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Effect of Low-Temperature Heat Treatment on Surface Morphology and Elemental Composition of Empty Fruit Bunch (EFB) Biomass Analyzed by SEM-EDS

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Abstract
This study investigates the effect of low-temperature heat treatment on the surface morphology and elemental composition of Empty Fruit Bunch (EFB) biomass using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). EFB samples were heated at various temperatures ranging from 150°C to 200°C to observe structural changes and the distribution of elements on the biomass surface. SEM analysis revealed that at 150°C, the EFB surface exhibited a rough and porous texture with visible fibres. Increasing the temperature to 190°C and 200°C resulted in smoother surfaces with denser and more compact morphologies due to thermal degradation and particle fusion. EDS results identified the dominant elements present, including Carbon (C), Oxygen (O), Potassium (K), Calcium (Ca), and smaller amounts of Magnesium (Mg), Phosphorus (P), Silicon (Si), and Chlorine (Cl). At 150°C, Carbon content was recorded at 51.43%, while Oxygen was 33.21%. As the temperature increased to 200°C, Carbon slightly increased to 53.77%, and Oxygen decreased to 30.84%, indicating partial carbonization. Furthermore, the presence of potassium and calcium somewhat reduced from 4.39% and 3.37% at 150°C to 3.17% and 3.08% at 200°C, respectively, which suggests mineral volatilization during heating. These findings demonstrate that temperature significantly influences the physical and chemical characteristics of EFB biomass, with the 190–200°C range showing the most notable changes in surface compaction and elemental reduction. This research provides valuable insights into optimizing EFB as a precursor material for bio-based applications through controlled thermal treatment.

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1. Introduction

Biomass is a renewable energy source with great potential to support the transition to environmentally friendly energy. One of the abundant biomass wastes is empty fruit bunches (EFB), or empty oil palm bunches, which are solid waste from the palm oil industry. Indonesia produces more than 20 million

tons of EFB annually, but its utilization is still limited as compost or direct fuel with low efficiency (Dolah, Karnik, & Hamdan, 2021; Erdiwansyah, Gani, Desvita, et al., 2024; Gani et al., 2025; NOOR, Arif, & Rusirawan, 2025). Therefore, developing EFB as a raw material for biocoke through a gradual heating process is a concern to improve the quality of energy and material characteristics. Thermal processes such as pyrolysis, carbonization, and torrefaction are often applied in biomass processing to increase the bioproduct structure's calorific value and stability. The torrefaction process at low to moderate temperatures (200–300°C) can improve the physicochemical properties of biomass by reducing volatile content and increasing fixed carbon content (Adeleke et al., 2021; Bahagia, Nizar, Yasin, Rosdi, & Faisal, 2025; Erdiwansyah et al., 2025; Iqbal, Rosdi, Muhtadin, Erdiwansyah, & Faisal, 2025; Jalaludin, Kamarulzaman, Sudrajad, Rosdi, & Erdiwansyah, 2025; Yana, Mufti, Hasiany, Viena, & Mahyudin, 2025). However, studies on the effect of low-temperature heating below 200°C on changes in surface morphology and EFB element content are still limited, even though this stage is essential as an initial process before further carbonization (Gani, Erdiwansyah, Desvita, Munawar, et al., 2024; Gani, Erdiwansyah, et al., 2023; Muhtadin, Rosdi, Faisal, Erdiwansyah, & Mahyudin, 2025; Phang et al., 2024).
Biomass surface characterization after heat treatment is essential to understand the changes in microstructure and distribution of its constituent elements (Erdiwansyah et al., 2023; Gani, Erdiwansyah, Desvita, Meilina, et al., 2024; Grams, 2022; Jalaludin et al., 2025). Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) methods are effective in evaluating biomass structural damage and analyzing dominant elements, such as carbon, oxygen, and heavy metals (Gani, Adisalamun, et al., 2023; Radenković et al., 2024; Rosdi, Maghfirah, Erdiwansyah, Syafrizal, & Muhibbuddin, 2025). By understanding these changes, the potential of EFB to be used as a solid fuel or raw material for the carbon industry can be further enhanced. In addition, heating temperature affects the distribution of ash and metals in biomass, which can affect combustion performance and emissions (Almardhiyah, Mahidin, Fauzi, Abnisa, & Khairil, 2025; Gani, Erdiwansyah, Desvita, Saisa, et al., 2024; Muhibbuddin, Hamidi, & Fitriyana, 2025; Odzijewicz, Wołejko, Wydro, Wasil, & Jabłońska-Trypuć, 2022). The study found that at temperatures above 200°C, there was an increase in the content of alkali elements, which could cause fouling in the combustion system. Therefore, studies at temperatures below 200°C are essential to map the potential for initial changes in the structure and elements of EFB before entering the advanced carbonization stage (Erdiwansyah, Gani, Mamat, et al., 2024; Fitriyana, Rusiyanto, & Maawa, 2025; Gani, Mahidin, et al., 2024; Pranoto, Rusiyanto, & Fitriyana, 2025).
This study uses SEM-EDS analysis to analyze the effect of EFB heat treatment at 150–200°C on surface morphology and constituent element content. This study is expected to fill the gap in data related to the initial characteristics of EFB after low heating and provide basic information related to the potential for improving the quality of biomass fuels based on palm oil waste. These findings are also expected to contribute to developing more efficient and environmentally friendly biomass energy technology. This study is expected to determine the optimal temperature for the initial heating of EFB before entering the carbonization stage to produce high-quality biocoke. In addition, the results of this study can be used as a reference in developing the EFB biomass industrialization process with a low-temperature approach, significantly minimising structural damage, optimising the distribution of essential elements, and maintaining material stability for sustainable energy applications.

