

Case Report

Towards low-cost and sustainable biochar production based on empty fruit bunch: Effect of pelletization, microwave power, residence time, and mass on biochar quality for commercial approach

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ABSTRACT

Biochar produced from biomass based Empty Fruit Bunches (EFB) pyrolysis presents an alternative energy source. Biomass through the pelletization process will produce bio-pellets with higher density, while microwave pyrolysis achieves better energy efficiency and cost effectiveness. This two-step process will produce a product with a longer combustion duration and higher thermochemical conversion efficiency. This study aims to utilize EFB waste through a two-step process (pelletization and microwave pyrolysis) to produce biochar as an alternative renewable energy approaching commercial standards. EFB bio-pellets with a mass of 80, 100, and 120 g were pyrolyzed in a microwave pyrolysis reactor with the power of 450, 600, and 850 W, and in time variations of 20, 30, and 40 min. The results showed that 850 W power and 80 g mass were the optimal conditions to produce biochar with the highest heating value (26.73 MJ/kg), moisture content (0.48 %) volatile matter content (12.56 %), ash content (22.33 %), and fixed carbon (64.62 %). The SEM-EDX analysis indicated that the biochar product showed an increase in carbon (C) content up to 67.4 %. It is expected that combination of the pelletization process and microwave assisted pyrolysis could become a new alternative for producing biochar that could be commercialized. This research was carried out to address the problem of accumulating EFB in palm oil mills and generating renewable energy from waste.

1. Introduction

The high consumption of energy resources and serious environmental pollution around the world, it is necessary to approach these problems. One of which is to convert biomass into clean energy [1]. Biomass is an abundant source of renewable energy, serving as an eco-friendly fuel alternative through various biochemical and thermochemical conversions [2]. Thermochemical conversion techniques include torrefaction, combustion, gasification, and pyrolysis [3].

One of the products that can be obtained from biomass conversion techniques for various purposes via thermochemistry is biochar [4]. Biochar is a porous material consisting of carbon-rich solids obtained from the thermal degradation of biomass under oxygen-limited

conditions [5]. Biochar applications include water purification, gas adsorption, soil remediation, microbial fermentation, energy storage, catalytic fields, and electrochemistry [1]. Large quantities of biomass can be processed in one or two steps with relatively high yields of 20–40 %. Biomass compositions potentially suitable for biochar include lignocellulosic-rich biomass with carbon, hydrogen, nitrogen, and sulfur components like coal, such as Empty Fruit Bunches (EFB) biomass [4,6,7].

The EFB of palm oil is the primary waste product of the palm oil processing industry [8]. EFB is estimated around 23 % of the total fresh fruit bunches processed [9]. Every ton of EFB contains nutrients such as nitrogen of 1.5 %, phosphorus of 0.5 %, potassium of 7.3 %, and magnesium of 0.9 %, which can be used as a substitute fertilizer material for oil

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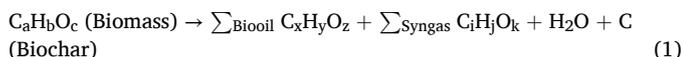
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palm plants [10]. EFB waste remains massively underutilized. So far, only about 20–25 % of the EFB waste is utilized as soil mulch or sometimes turned into compost, while the rest is left to accumulate. If the accumulation is left unchecked, empty fruit bunches of oil palm can lead to waste management issues [11]. Additionally, there are weaknesses in the physical and chemical characteristics of EFB, such as high moisture and oxygen content, lower fuel properties, large size, and heterogeneous size, which have limited its potential for direct use as fuel for thermal conversion processes [12,13]. One promising option to improve the physical and fuel properties and expand its use and marketability as fuel is through the pelletization [14,15].

Pelletization involves the initial treatment of raw materials to reduce size and moisture and compress biomass into fuel with higher and more uniform density using flat or ring pellet molds [16]. EFB pellets are one of the semi-finished products from the processing of solid EFB waste. EFB pellets provide advantages in industrial waste management, and the biochar produced from EFB pellets improves solid fuel properties compared to its raw material, resulting in better combustion efficiency, ease of handling, storage, logistics, and positive environmental impact. The pelletization process can increase the density of EFB pellets to $>650 \text{ kg}\cdot\text{m}^{-3}$, significantly better than raw feed and powder materials ranging from 62 to $152 \text{ kg}\cdot\text{m}^{-3}$ [13,17]. The rapid increase in EFB waste in Indonesia, limited landfill locations, and the considerable potential of EFB pellets as a renewable energy source has spurred interest in converting this waste material into biochar as a solid fuel using pyrolysis processes [18,19]. Pyrolysis is a thermochemical process in which biomass undergoes thermal shrinkage in its chemical constituents with an inert stoichiometric oxygen atmosphere. Typical pyrolysis reactions are as Eq. (1).



Based on the heating mechanism, pyrolysis is divided into two types, namely microwave pyrolysis and conventional pyrolysis. Conventional pyrolysis is part of convection and conduction heating [20]. Conventional pyrolysis has an obstacle to the moisture level of the raw material, which results in limited heat transfer due to the heat transfer that propagates from the wall surface to the material depending on the thermal diffusion properties of the material and the evaporation of water vapor [18,21]. Microwave pyrolysis is a process of heating raw materials by converting electromagnetic energy into molecular kinetic energy through a radiation process. The dielectric motion in its molecules uses an alternating electric field. It generates microwaves that induce heat at the molecular level and transfer heat from the inside to the irradiated surface of the biomass [22,23].

According to Said et al. (2022), the power generated from microwave pyrolysis comes from the high temperature to produce biochar yield because the energy supplied to the biomass can enlarge the biochar energy during the primary pyrolysis reaction changes to the secondary pyrolysis reaction, where the change causes the formation of liquid and gas products [24]. Other advantages of microwave pyrolysis are more practicality, high energy efficiency, and cost effectiveness [25]. Biochar produced from microwave pyrolysis is based on the high surface area, and high carbon content in biochar materials. Biochar products have better thermal characteristics and stability compared to other bio-energy products [26]. The selection of microwave pyrolysis conditions in this study is based on the further development of previous research. The pyrolysis power was chosen based on the study by Antunes et al. (2017), which utilized power of 600 W with biosolids as the feedstock [27]. The reaction time was selected by referring to the study by Hanafi et al. (2024), which employed reaction times of 20, 30, and 40 min using EFB as the feedstock in microwave-assisted pyrolysis with aluminum mesh [28]. The sample mass conditions were based on the study by Bhatta et al. (2018), in which a sample mass of 80 g was used with sewage biosolid as the feedstock [23]. In previous studies, the

pyrolysis process of empty fruit bunches has been carried out, both conventionally using a furnace and also using a microwave [29,30]. The biochar produced from conventional pyrolysis generally has a fairly high ash content due to the high heating temperature and longer heating time, besides that the heating that occurs is often uneven, because it only decomposes part of the raw material, while microwave assisted pyrolysis has a higher effectiveness because it can reduce pyrolysis time and can improve the quality of pyrolysis products due to more even heat distribution. Therefore, further research was conducted by combining microwave assisted pyrolysis with a pelletization process to produce products with higher thermochemical conversion efficiency.

It is necessary to process EFB into biochar using the pyrolysis process. This study aims to utilize EFB waste through a two-step process (pelletization and microwave pyrolysis) to produce biochar as an alternative renewable energy approaching commercial standards. The novelty of this study are the use of EFB pelletization processes and microwave assisted pyrolysis as the one option in the step of upgrading conventional processes to the commercial level to handle large amounts of EFB biomass. Therefore, further research was carried out on biochar production from EFB bio-pellet via microwave assisted pyrolysis. This research will look at the influence of several conditions and variations in the parameters of microwave power, microwave residence time, and mass of the EFB bio-pellet used.

2. Materials and methods

2.1. Materials

Empty Fruit Bunch (EFB) as raw material to produce EFB bio-pellets are supplied by one of the palm oil mills in Sumatera Utara, Indonesia. The tools needed in this research are a series of microwave assisted pyrolysis units, erlenmeyer, digital balance, beaker glass, stopwatch, desiccator, oven, and furnace for analytical purposes. Analysis of high heating value using a bomb calorimeter was carried out at the Laboratorium Motor Bakar, Universitas Sumatera Utara, Medan, Indonesia. Scanning Electron Microscope - Energy Dispersive X-Ray (SEM-EDX) analysis was carried out at the Laboratorium Mikroskop Elektron, Universitas Syiah Kuala, Banda Aceh, Indonesia. Fourier Transform Infra-Red (FTIR) analysis was conducted at UPT Laboratorium Terpadu dan Sentra Inovasi Teknologi, Universitas Lampung, Lampung, Indonesia. Gas Chromatography - Mass Spectrometry (GC-MS) analysis were carried out at the Laboratorium Kimia Organik, Universitas Gajah Mada, Yogyakarta, Indonesia.

2.2. Pelletization process

EFB was chopped and grounded using a ball mill until it was in the form of powder with a size of 100–150 μm (100 mesh), then dried in an oven at 110 $^{\circ}\text{C}$, wrapped with plastic, and stored in sacks so that the moisture content was maintained during the storage process until further usage. Pelletization was carried out using a pelletizer. In the pelletization process the sample was inserted into the press hole (diameter of 7 mm and length 4.5 cm formed from a plate measuring 15 \times 15 cm with a thickness of 4.5 cm). The shape of the pelletizer can be

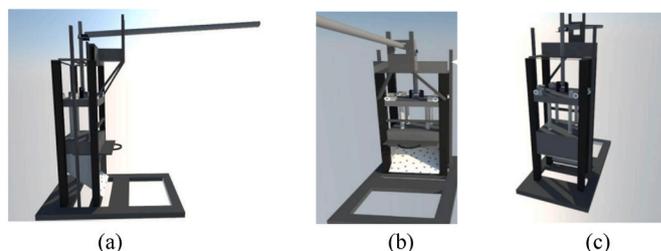


Fig. 1. Pelletizer (a) side view, (b) front view, (c) back view.

seen in Fig. 1 (a), (b), and (c).

