential reuse.

Table 4 Summary of elemental composition of ash at different temperatures

Element	Temperature		
	200°C	300°C	400°C
C	5.46	5.72	2.73
O	49.62	46.70	48.96
Mg	3.16	2.40	2.29
Al	1.66	1.11	1.29
Si	12.29	14.61	18.37
P	1.55	1.63	1.00
S	0.76	0.42	0.28
K	20.30	21.57	20.66
Ca	5.20	5.84	4.41

3.4 X-ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) analysis was performed to identify the crystalline phases present in the bottom ash samples produced at 200°C, 300°C, and 400°C over a 60-minute combustion period. The results indicate a clear transformation of mineral phases and crystallinity depending on the combustion temperature, reflecting the extent of thermal decomposition and mineral restructuring in the ash.

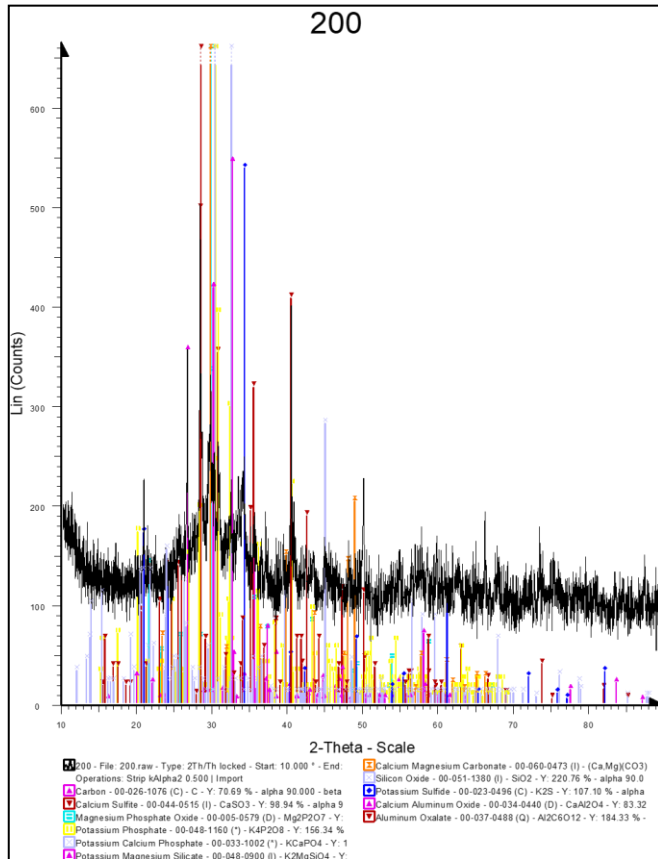


Fig. 5 XRD result for 200°C (60 minutes)

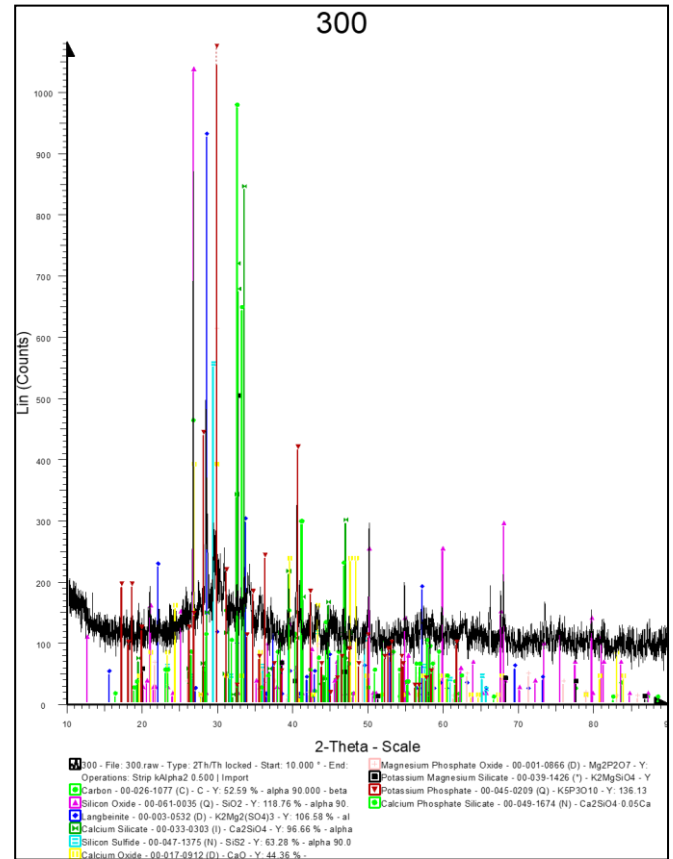


Fig. 6 XRD Result for 300°C (60 minutes)

At a combustion temperature of 200°C, the pattern shown in Fig. 5 is mainly dominated by high-intensity peaks of silica (SiO_2), meaning silica is the most abundant and well-formed crystal in the sample. Strong peaks related to potassium phosphate and potassium sulfide indicate that potassium-rich minerals are also present in significant amounts, likely formed during combustion. Other compounds such as calcium- and magnesium-based oxides and carbonates appear with lower intensities, showing that they are present in smaller amounts. The relatively low carbon peak suggests some unburnt carbon remains due to incomplete combustion.