2. Methodology

Fig. 1 shows a schematic of the equipment used in the biocoke manufacturing and testing process. In part (A), the system consists of several main components such as biocoke mould (1), heater (2), thermocouple (3), digital temperature controller (4), hydraulic pump (5), hydraulic system (6), and digital pressure controller (7). Biomass raw material (8) is fed into the biocoke mould and heated to a specific temperature to form a biocoke (9). The heater provides a hot temperature controlled by a digital temperature controller. At the same time, pressure is applied using a hydraulic system to help the biomass compaction process into biocoke with optimal mechanical strength.

Meanwhile, part (B) illustrates a schematic of a high-temperature compression tester used to test the strength of the biocoke after the manufacturing process. In this system, biocoke is placed in a furnace (heater) with a heat shield, and pressure is applied through a crosshead connected to a load cell to measure the compressive force. A temperature controller controls the temperature during testing, and test result data such as temperature and pressure are recorded through a data collection device. This test is essential to determine the resistance of biocoke to pressure at high temperatures, which is a necessary parameter in its application as an alternative solid fuel in industry.

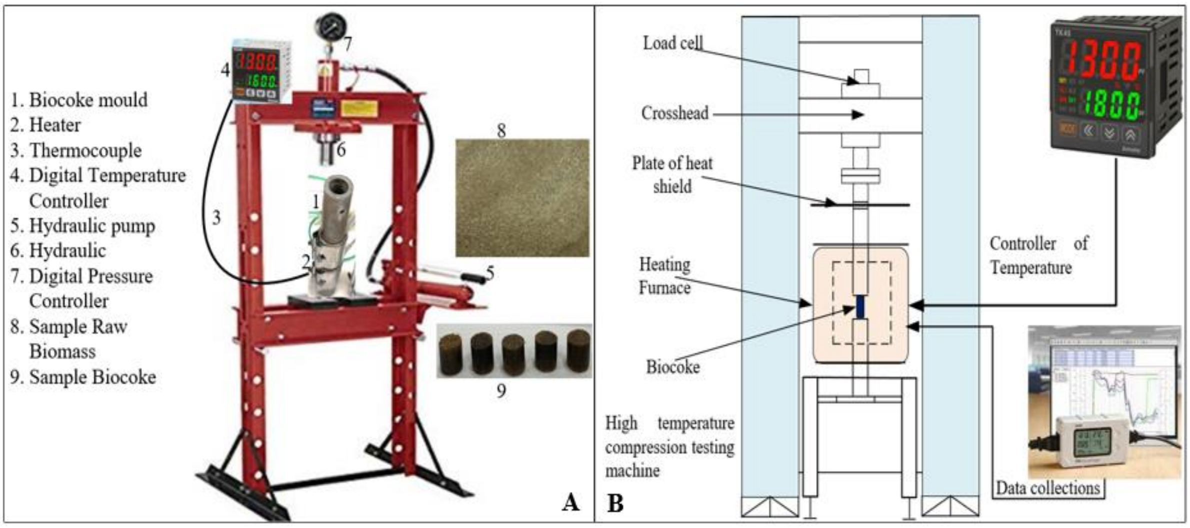
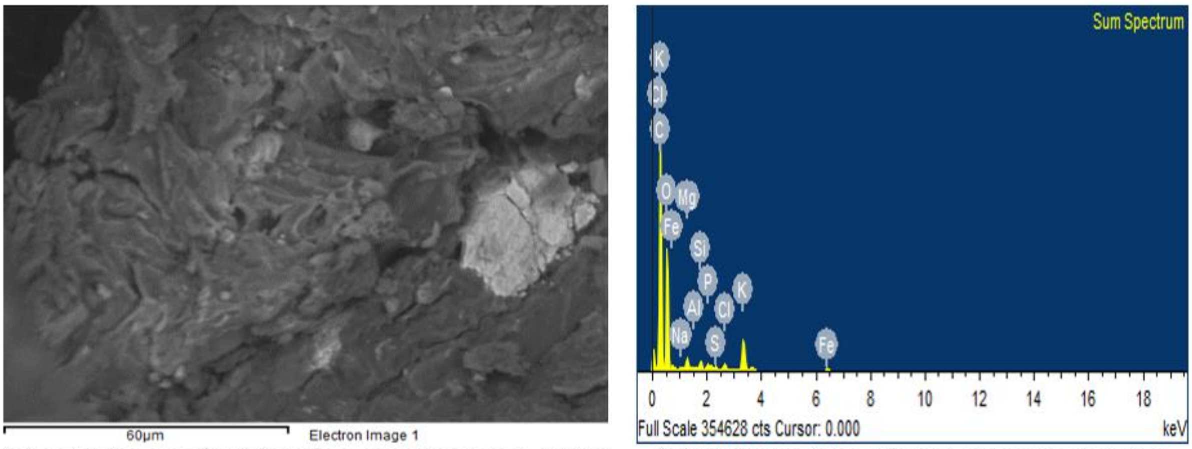