Pelletization was done by pressing the press hole by four pipes as st. round bar with a diameter of 7 mm and a length of 12 cm, which is then moved up and down with the help of handle and bearings, with a width 5/8 inches. Then, pressed the hole along with the pipe as st. round bar, simultaneously. The output (EFB bio-pellets) from this tool is held by a plate measuring 15 × 15 cm with a thickness of 0.5 cm, which is located below the press hole plate. After completion, the press hole is pressed again by the pipe as st. round bar with the help of a handle so that the EFB bio-pellets that have been formed come out, then cut to a size of 1 cm and put into a plastic sample. The characteristics of empty fruit bunch and EFB bio-pellets are listed in Table 1.

2.3. Experimental setup

Pyrolysis was carried out in a customized microwave reactor that can be seen in Fig. 2, which consists of a microwave as a pyrolysis reactor, pipe, two container boxes, consisted of a cold-water reservoir and a cold-water tank for the condensation process, gas trap, and gas bag to store incondensable gas, digital thermometer placed on top of the microwave for temperature display, pipe as a gas outlet, and pump to pump water from the cold water tank to the cold water reservoir.

Nitrogen gas tank that stores nitrogen gas that will be flowed into the microwave to remove oxygen in the reactor. The specifications of the microwave used in this research are located in Table 2.

2.4. Pyrolysis process

Prior to the pyrolysis process, EFB bio-pellets were dried in an oven at 110 °C. Microwave assisted pyrolysis was carried out by putting the dried EFB bio-pellets into a microwave to be pyrolyzed by microwaves. Nitrogen was flowed into the pyrolysis reactor at a rate of 0.5 L/min before the pyrolysis process to maintain oxygen poor environments.

The pyrolysis process was carried out using a microwave device (Samsung) with a frequency of 220 V/50 Hz. EFB bio-pellet raw material with a mass of 80 g was pyrolyzed with 450 W power for 20 min to complete the pyrolysis process. Then, after the pyrolysis process was finished, biochar will be placed in a desiccator for 30 min and then weighed with a digital balance, pyrolysis process will then continue for the next variations used for the pyrolysis process, with variations in the mass of raw materials of 100 g and 120 g, with 600 W and 850 W power, and the addition of variations in residence time of 30 and 40 min. Each run of pyrolysis condition variations was repeated three times. This process is carried out to maximize the pyrolysis process and to see the best results from several variations of processing raw materials into biochar.

In this study, the optimal pyrolysis conditions were determined by examining the characteristics of the produced biochar, such as moisture content, volatile matter content, ash content, fixed carbon, high heating value, and carbon content. From the overall analysis, the pyrolysis conditions that resulted in biochar with the best characteristics will be selected, so as to enhance the commercial value of the biochar.

Fig. 3 shows the scheme of the biochar production process from pelletization until microwave assisted pyrolysis process. Biochar yield

Table 1
Characteristic of EFB and EFB bio-pellet.

Parameter	Unit	Result	
		EFB	EFB Bio-pellet
Moisture content	%	50.730	9.886
Volatile matter content	%	91.463	82.088
Ash Content	%	2.082	3.581
Fixed carbon	%	6.453	15.601
Bulk density	g/cm ³	0.201	0.646
High heating value	J/g	13,752	19,411

was determined using data from the mass of EFB bio-pellets before and after pyrolysis, and then entered into Eq. (2).

$$\text{Biochar yield} = \frac{\text{weight of char}}{\text{weight of pellet}} \times 100\% \quad (2)$$

2.5. Characterization and analyses

Proximate analysis is carried out to see the characteristics of the biochar produced. The analysis is carried out to measure moisture content, volatile matter content, ash content, fixed carbon and high heating value (HHV). Analysis of moisture content in biochar is carried out in accordance with ASTM D 1762-84, using an oven at 105 °C. Eq. (3) is used to calculate the moisture content value based on the data obtained.

$$\% \text{ Moisture Content} = \frac{\text{Char mass} - \text{Char mass after drying}}{\text{Char mass}} \times 100\% \quad (3)$$

Volatile matter content (VMC) analysis on biochar is carried out in accordance with ISO 562-1981 methodology. This analysis uses a heating process, in the furnace for 7 min at 900 °C. The sample used is biochar that has been dried from the moisture content analysis process. The data obtained is entered into Eq. (4) below to find the value of volatile matter content.

$$\% \text{ VMC} = \frac{100 (\text{char mass} - \text{residue mass})}{\text{char mass} - \text{empty lid mass}} - M \quad (4)$$

The procedure for analyzing the ash content of biochar was carried out by adopting the methodology for determining ash content according to ASTM D 1762-84. The analysis process was carried out by heating at 750 °C, for 6 h. The data that has been obtained is then entered into the Eq. (5) to find the value of ash content.

$$\% \text{ Ash Content} = \frac{\text{Residue mass}}{\text{Char mass after drying}} \times 100\% \quad (5)$$

The process of analyzing fixed carbon in biochar products is carried out in accordance with ASTM D 1762-84. Data from moisture content (MC) analysis, volatile matter content (VMC), and ash content of biochar products are needed for fixed carbon analysis. After all the data is obtained, it is entered into Eq. (6) to get the fixed carbon value of biochar products.

$$\% \text{ Fixed Carbon} = 100\% - (\text{MC} + \text{VMC} + \text{Ash Content}) \quad (6)$$

High heating value analysis is carried out to see the energy output produced by biochar when heated. From the high heating value analysis can be found out the potential of biochar products that have been produced to become fuel. In this study, the high heating value analysis of biochar products was carried out using a bomb calorimeter. The mass of the sample used for testing the high heating value with a bomb calorimeter equipment is 0.15 g. After obtaining the coolant temperature data before (T_1) and after turning on the bomb calorimeter (T_2), the data is entered into the Eq. (7) to get the high heating value of the biochar product.

$$\text{HHV} = (T_2 - T_1 - T_{kp}) \times C_v \quad (7)$$

Where:

T_1 = Coolant temperature before turning on (°C)

T_2 = Coolant temperature after power on (°C)

T_{kp} = Temperature rise due to igniter wire (0.005 °C)

C_v = Specific heat of bomb calorimeter (73,259.6 kJ/kg °C)

Surface analysis of the raw material EFB bio-pellets and biochar products is carried out using a Scanning Electron Microscope magnified 5000 X, and SEM-EDX spectroscopy to analyze the carbon and oxygen content on the surface of the samples. Both samples will be crushed and grounded first before being analyzed by SEM-EDX. Comparison of the

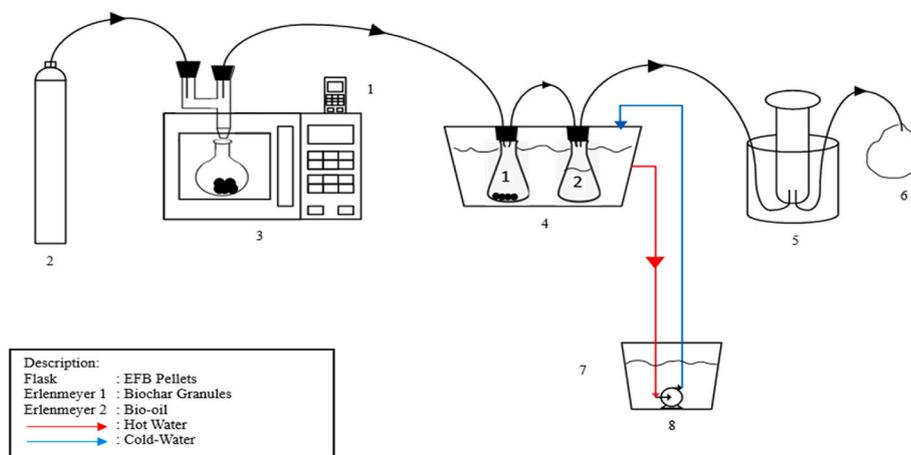


Fig. 2. Customized microwave reactor setup. (1) digital thermometer, (2) nitrogen gas tank, (3) microwave reactor, (4) cold water reservoir, (5) gas trap, (6) gas bag, (7) cold water tank, (8) pump.

Table 2
 Specification of microwave reactor.

Characteristic	Size
Height	29.73 cm
Length	51.7 cm
Width	41 cm
Volume	30 L
Maximum Time	6 h
Maximum Power	1,200 W
Maximum Temperature	2,000 °F

surface functional groups of the EFB bio-pellets sample with the biochar product was analyzed using a Fourier Transform Infra-Red spectrophotometer, where both samples were crushed and analyzed with FTIR in the range of 400–4,000 cm^{-1} . The composition of the bio oil produced from the pyrolysis process was analyzed using Gas Chromatography Mass Spectrometry (GC-MS).

The statistical analysis was conducted using the Statistical Package for the Social Sciences (SPSS, USA) as a post-hoc analysis with the

Bonferroni method to determine the significance of the data obtained from the analysis and to prevent experimental outliers.