With increasing combustion temperature to 300 °C, the XRD pattern in Fig 6 exhibited that silica is still one of the main phases, but its intensity is slightly lower compared to 200°C sample. At the same time, potassium phosphate and sulfate-containing compounds show stronger peaks, indicating better formation of more stable mineral phases. Calcium silicate also becomes more noticeable, suggesting that calcium and silica reacted more as the treatment conditions increased. The lower carbon intensity compared to sample 200 shows that combustion was more complete and less unburnt material remained.

After a further increase in combustion temperature up to 400 °C, the XRD pattern shows very strong and sharp peaks, especially for silica and calcium silicate, indicating that these compounds are highly crystalline and stable. This result shown in Fig 7. Potassium sulfate also shows high intensity, suggesting that potassium has transformed into a more stable form. In terms of magnesium and aluminum-based oxides, it appears more clearly, which usually form at higher temperatures. The carbon detected in this sample is likely more ordered in structure rather than leftover unburnt carbon. Overall, the increase in peak intensity and sharpness from the sample of 200°C to 400°C shows that the minerals become more stable and well-crystallized as the treatment conditions become more severe.

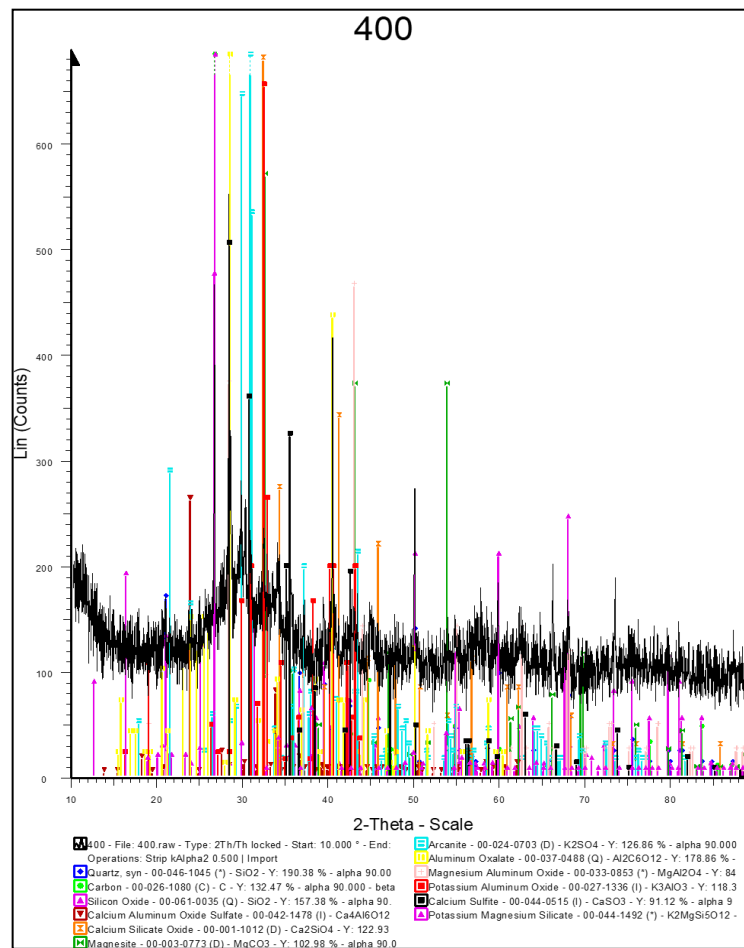


Fig. 7 XRD result for 400°C (60 minutes)

In summary, the changes in Y-intensity across the samples of 200°C, 300°C, and 400°C indicate a clear progression in mineral formation and stability. As the treatment conditions increase, the XRD peaks become stronger and sharper, showing improved crystallinity and the transformation of less stable, alkali-rich phases into more stable silicate, sulfate, and oxide compounds. This trend suggests that higher treatment severity promotes mineral stabilization and a more well-defined crystalline structure in the final material.