Fig. 1. Schematic Diagram

3. Result & Discussion

Fig. 2 shows the results of morphological analysis and elemental composition of the EFB (Empty Fruit Bunch) sample that has been heated at a temperature of 150°C using SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy) techniques. In **Fig. (a)**, the SEM image reveals the surface structure of the EFB after heat treatment, which appears to have changed texture and porosity. The observed morphology shows the presence of cracks and pores that can contribute to increasing the surface area of the material, which is essential in applications such as adsorption and thermal reactivity. These changes are likely due to the evaporation of volatile compounds and the initial decomposition of lignocellulosic components in biomass due to heating. Meanwhile, **Fig. (b)** displays the EDS spectrum that identifies the main elements contained in the EFB sample after heating. The spectrum shows the presence of elements such as carbon (C), oxygen (O), magnesium (Mg), silica (Si), phosphorus (P), potassium (K), calcium (Ca), and iron (Fe), which are natural components of biomass and inorganic residues after heat treatment. These mineral contents are essential in determining the combustion characteristics and ash potential of EFB-based biocoke. This SEM-EDS analysis provides insight into how heating affects the structure and elemental composition of EFB, which may contribute to the development of biocoke as an alternative fuel with better efficiency.
Fig. 3 shows the results of surface morphology analysis and elemental composition of the EFB (Empty Fruit Bunch) sample after being heated at a temperature of 160°C using the SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy) methods. In **Fig. (a)**, the SEM image shows changes in the surface structure, which are increasingly dense and homogeneous compared to the previous temperature. The EFB surface begins to experience pore shrinkage and coating due to the heating process, which indicates the occurrence of microstructural reconstruction of the biomass material. This condition can potentially affect the physical properties of EFB, such as mechanical strength and thermal stability, which are essential in the biocoke production process. Meanwhile, **Fig. (b)** displays the EDS spectrum that identifies the elemental content in the EFB sample at a temperature