3. Result and discussion

3.1. Yield biochar analysis

Fig. 4 showed the biochar yield increase along with the increase in EFB bio-pellet mass, while adding power and pyrolysis time will decrease the biochar yield. The highest biochar yield on the graph was obtained at an EFB bio-pellet mass of 80 g, 450 W power with a pyrolysis time of 40 min with a yield of 87.11 %, while the lowest bio-char yield was obtained at an EFB bio-pellet mass of 80 g, 850 W power with a pyrolysis time of 20 min with a yield of 45.35 %. With the lower temperature and the less heating time, biochar which is decomposed from the heating process is also less, so the biochar yield will increase, and on the contrary, biochar will be more decomposed in longer heating times and higher temperatures, resulted in biochar yield getting smaller [31, 32]. The percentage yield can be seen with the increasing mass and size

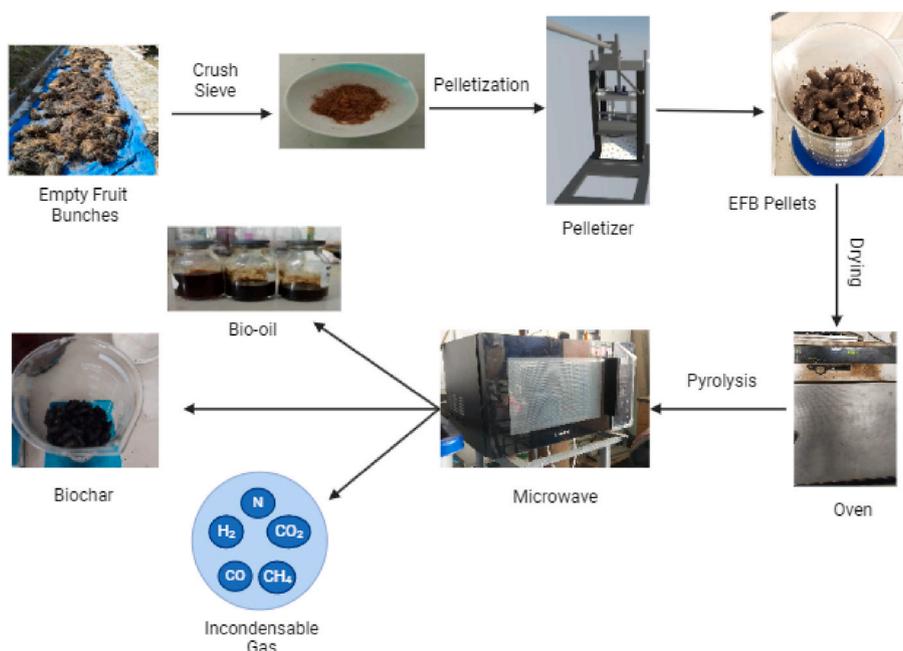


Fig. 3. The schematic route of Biochar production.

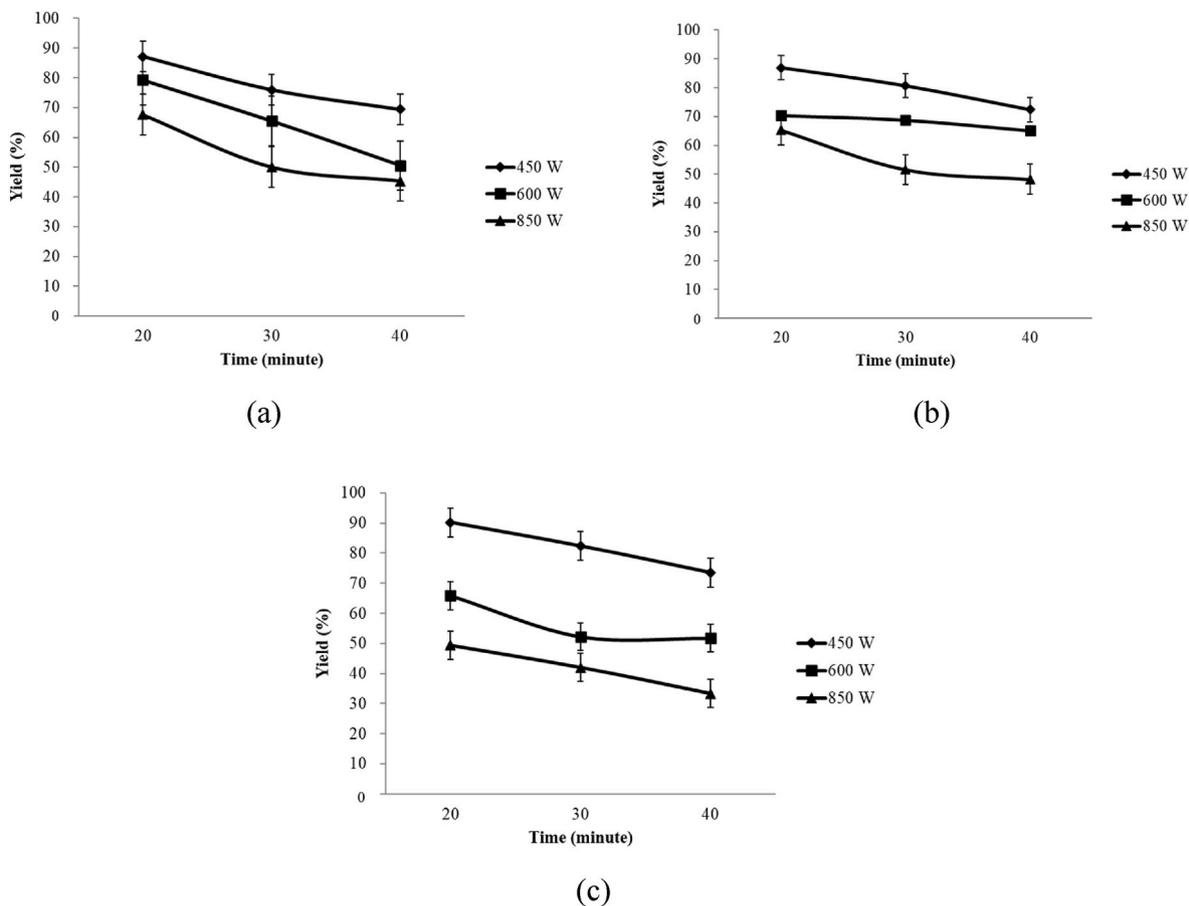


Fig. 4. Biochar yield along time graph (a) 80 g EFB bio-pellet mass, (b) 100 g EFB bio-pellet mass, (c) 120 g EFB bio-pellet mass.

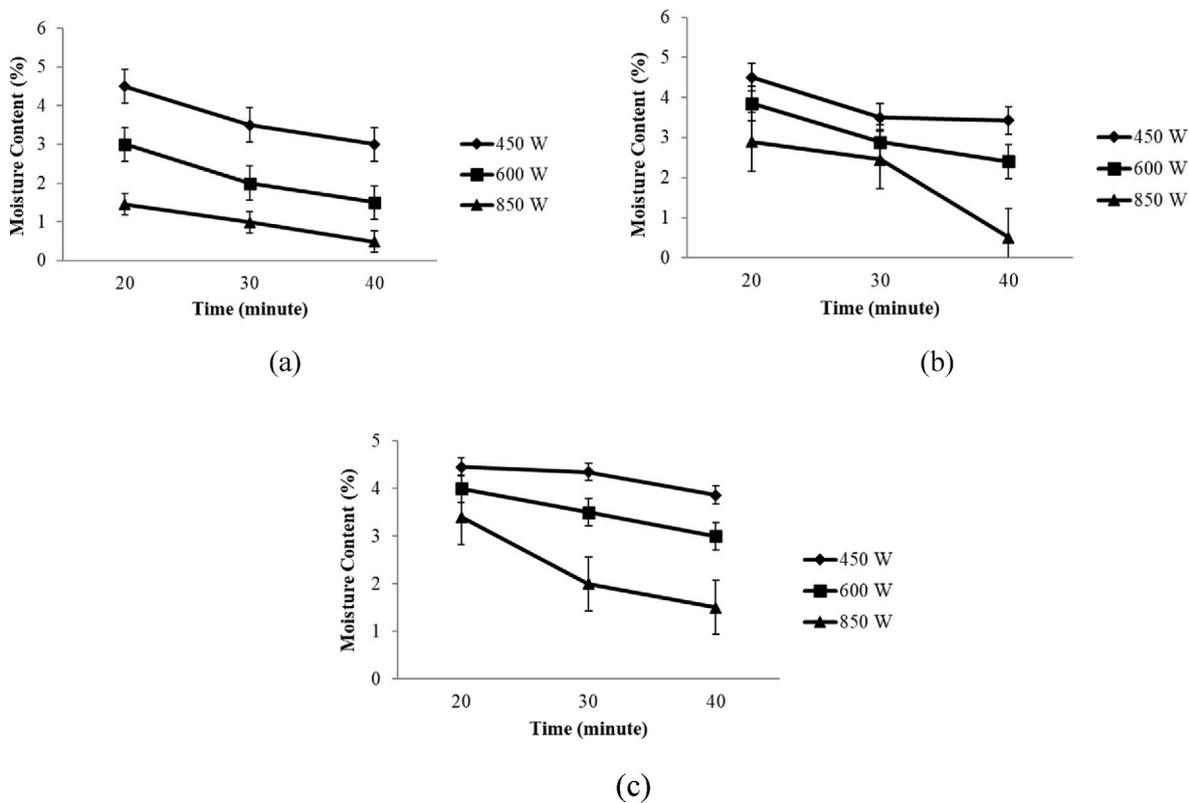


Fig. 5. Moisture content along time graph (a) 80 g EFB bio-pellet mass, (b) 100 g EFB bio-pellet mass, and (c) 120 g EFB bio-pellet mass.

of the EFB bio-pellet, which will be able to increase the biochar yield value [33]. In the process of converting EFB bio-pellets into biochar, the smaller the yield obtained, the better it will be because more volatile substances and moisture content will be evaporated. Therefore, the higher power used in the microwave assisted pyrolysis process was better due to the need for high temperatures so that the decomposition process can be carried out perfectly. However, if the mass or size of the EFB bio-pellets is too large, it will limit the movement of heat energy, thus inhibiting the decomposition process. This is because larger pellets provide less surface area for heat transfer, potentially slowing down the thermal decomposition of biomass [34]. It can be observed that the higher the mass of EFB bio-pellets, the more biochar yield is obtained, which means the decomposition process is still not perfectly conducted.

3.2. Moisture content analysis of biochar

The calculation of moisture content is determined by calculating the loss of sample mass when heated under certain conditions controlled by temperature, time, sample mass, and equipment specifications. The percentage (%) of moisture content can be seen in Figs. 5–6.

Fig. 5 shown that graph of moisture content biochar at various power and pyrolysis time with EFB bio-pellet mass of 80 g. The moisture content value tends to decrease with the addition of power and pyrolysis time. The highest moisture content was obtained at a time of 20 min with an EFB bio-pellet mass of 80 g and 450 W power, which amounted to 4.50 %, while the lowest moisture content was obtained at a time of 40 min with an EFB bio-pellet mass of 80 g and 850 W power, which amounted to 0.48 %. The results of the moisture content analysis are in accordance with research by Li et al. (2016), the moisture content value decreased as the pyrolysis time increased, this was caused by incomplete moisture removal [25]. Heat transfer that propagates from the wall surface to the material can be seen from the evaporation of moisture content [21]. Based on Fig. 5 it can conclude that temperature and heating time have a significant influence in evaporating the moisture content of EFB bio-pellets so that it can be converted into biochar completely. The minimum amount of water content remaining in the biochar indicates that the pyrolysis process has been performed properly. From the analysis that has been carried out, 850 W power is the most suitable power for carrying out the pyrolysis process because it can produce biochar with less moisture content.