Table 5 Summary of XRD Results for Bottom Ash Samples Produced at 200°C, 300°C, and 400°C

Temperature (°C)	Major compound/element	Chemical formula	Crystalline structure	Relative intensity (Y%)	Remarks
200	Silicon oxide	SiO ₂	Hexagonal	220.76	Indicates silica-rich biomass ash and low-temperature combustion limits full crystallization
	Aluminium oxalate	Al ₂ C ₆ O ₁₂	Monoclinic	184.33	Suggests incomplete thermal decomposition of aluminium-organic complexes
	Potassium phosphate	K ₄ P ₂ O ₈	Orthorhombic	156.34	Represents nutrient-rich mineral retained at low combustion temperature
	Potassium sulfide	K ₂ S	Cubic	107.10	Formed under oxygen-limited

					conditions and indicates incomplete oxidation
	Calcium sulfite	CaSO_3	Monoclinic	98.94	Suggesting partial oxidation of sulfur species
	Calcium aluminium oxide	CaAl_2O_4	Orthorhombic	83.32	Early-stage mineral transformation
	Carbon	C	-	70.69	Indicates unburned carbon due to insufficient combustion temperature
300	Potassium phosphate	$\text{K}_5\text{P}_3\text{O}_{10}$	Orthorhombic	136.13	Stable potassium-phosphorus phase and beneficial for fertilizer potential [6]
	Silicon oxide	SiO_2	Hexagonal	118.76	Increased crystallinity due to higher thermal exposure
	Langbeinite	$\text{K}_2\text{Mg}_2(\text{SO}_4)_3$	Cubic	106.58	Sulfate mineral formed during mineral recombination
	Calcium silicate	Ca_2SiO_4	Monoclinic	96.66	Indicates interaction between Ca and Si at elevated temperature
	Silicon sulfide	SiS_2	Orthorhombic	63.28	Minor sulfur compound formed under limited oxygen availability
	Carbon	C	-	52.59	Reduced but still present unburned carbon
	Calcium oxide	CaO	Cubic	44.36	Product of CaCO_3 decomposition [7] and indicating progressing combustion
400	Quartz	SiO_2	Hexagonal	190.38	Thermally stable silica phase at higher combustion temperature
	Aluminium Oxalate	$\text{Al}_2\text{C}_6\text{O}_{12}$	Monoclinic	178.86	Indicates partial persistence of aluminium-organic compounds
	Carbon	C	-	132.47	Significantly reduced unburned carbon content
	Arcanite	K_2SO_4	Orthorhombic	126.86	Stable potassium sulfate formed under more complete

Calcium silicate oxide	Ca ₂ SiO ₄	Monoclinic	122.93	oxidation A thermally stable calcium–silicate phase and contributing to ash stability and influencing its alkalinity and agronomic behavior
Potassium aluminium oxide	K ₃ AlO ₃	Cubic	118.3	Result of alkali–alumina interaction at higher temperature
Magnesite	MgCO ₃	Trigonal	102.98	Indicates magnesium retention and incomplete carbonate decomposition
Calcium sulfite	CaSO ₃	Monoclinic	91.12	Formed under partial oxidation conditions, indicating retained sulfur in the ash
Magnesium aluminium oxide	MgAl ₂ O ₄	Face-centered cubic	84	High-temperature stable mineral phase indicating advanced ash transformation

4. Conclusion

This study systematically evaluated the combustion behavior of Empty Fruit Bunch (EFB) pellets and the characteristics of the resulting bottom ash produced at different combustion temperatures. Thermogravimetric analysis revealed that the major thermal decomposition of EFB occurred between 200 °C and 380 °C, primarily due to the degradation of hemicellulose and cellulose, while lignin contributed to thermal stability at higher temperatures. SEM observations showed irregular, porous ash particles with agglomerated fine structures, indicating incomplete combustion and limited mineral fusion across all temperatures studied. Elemental analysis confirmed the presence of agriculturally important nutrients, including potassium, calcium, magnesium, phosphorus, and silicon, with a noticeable reduction in residual carbon at higher combustion temperatures.

XRD analysis demonstrated progressive mineral transformation with increasing temperature, characterized by enhanced crystallinity and the formation of stable silicate, phosphate, and sulfate phases. Combustion at 300 °C produced a well-balanced ash composition with improved mineral stability, reduced unburned carbon, and minimal sintering effects, making it the most favorable condition among those investigated. Overall, the results indicate that EFB bottom ash generated under controlled combustion conditions possesses characteristics suitable for potential fertilizer or soil amendment applications. This study supports the sustainable utilization of palm oil biomass residues, contributing to waste reduction, nutrient recovery, and the advancement of circular economy practices within the palm oil industry.

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Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Hannah Batrisyia Azzhar, Mohd Faizal Tukimon, Shahrin Hisham Amirnordin; **data collection:** Hannah Batrisyia Azzhar; **analysis and**

interpretation of results: Hannah Batrisyia Azzhar, Mohd Faizal Tukimon, Shahrin Hisham Amirnordin; **draft manuscript preparation:** Hannah Batrisyia Azzhar, Mohd Faizal Tukimon, Shahrin Hisham Amirnordin, Md Asrul Nasid Masrom, Mohammad Faizal Jabbar. All authors reviewed the results and approved the final version of the manuscript

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