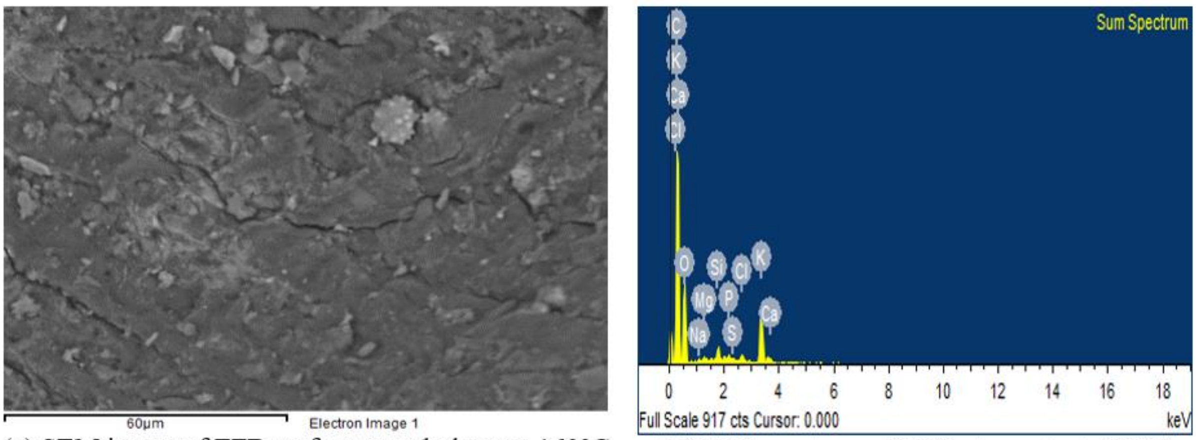
of 160°C. The main elements detected include carbon (C), oxygen (O), potassium (K), calcium (Ca), silica (Si), magnesium (Mg), phosphorus (P), sodium (Na), and sulfur (S). A reasonably high carbon content indicates good energy potential in the EFB from this heating. At the same time, other mineral elements can affect the formation of ash and the combustion properties of the material. With a change in temperature from 150°C to 160°C, a shift in the distribution of these elements can occur as a result of a more intensive thermal reaction, so this SEM-EDS analysis is essential to understand the effect of temperature on the quality of EFB waste-based biocoke.



(a) SEM image of EFB surface morphology at 150°C (b) EDS spectrum of EFB elements at 150°C

Fig. 2. SEM image and EDS spectrum of EFB sample at 150°C

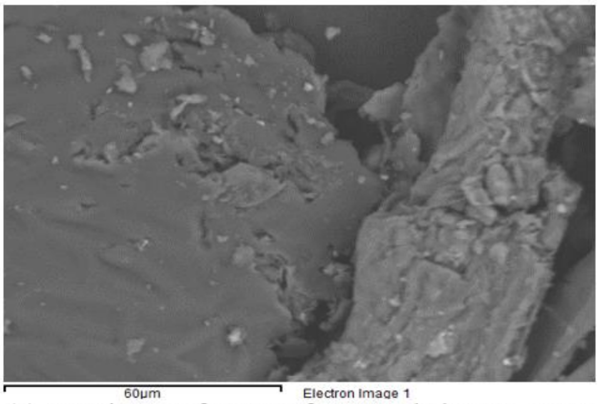
Fig. 4 shows the results of the analysis of the surface morphology and elemental composition of the EFB (Empty Fruit Bunch) sample after the heating process at a temperature of 170°C using the SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy) techniques. **Fig. (a)**, the SEM image shows a significant change in the surface structure of the EFB compared to the previous temperature, where cracks, fractures, and a more fragile and porous texture are visible. This indicates further thermal degradation due to increased temperature, which causes the release of volatile compounds and damage to the lignocellulose structure. These morphological changes affect the material's physical properties, such as decreased mechanical strength but increased surface area, which can be beneficial in subsequent reaction processes. **Fig. (b)**, the EDS spectrum shows the elemental composition of the EFB sample after being heated at a temperature of 170°C. The dominant elements detected include carbon (C), oxygen (O), potassium (K), calcium (Ca), silicon (Si), magnesium (Mg), phosphorus (P), sodium (Na), sulfur (S), and aluminium (Al). The temperature increase can cause an increase in the relative concentration of carbon elements due to the initial carbonization process.