Fig. 6 shown the moisture content biochar graph at various EFB bio-pellet mass with 850 W power and 20 min time. The moisture content value tends to increase as the EFB bio-pellet mass increases. The mass of the sample used in the pyrolysis process is also a factor that determines the success of the pyrolysis process. This is caused by the more mass used in the pyrolysis, the more it will hinder the process of heat penetration into the EFB bio-pellet, so the moisture content will not be completely evaporated. From Fig. 6, it can be seen that the mass of 80 g EFB bio-pellets is the suitable mass for carrying out the pyrolysis process in order to obtain biochar with less moisture content value.

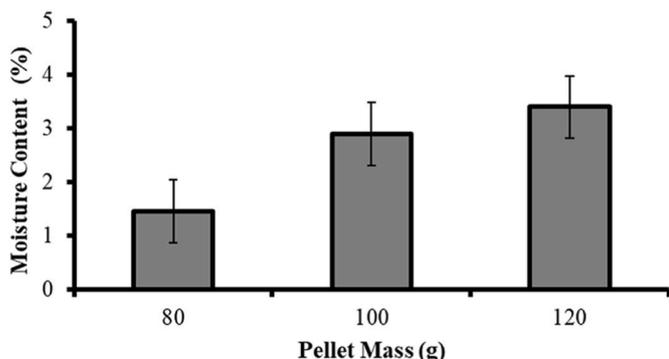


Fig. 6. Moisture content with the variation of EFB bio-pellets mass.

3.3. Volatile matter content analysis of biochar

The calculation of volatile matter content by calculating the volatile content left in the raw material where, the lower the yield, the volatile matter will decrease. Percentage (%) of volatile matter content can be seen in Figs. 7–8.

Fig. 7 shown the graph of volatile matter content biochar at various power and pyrolysis time with EFB Bio-pellet Mass of 120 g. The highest volatile matter content value is obtained at an EFB bio-pellet mass of 120 g, 450 W power, and pyrolysis time of 20 min, which is 30.57 %. In comparison, the lowest volatile matter content is obtained at an EFB bio-pellet mass of 120 g, 850 W power, and pyrolysis time of 40 min, which is 14.15 %. Increasing microwave power will increase the carbonization temperature, resulting in high conversion of biochar into volatile pyrolysis products [35]. The value of volatile matter decreased with increasing power and pyrolysis time. Heat transfer occurs from the outside of the particle to the inside. Therefore the released volatile substances diffuse from the inside to the outside of the particle as the microwave power increases [36]. From the graph above, it can be observed that there are significant differences in the value of volatile matter content in biochar produced with a power of 450 W, compared to the volatile matter content in biochar produced with a power of 600 and 850 W. It can be concluded that, with a power of 450 W the heat energy produced is not enough to completely decompose or convert the volatile substances contained in EFB bio-pellets. Therefore, a greater power is needed, such as 600 and 850 W, so the conversion process can be carried out perfectly.

Fig. 8 shown the graph of volatile matter content biochar at various mass of EFB bio-pellets with 850 W power and 40 min. The volatile matter content value in graph Fig. 8 tends to increase with increasing EFB bio-pellet mass. Increasing carbonization temperature tends to reduce volatile content. However, the size and mass of the EFB bio-pellet will reduce the amount of volatile compounds that evaporate because there are obstructions to heat transfer at a larger mass, so the decomposition process does not occur thoroughly.

3.4. Ash content analysis of biochar

The calculation of ash content is determined by calculating the mineral content that remains after carbon, oxygen, sulfur and water have been separated during combustion. The percentage (%) of ash content can be seen in Fig. 9.

Fig. 9 shown the ash content graph of biochar at various power and pyrolysis time, the highest ash content value was obtained at an EFB bio-pellet mass of 120 g, 850 W power, and pyrolysis time of 40 min, which amounted to 27.72 %, while the lowest ash content in this study was obtained at an EFB bio-pellet mass of 120 g, 450 W power, and pyrolysis time of 20 min which amounted to 11.76 %. From the graph it can be observed that there is an increase in ash content, align with the increase in temperature and time used in the pyrolysis process, and this happens in all pyrolysis variations. Ash content consists of non-organic substances in biomass raw materials that are retained in biochar after the pyrolysis process [21]. The results of the ash content analysis are in accordance with research by Fodah et al. (2021), the ash content value increases as the pyrolysis power and time increase. The higher the microwave power used, the carbonation temperature of the biochar will increase so that the resulting ash content is also higher [37]. During the heating process, biochar produces ash, which increase ash content in biochar after the pyrolysis process [38]. The increase in ash content along with increasing temperature and pyrolysis time occurs due to the evaporation of volatile substances in the EFB bio-pellets, leaving a residue containing a lot of ash content. Therefore the higher the pyrolysis temperature used will resulted higher ash content in biochar.

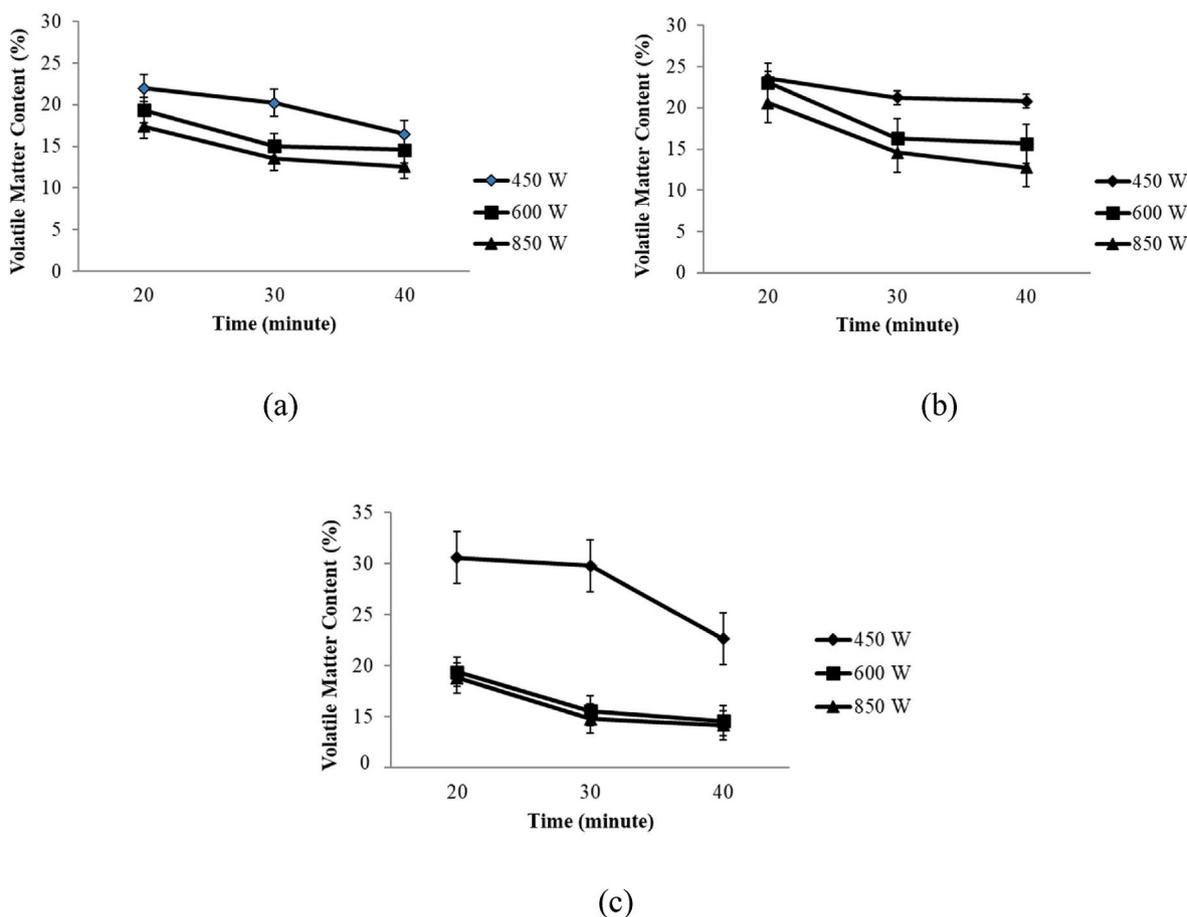


Fig. 7. Volatile matter content along time graph (a) 80 g EFB bio-pellet mass, (b) 100 g EFB bio-pellet mass, (c) 120 g EFB bio-pellet mass.

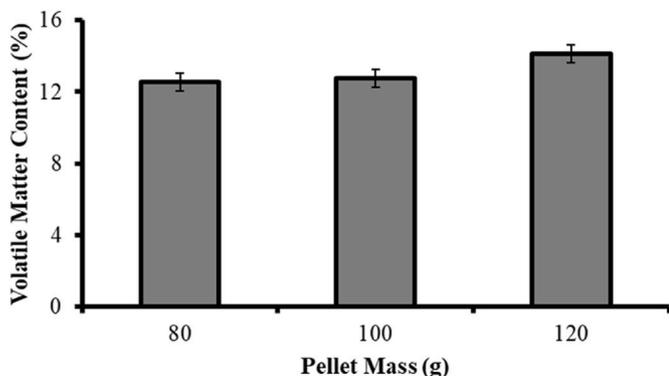


Fig. 8. Volatile matter content with the variation of EFB bio-pellets mass.

3.5. Fixed carbon analysis of biochar

The calculation of fixed carbon is determined by calculating the remaining material after the volatile material is separated. The amount of bound carbon (fixed carbon) can be determined through the difference between the total percentage and the sum of the percentage of moisture content, volatile matter content, and ash content of biochar. The percentage (%) of fixed carbon can be seen in Fig. 10.

Fig. 10 shown graph of fixed carbon biochar at various pyrolysis power and time with EFB bio-pellet mass of 80, 100, and 120 g, where fixed carbon levels fluctuate with increasing power and pyrolysis time. Because the temperature is too high, the pyrolysis process tends to damage the carbon pore walls so that less carbon is formed [25]. Ash

content in biochar can come from inorganic compounds in the form of metal oxide minerals that cannot evaporate and have non-flammable properties during the pyrolysis process. The greater the amount of ash that is decomposed, the more levels of inorganic compounds are contained in biochar so that it can affect the quality of biochar by forming blockages in biochar pores. In addition, it also causes a narrow surface area and affects the value of carbon content bound by fixed carbon [39]. The highest fixed carbon content was obtained at an EFB bio-pellet mass of 80 g, 600 W power, and pyrolysis time of 30 min, which amounted to 71.32 %. The lowest fixed carbon content was obtained at an EFB bio-pellet mass of 100 g, power of 450 W, and time of 40 min, which amounted to 51.36 %. In Fig. 10c it can be seen that there is significant variation between 30 min and 40 min. However, the post-hoc test analysis using Statistical Package for the Social Sciences (SPSS, USA) resulted in a p-value that was not significant, indicating that the data is not an experimental outlier.

Fig. 11 shown the graph of fixed carbon biochar at various mass of EFB bio-pellets with 600 W power and 30 min time showing a comparison of fixed carbon levels at various EFB bio-pellet mass and fluctuations. Fixed carbon levels tend to decrease due to ash content, which has an unequal size, lowering the fixed carbon levels. Extreme temperature also affected some parts of the biochar that can no longer decompose, which causes the results tend to close [25]. The value of fixed carbon biochar that decreases often increases in power from 600 to 850 W causes empty space to cause limited decomposition during the pyrolysis process. The fixed carbon content of biochar first increases progressively and then decreases slightly with increasing temperature or power [39].

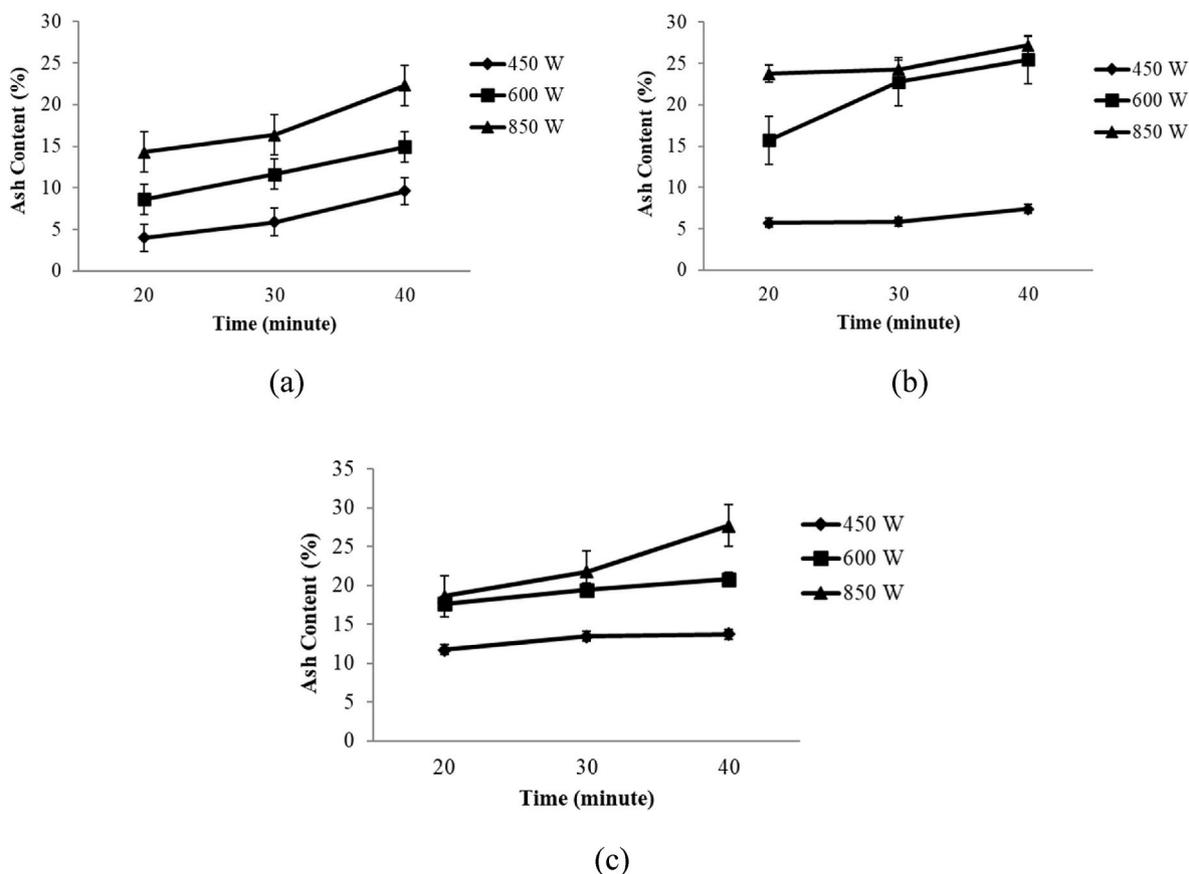


Fig. 9. Ash content along time graph (a) 80 g EFB bio-pellet mass, (b) 100 g EFB bio-pellet mass, (c) 120 g EFB bio-pellet mass.

3.6. High heating value analysis of biochar

The calculation of heating value is determined by the energy content released when the material is burned, including the latent heat contained in water vapor, and is the maximum amount of energy potentially recoverable from a given biomass source. The results of the EFB biochar analysis data on the heating value test continue to increase as the power increases can be seen in Figs. 12 and 13.

Fig. 12 shown the graph of biochar calorific value at various powers and pyrolysis times with a mass of 80 gr EFB bio-pellets shows that the heating value of biochar tends to increase. The heating value is influenced by moisture content and ash content. The lower the moisture content and ash content, the higher the heating value as the pyrolysis temperature increases [40]. Other factors affecting the high heating value are the increase in temperature and reaction rate, causing proximate factors such as moisture content, ash content, volatile matter content and fixed carbon. The influence of the chemical composition derived from lignin has a higher heating value than cellulose. Meanwhile, the presence of accompanying resin in lignin can also affect the heating value produced because lignin is difficult to decompose, and its decomposition almost occurs during the pyrolysis process [41]. The greater the heating value, the slower the combustion speed, because the combustible elements it contains will be less [42]. The highest heating value of biochar in this study was obtained at an EFB bio-pellet mass of 80 g, 850 W power, and pyrolysis time of 40 min, which amounted to 26.73 MJ/kg. In comparison, the lowest was obtained at an EFB bio-pellet mass of 80 g, 450 W power, and pyrolysis time of 20 min, which amounted to 19.41 MJ/kg.

Fig. 13 shown the graph of biochar calorific value at various mass and EFB bio-pellet power with 40 min time, a decrease in heating value as the mass of EFB bio-pellets increases. The addition of mass will affect the amount of carbon produced. A dense and large mass will produce

little carbon [40]. The higher the specific gravity of the fuel, the higher the heating value obtained. Biomass that has a high specific gravity has a high calorific value. When the biomass is combusted, the combustion speed is slower than that of biomass with a lower specific gravity [42].

3.6.1. Results of Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray (EDX)

Scanning Electron Microscopy (SEM) aims to determine the presence of pores on the surface. SEM is done by scanning the electron beam on the surface of biochar. As for obtaining information on two data simultaneously, namely in the form of pore shape data from the sample electron morphology and a number of minerals contained in biochar components. Measurements were also carried out using a combination of Scanning electron microscopy- Energy Dispersive X-Ray (SEM-EDX) spectrophotometer instruments [43].

Fig. 14 shows the results of morphological analysis of EFB bio-pellets used as raw material for biochar production and the resulting biochar products. Fig. 14 (a) refers to EFB bio-pellets and (b) refers to biochar products. Fig. 14 (c) shows the surface of EFB bio-pellets morphology is quite rough and has few pores. The results of SEM characterization on the biochar surface show the shape of the surface morphology, which is mostly still in the form of large pores. This large pore shape can maintain the density of biochar and show the hardness and acid resistance properties of biochar [44]. Pores in a material are known to act as gaps or regular cavities that can bind the two materials to interact, thus providing benefits to each other [45].

Fig. 14 (a) is a visual image of the EFB bio-pellet before undergoing the pyrolysis process, while Fig. 14 (b) is a visual image of the EFB biochar resulting from the microwave pyrolysis process. The results of the analysis can be seen in Fig. 14 (c and d), which can be observed from graphs and tables of the results of the component analysis of biochar and EFB bio-pellets. The results of the SEM-EDX characterization of biochar