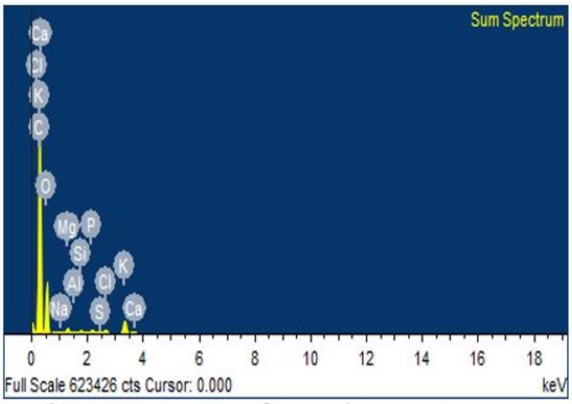
(a) SEM image of EFB surface morphology at 160°C (b) EDS spectrum of EFB elements at 160°C

Fig. 3. SEM image and EDS spectrum of EFB sample at 160°C

In contrast, other mineral elements remain as inorganic residues. The content of these elements is essential to evaluate the energy potential of biocoke and its impact on ash formation and emissions during the combustion process. This SEM-EDS analysis provides a clear picture of the effects of high temperatures on the physical and chemical characteristics of EFB, which can help optimise the temperature of the biocoke production process.



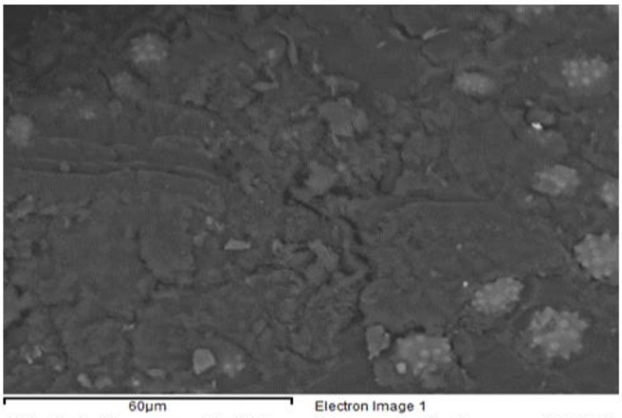
(a) SEM image of EFB surface morphology at 170°C



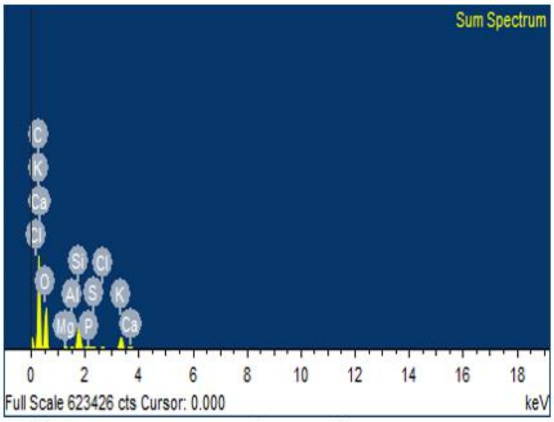
(b) EDS spectrum of EFB elements at 170°C

Fig. 4. SEM image and EDS spectrum of EFB sample at 170°C

Fig. 5 shows the results of surface morphology analysis and elemental content of the EFB (Empty Fruit Bunch) sample after heating at 180°C using the SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy) methods. **Fig. (a)**, the SEM image shows that the EFB surface is increasingly experiencing structural damage compared to the previous temperature, characterized by a rougher surface, small cracks, and scattered fine grains. This shows that increasing temperature significantly impacts the decomposition of organic material, which causes changes in texture due to the release of volatile compounds and damage to the biomass fibre network. This kind of morphology usually indicates the beginning of a mild carbonization process, which can increase the carbon content but reduce the stability of its physical structure. Meanwhile, **Fig. (b)** displays the EDS spectrum showing the distribution of elements in the EFB sample after heating at 180°C. The dominant elements detected include carbon (C), oxygen (O), potassium (K), calcium (Ca), silicon (Si), magnesium (Mg), phosphorus (P), sodium (Na), sulfur (S), and chlorine (Cl). The carbon content remains high, reflecting the organic decomposition process increasingly developing with increasing temperature. In addition, inorganic elements such as potassium and calcium remain consistent, indicating that these minerals persist after heating. This analysis is essential to understand how high temperatures affect the chemical composition and morphology of EFB, which determines the quality and potential of EFB as an alternative fuel in the form of biocoke.