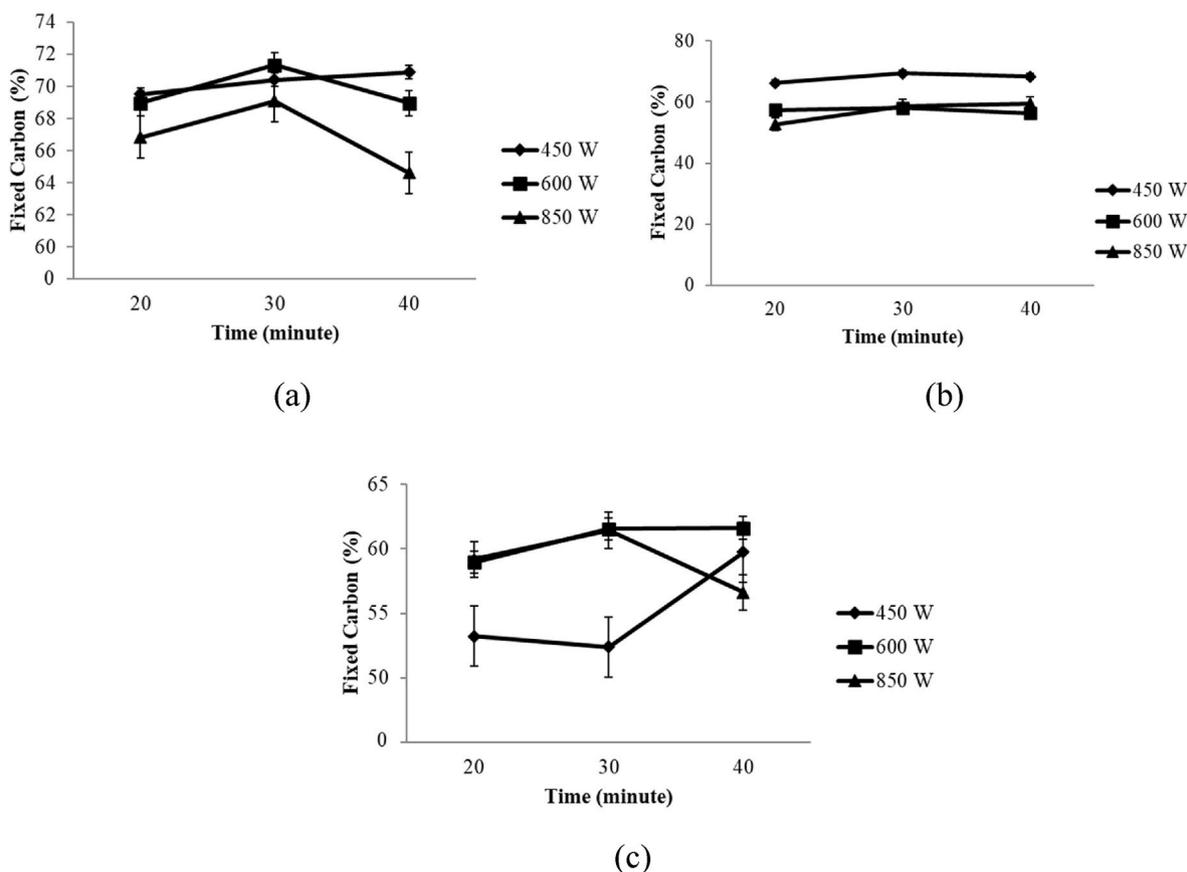


Fig. 10. Fixed carbon content along time graph (a) 80 g EFB bio-pellet mass, (b) 100 g EFB bio-pellet mass, (c) 120 g EFB bio-pellet mass.

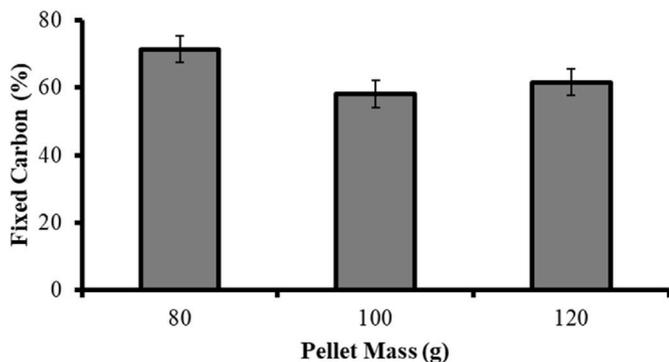


Fig. 11. Fixed carbon content with the variation of EFB bio-pellets mass.

show an irregular structural morphology and no visible pore distribution. The function of the presence of pore distribution in a material is known to act as a gap or cavity that can bind the two materials to interact, thus providing benefits to each other [44]. The existence of pores in the form of micropores found on the surface of biochar and the role of pores in biochar can fill the pores of peat with very high porosity with pores in the form of macro pores that could maintain the density of biochar [46]. The elemental content of the adsorbent is presented in Table 3.

The result of SEM-EDX analysis indicated that the biochar product showed more carbon (C) content than EFB bio-pellets, with an increase in C content of up to 67.4 %. There was a reduction from 38.5 % to 26.7 % for O content. From this analysis, it can be proven that the effectiveness of the pyrolysis process is in increasing the carbon content of the EFB bio-pellet samples. The mineral content coated on the biochar surface

and the organo-mineral composition of the particles are characterized as organic matter with high mineral contribution [47]. It is known that the pyrolysis biochar has more macro pores, which indicates that the pyrolysis process has been successful in increasing the porosity of the biochar product. Moreover, SEM-EDX analysis was carried out to determine the composition of the EFB bio-pellets and biochar samples, from which it can determine the composition of C, O, K, Si, Ca, Al, Mg, and Cl.

In the research by Said et al. (2022), biochar was produced from microwave pyrolysis in an alumina susceptor at 400 °C [24], and in research by Sukiran et al. (2018), biochar was produced from conventional pyrolysis at 500 °C [12]. From Table 4 it can be concluded that the results obtained from this study can be compared with other studies, where the results obtained are good enough and even in some sectors obtained better results, such as in the value of high heating value that can reach the level of 26.73 MJ/kg, the C content with a value of 67.4 %, and lower moisture content of 0.48 %. From these results, it can be concluded that microwave assisted pyrolysis has good efficiency and can even exceed conventional pyrolysis in terms of product quality, as well as in time efficiency. Based on the results of the post-hoc analysis using the Statistical Package for the Social Sciences (SPSS), it is known that p-values for most of the analytical results in various research variables are above 0.050, which meaning insignificant. Summary of experimental runs, variables, analysis, and results can be seen in Table 5.

3.7. Results of the fourier transform infrared (FTIR)

Samples analyzed by Fourier Transform Infrared (FTIR) are variations of biochar produced. The purpose of this analysis is to see the groups that exist in biochar. Changes in sample wave peaks can be seen in Figs. 15 and 16.

The results of the FTIR analysis above can be converted in the form of

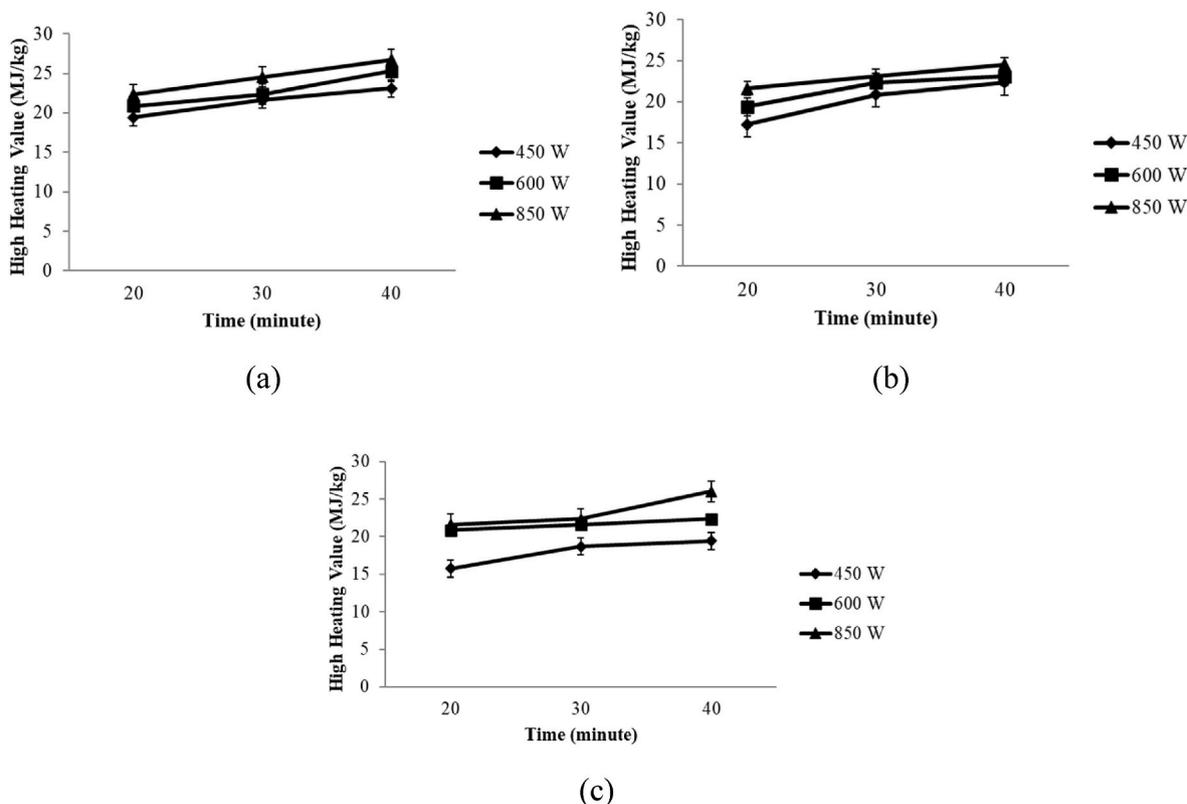


Fig. 12. High heating value along time graph (a) 80 g EFB bio-pellet mass, (b) 100 g EFB bio-pellet mass, (c) 120 g EFB bio-pellet mass.

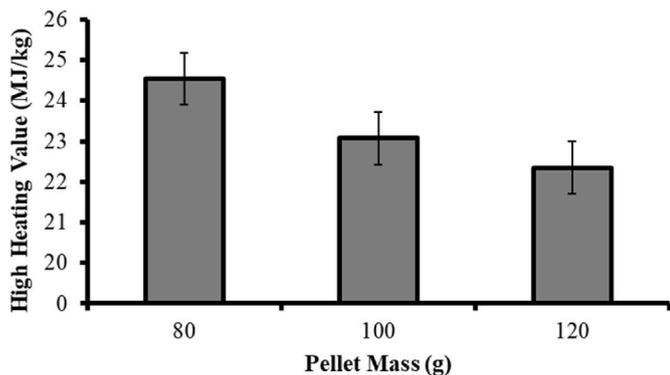


Fig. 13. High heating value with the variation of EFB bio-pellets mass.

percent transmittance spectrum data against wave number [48]. The FTIR value for the sample before pyrolysis in Fig. 15 shows the presence of OH groups and stretching vibrations occurring at the wave number of the first absorption peak $3,287.5 \text{ cm}^{-1}$, and the second absorption peak $2,922.2 \text{ cm}^{-1}$. The second absorption peak wavelength widens until the fourth absorption peak of $1,915.9 \text{ cm}^{-1}$. OH groups come from water, cellulose, phenol, and carboxyl groups [44]. The functional groups of the compounds that play a role in the biochar component are the result of the decomposition of lignin, cellulose, and hemicellulose compounds that have undergone shrinkage during the pyrolysis process to produce C-organic [49]. FTIR results also showed aliphatic C-H groups derived from cellulose, and vibrations occurred at the first absorption peak of $2,102.2 \text{ cm}^{-1}$, increasing at the second absorption peak of $2,102.8 \text{ cm}^{-1}$. The aliphatic group continues to decrease until the fourth absorption peak of $1,416.4 \text{ cm}^{-1}$. The stretching vibration also occurred at wave number $1,319.5 \text{ cm}^{-1}$ as the first absorption peak and decreased at the second absorption peak of $1,028.7 \text{ cm}^{-1}$, indicating the presence of the

C-O group [50].