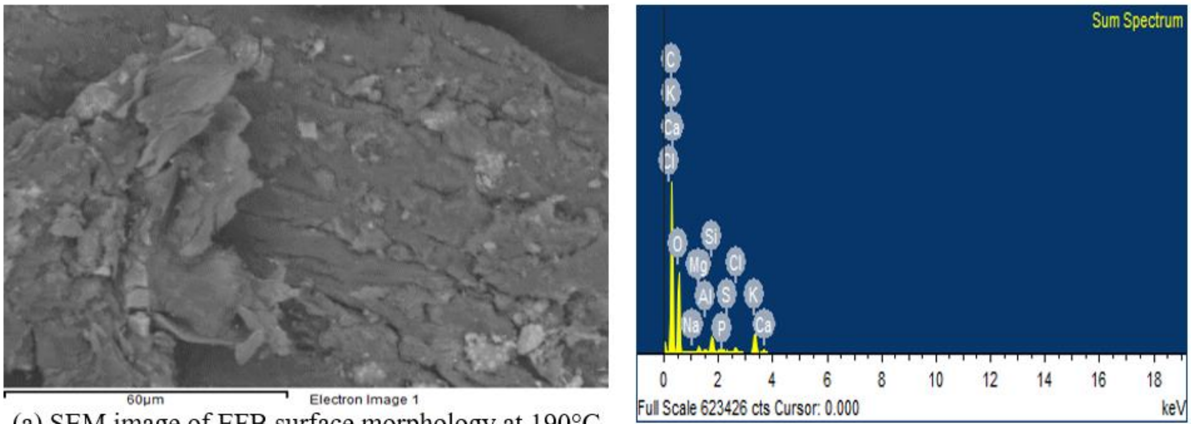
(a) SEM image of EFB surface morphology at 180°C



(b) EDS spectrum of EFB elements at 180°C

Fig. 5. SEM image and EDS spectrum of EFB sample at 180°C

Fig. 6 shows the results of surface morphology analysis and element content of EFB (Empty Fruit Bunch) samples after heating at 190°C using SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy) methods. **Fig. (a)**, the SEM image shows the surface structure of EFB, which has undergone further changes compared to the previous temperature. A fibre layer is starting to peel off, cracks are increasingly apparent, and the surface is rougher due to increasingly intensive thermal degradation. Increasing the temperature to 190°C causes damage to the lignocellulose structure, resulting in small fragments being released from the surface, indicating the increasingly widespread thermal decomposition process of biomass. **Fig. (b)**, the EDS spectrum illustrates the elements' distribution in the EFB sample at 190°C. The main elements detected are still dominated by carbon (C), oxygen (O), potassium (K), calcium (Ca), silicon (Si), magnesium (Mg), phosphorus (P), sodium (Na), sulfur (S), chlorine (Cl), and aluminium (Al). This temperature increase tends to increase the relative proportion of carbon due to further carbonization, while mineral elements remain and are evenly distributed on the surface. These data provide important insights into the stability of inorganic elements during the heating process and the potential for improving the energy quality of EFB-processed biocoke at higher temperatures while also assisting in evaluating the characteristics of ash that may be formed during combustion.



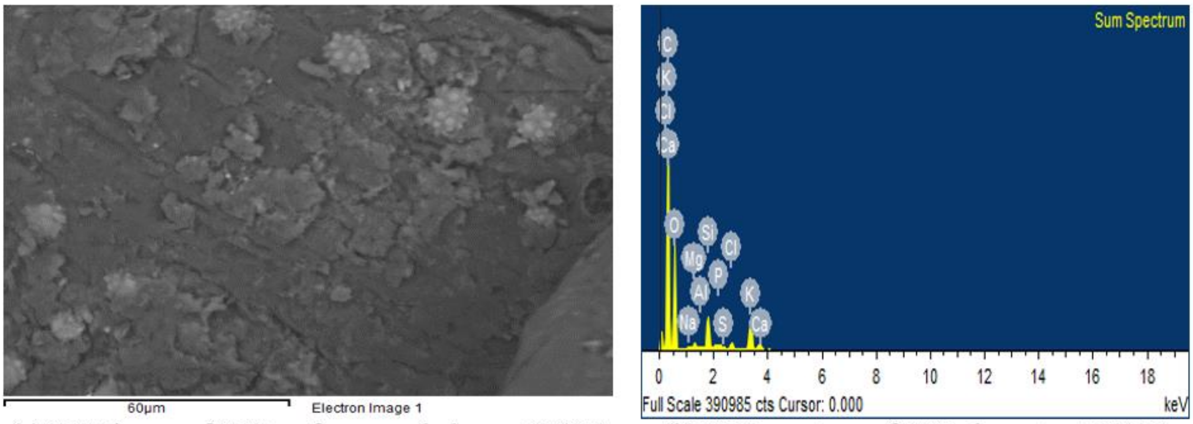
(a) SEM image of EFB surface morphology at 190°C

(b) EDS spectrum of EFB elements at 190°C

Fig. 6. SEM image and EDS spectrum of EFB sample at 190°C

Fig. 7 shows the results of surface analysis and elemental composition of the EFB (Empty Fruit Bunch) sample after heating at 200°C using the SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy) methods. **Fig. (a)**, the SEM image shows that the EFB surface has experienced more significant damage than the previous temperature. There is a rougher surface with many small particles attached, indicating the beginning of the advanced carbonization process. The fibre structure appears increasingly fragile and begins to lose its original shape, indicating that the temperature of 200°C accelerates the decomposition process of organic material in the EFB. **Fig. (b)**, the results of the EDS spectrum describe the composition of the elements contained in the sample after heating at 200°C. The main components that are still detected are carbon (C), oxygen (O), potassium (K), calcium (Ca), silicon (Si), magnesium (Mg), phosphorus (P), sodium (Na), sulfur (S), chlorine (Cl), and aluminium (Al). At this temperature, the increase in carbon proportion is more evident due to the mild pyrolysis process, which causes a reduction in volatile content and increases solid carbon residue. Meanwhile, other mineral elements remain stable and are essential in determining the ash properties and the quality of the solid fuel produced. These results illustrate that a temperature of 200°C is a critical stage in modifying the structure and chemical composition of EFB biomass before reaching complete carbonization.

Based on the results of SEM and EDS analysis at a temperature range of 150°C to 200°C, there are changes in the surface morphology and elemental composition of the EFB (Empty Fruit Bunch) sample as the heating temperature increases. At low temperatures such as 150°C, the surface structure of the EFB is still relatively intact with the presence of fine fibres. In comparison, at a temperature of 200°C, the surface becomes rougher, more brittle, and shows signs of initial carbonization with an apparent reduction in fibre shape. In addition, increasing temperature also affects the distribution of elements, where the carbon (C) content increases. In contrast, volatile components such as oxygen (O) tend to decrease due to the dehydration and devolatilization processes. These changes indicate that the heating temperature plays an essential role in the thermal transformation process of biomass into biocarbon and affects the physical and chemical properties of the resulting biomass.



(a) SEM image of EFB surface morphology at 200°C (b) EDS spectrum of EFB elements at 200°C

Fig. 7. SEM image and EDS spectrum of EFB sample at 200°C

The novelty of this study lies in the comprehensive analysis of the effect of gradual heating temperature on the morphological characteristics and chemical elements of EFB biomass using a combination of SEM-EDS approaches. This study provides a detailed description of the initial critical temperature of carbonization in EFB before entering the complete pyrolysis process, which is rarely explicitly discussed in previous studies. By knowing the changes in structure and element composition at certain temperatures, this study contributes to the optimization of the biomass heating process temperature to produce high-quality biocarbon with a more dominant carbon content and better material stability so that it can be optimally utilized as an environmentally friendly alternative fuel or other carbon materials.

4. Conclusion

Based on the results of surface morphology analysis and element content using SEM-EDS on EFB (Empty Fruit Bunch) biomass heated at temperatures of 150°C to 200°C, it is known that increasing temperature has a significant effect on changes in the physical structure and chemical composition of biomass. At a temperature of 150°C, the EFB surface still looks solid with a reasonably well-maintained fibre structure but begins to experience minor damage. As the temperature increases to 200°C, the surface morphology worsens, cracks, and shows early signs of carbonization. The findings show that the carbon (C) content rises from 37.28% at 150°C to 44.51% at 200°C. The oxygen (O) content decreases from 35.24% to 30.41% at the same temperature, indicating the devolatilization process and release of volatile compounds. In addition, other elements such as potassium (K), calcium (Ca), and silicon (Si) also fluctuate with increasing temperature but remain in relatively small amounts. An important finding in this study is that the temperature of 200°C is a potential starting point for significant changes in EFB biomass before entering the complete carbonization process, marked by an increase in carbon content of 19.4% compared to the lowest temperature and a drastic decrease in oxygen of 13.7%. Thus, this temperature can be recommended as the initial stage of the EFB biomass processing process towards better quality biocarbon. This study also provides a new contribution to the study of palm biomass characterization with a low-temperature stepwise approach, which has rarely been explored in detail in previous studies. These findings can be used to develop sustainable thermal processes to produce renewable energy based on biomass waste.

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