Fig. 16 showed the difference in the results obtained after the pyrolysis process on biochar products and significant changes in the chemical characteristics occur. The OH group increased at the first absorption peak of $3,615.5 \text{ cm}^{-1}$ and there was a stretching vibration until the fourth absorption peak of $1,915.9 \text{ cm}^{-1}$, and absorption occurred. The absorption of the hydroxy group (O-H) indicates that there is hydrogen bonding both from residual water vapor and hydrocarbon compounds [42]. The pyrolysis process will greatly affect the C-organic produced where the higher the temperature in the combustion process, it will produce biochar with excellent C-organic quality and with high pH [51]. While at wave numbers $3,332.2 \text{ cm}^{-1}$ and $1,915.93 \text{ cm}^{-1}$ showed the absorption of alkane compound functional groups (C-H) stretching of cellulose. There are also aromatic groups shown by vibrations at the wave number of the first absorption peak of $1,566.6 \text{ cm}^{-1}$ and decreased at the third absorption peak of $1,006.4 \text{ cm}^{-1}$ where aromatic groups come from lignin during the pyrolysis process. The C-O group contained in the biochar product shows a decrease in the absorption peak from that shown in Fig. 16. The first absorption peak is 678.4 cm^{-1} and increases at the second absorption peak of 752.9 cm^{-1} [52].

The pyrolysis process significantly influences the organic carbon (C-organic) content produced. Higher combustion temperatures yield biochar with superior C-organic quality, accompanied by an increased pH level [50]. At wavenumbers $2,124.8 \text{ cm}^{-1}$ and $1,915.9 \text{ cm}^{-1}$, peaks indicate the presence of alkane functional groups (C-H) resulting from cellulose stretching. Aromatic groups are also identified, as evidenced by the vibrations at the first absorption peak at $1,566.6 \text{ cm}^{-1}$. This peak is typically associated with C=C double bonds within aromatic structures, which are key components of biochar. This suggests that the material possesses a conjugated or aromatic carbon structure formed during pyrolysis. The intensity of this peak decreases at the third absorption peak at $1,006.4 \text{ cm}^{-1}$, indicating that the aromatic groups originate from lignin.

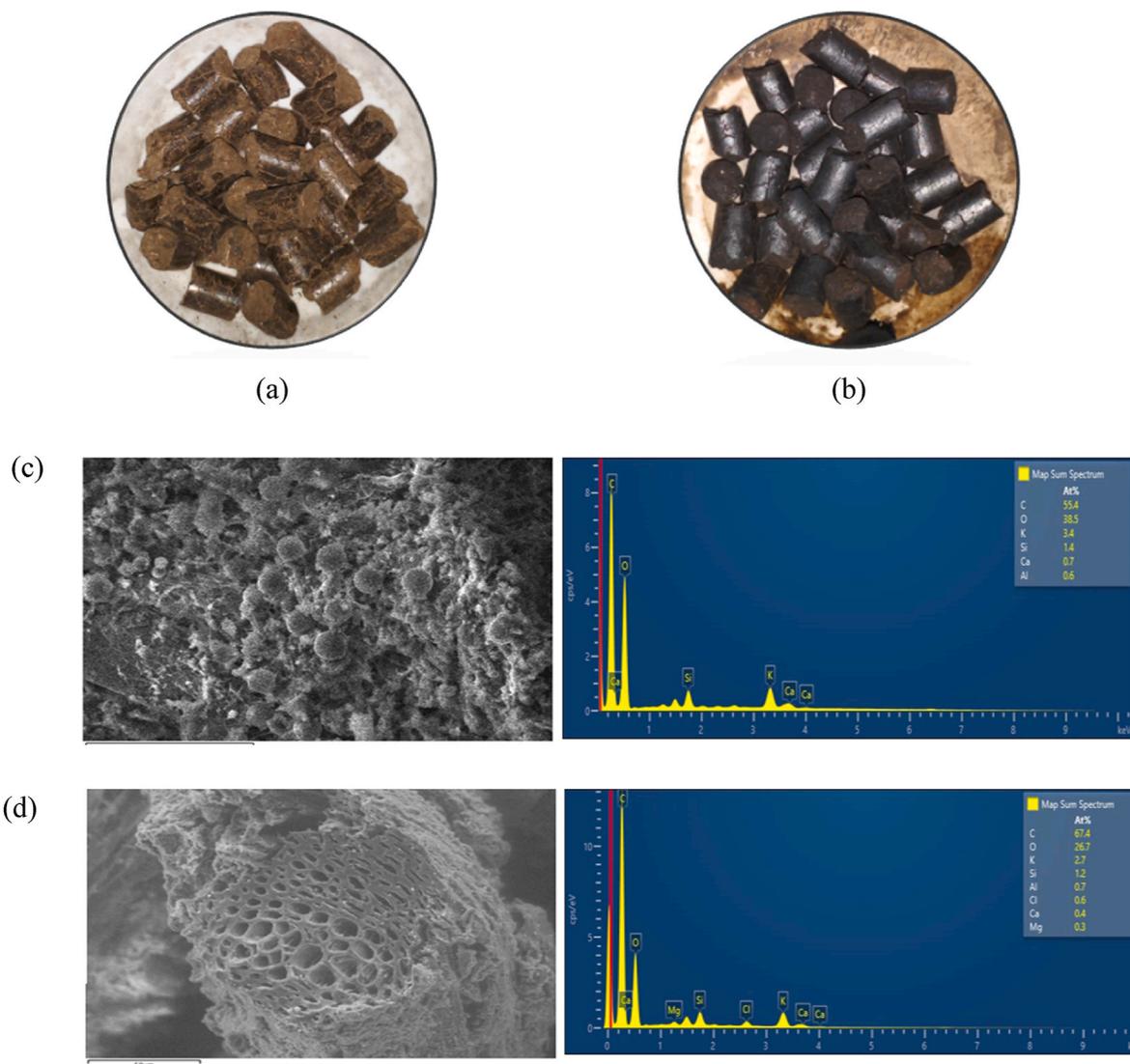


Fig. 14. (a) EFB Bio-pellet before pyrolysis process, (b) EFB biochar after pyrolysis process, (c) SEM result of EFB bio-pellet surface morphology before pyrolysis with 5000 times magnification, (d) SEM result of biochar surface morphology after pyrolysis in microwave at a power of 850 W with 5000 times magnification.

Table 3

EDX analysis results on biochar before and after pyrolysis process at 5000 time magnification.

No	Chemical Compounds	% Atom	
		Before Pyrolysis	After Pyrolysis
1	C	55.4	67.4
2	O	38.5	26.7
3	K	3.4	2.7
4	Si	1.4	1.2
5	Ca	0.7	0.4
6	Al	0.6	0.7
7	Cl	–	0.6
8	Mg	–	0.3

The presence of C-O groups in the biochar product indicate that oxygenated compounds are not completely decomposed during pyrolysis. The first absorption peak at 678.4 cm^{-1} and the subsequent increase at the second absorption peak at 752.9 cm^{-1} [51] suggest the presence of out-of-plane aromatic C-H vibrations. These peaks often indicate substituted aromatic rings, which are common in biochar structures. Overall, this biochar demonstrates potential for use in various environmental and agricultural applications, particularly due to its stable

Table 4

Comparison of biochar characteristics.

Specification	Unit	Present Study Biochar from EFB Bio-pellets	Other Studies Biochar from EFB pellets [24]	Other Studies Biochar from EFB [12]
Proximate Analysis	Moisture	%	0.48	7.13
	Volatile Matter	%	12.56	43.59
	Ash Content	%	4.00	10.64
	Fixed Carbon	%	71.32	38.64
Ultimate Analysis	C	%	67.4	69.69
	O	%	26.7	23.25
General	HHV	MJ/kg	26.73	24.34

Table 5
Summary of experimental runs, parameter, and analysis results.

Run	Power (W)	Mass (g)	Time (min)	Yield (%)	Moisture Content (%)	Volatile Matter Content (%)	Ash Content (%)	Fixed Carbon (%)	High Heating Value (MJ/kg)
1	450	80	20	87.11	4.50	21.99	4.00	69.51	19.41
2			30	76.00	3.50	20.21	5.88	70.41	21.61
3			40	69.43	3.00	16.49	9.62	70.89	23.08
4		100	20	86.90	4.50	23.56	5.71	66.23	17.22
5			30	80.67	3.50	21.24	5.88	69.37	20.88
6			40	72.42	3.43	20.81	7.41	68.35	22.34
7		120	20	90.16	4.46	30.57	11.76	53.21	15.75
8			30	82.35	4.35	29.80	13.46	52.39	18.68
9			40	73.49	3.86	22.61	13.76	59.76	19.41
10	600	80	20	79.25	3.00	19.39	8.65	68.96	20.88
11			30	65.50	2.00	15.03	11.65	71.32	22.34
12			40	50.54	1.50	14.58	14.95	68.96	25.27
13		100	20	70.30	3.85	23.04	15.74	57.38	19.41
14			30	68.73	2.88	16.32	22.77	58.03	22.34
15			40	65.10	2.40	15.68	25.49	56.43	23.08
16		120	20	65.86	4.00	19.39	17.65	58.97	20.88
17			30	52.27	3.50	15.54	19.42	61.54	21.61
18			40	51.73	3.00	14.58	20.79	61.62	22.34
19	850	80	20	67.66	1.46	17.41	14.29	66.85	22.34
20			30	50.04	0.99	13.57	16.35	69.10	24.54
21			40	45.35	0.48	12.56	22.33	64.62	26.73
22		100	20	65.33	2.90	20.60	23.76	52.74	21.61
23			30	51.57	2.45	14.57	24.30	58.68	23.08
24			40	48.20	0.50	12.76	27.18	59.56	24.54
25		120	20	49.45	3.40	18.78	18.63	59.19	21.61
26			30	42.06	2.00	14.78	21.78	61.44	22.34
27			40	33.34	1.50	14.15	27.72	56.63	26.01

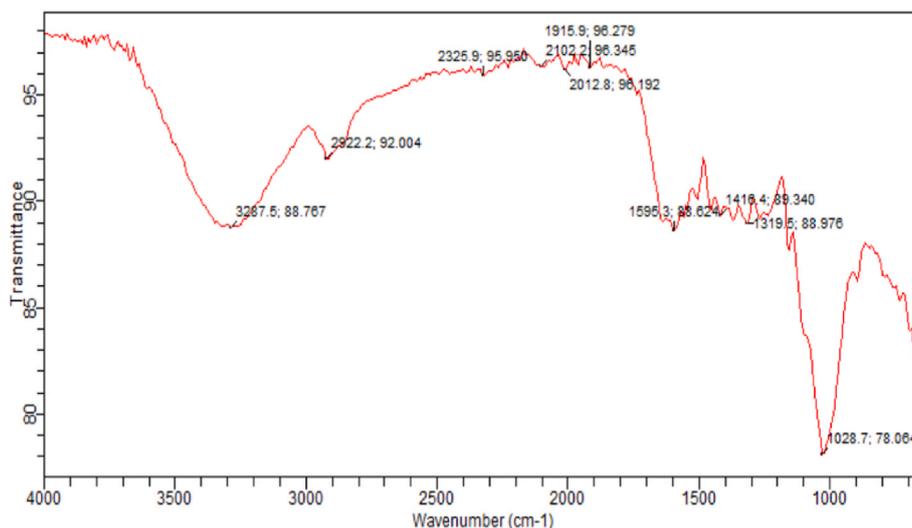


Fig. 15. IR transmittance spectrum of EFB Bio-pellet before pyrolysis process.

aromatic structure and possible adsorption capabilities.

3.8. Results of the gas chromatography - mass spectrometry (GC-MS)

GC-MS analysis was performed on one sample selected based on the bio-oil sample from pyrolysis of EFB bio-pellets of 1 cm size, pyrolysis power of 850 W and pyrolysis time of 40 min.

Fig. 17 showed the chromatogram data from GC-MS analysis of bio-oil. From the data it can be seen that the most constituent components in bio-oil in this study are Phenol (C_6H_6O) as much as 22.66 %, Methyl palmitate ($C_{17}H_{34}O_2$) as much as 19.73 % and Methyl octadec-9-enoate ($C_{19}H_{36}O_2$) as much as 17.05 %. The components decomposed and released from EFB pellets during conversion into bio-oil are rich in phenolic compounds, which often result in biochar that is more polymerized and has a thermally stable carbon structure. The compounds

present in the bio-oil are listed in Table 6.

The presence of other components in bio-oil products, such as phenols, alkane hydrocarbon compounds, and ester compounds, made bio-oil suitable for the production of fuels or other valuable chemical products [53]. Pentadecane ($C_{15}H_{32}$), nonadecane ($C_{19}H_{40}$) and dodecane ($C_{12}H_{26}$), which belong to the class of alkane hydrocarbon compounds, can be used as solvents and can even be applied in producing ionic or nonionic detergents. The presence of ester compounds in bio-oil components such as methyl laurate ($C_{13}H_{26}O_2$), methyl myristate ($C_{15}H_{30}O_2$), methyl palmitate ($C_{17}H_{34}O_2$), and methyl linoleate ($C_{19}H_{34}O_2$) proves that the bio-oil produced in this study can be used as an additive in the food industry as a preservative or in cosmetic equipment. Table 7 shows the percentage of various types of fuel based on the length of the carbon chain in bio-oil.

Based on the standards of the American Petroleum Institute in 2001,

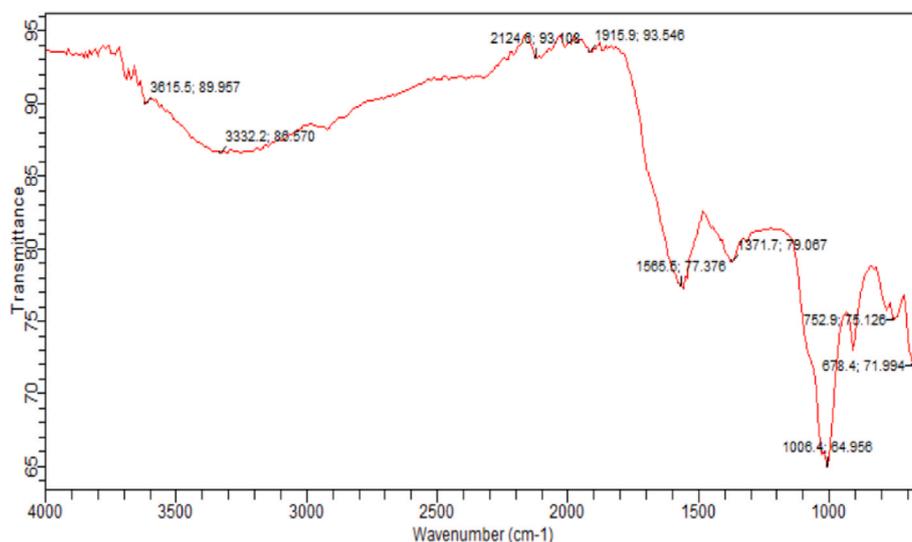


Fig. 16. IR transmittance spectrum of Biochar after pyrolysis process in microwave at a power of 850 W.

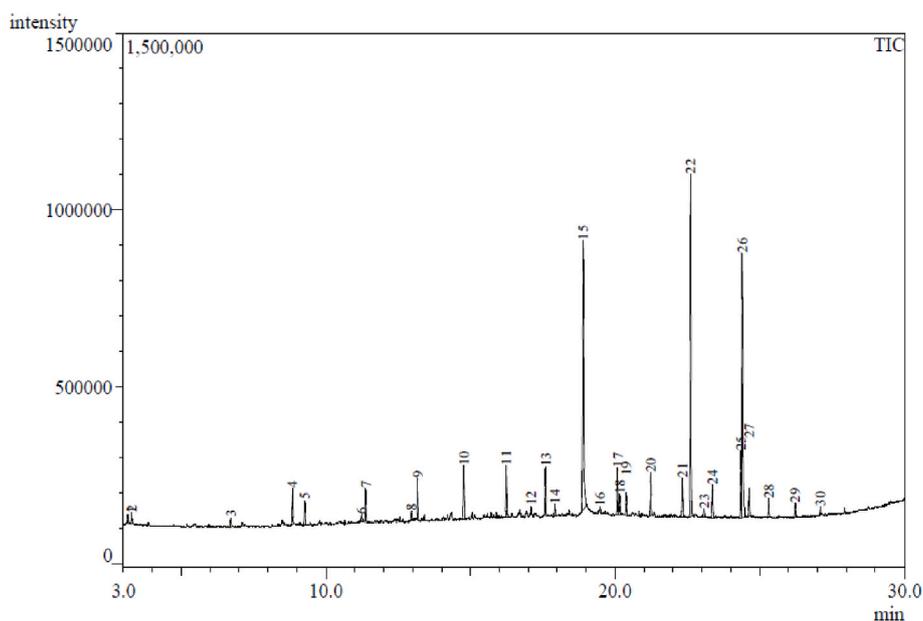


Fig. 17. Chromatogram of GC-MS analysis results of bio-oil.

the bio-oil produced in this study is included in the diesel type because it has the most components in the C_{14} - C_{19} range. Based on the length of the carbon chain, liquid fuels with compounds C_{14} - C_{19} as fuel range compounds and compounds longer than C_{20} as high molecular compounds [55]. The hydrocarbon components contained in liquid fuels will affect several liquid fuel quality parameters such as cetane number, pour point, and dew point, which affecting engine performance [56]. Therefore, liquid fuels with a high fraction indicate low quality [54], otherwise if the compound components contained are close to the range of fuel compounds (fuel range) have better quality.

4. Conclusion

Microwave assisted pyrolysis was carried out with various variables such as mass, power, and time to find the optimal conditions for biochar production from EFB bio-pellets. The use of microwave assisted pyrolysis reduces the duration of pyrolysis time while increasing the efficiency of the pyrolysis process. It can also improve the quality of the

pyrolysis product. From this study, resulted that 80 g mass of EFB bio-pellet using 850 W power in 40 min is the most ideal pyrolysis condition for biochar production, with biochar yield (45.35 %), the biochar produced has the best characteristics, such as the highest heating value of 26.73 MJ/kg, the lowest moisture content (0.48 %) and the lowest volatile matter content (12.56 %), and ash content value (22.33 %). In the results of fixed carbon analysis, the data tended to fluctuate, but the highest fixed carbon value (71.32 %) was obtained, at a power of 600 W, and a pyrolysis time of 30 min. The main focus of this research is to find the pyrolysis conditions that produce biochar with the best characteristics. Although the fact that a higher yield can be achieved by using lower pyrolysis power, this approach may compromise the quality of the biochar in terms of carbon content, moisture content, high heating value, and other characteristics. The result of SEM-EDX analysis indicated that the biochar product showed more carbon (C) content up to 67.4 %. Fourier transform infrared (FTIR) analysis showed differences in the results obtained, where after the pyrolysis process on the biochar product the OH group increased at the first absorption peak of 3,615.5

Table 6

Component compounds contained in bio-oil.

Peak Numbers	Ret. Time (minute)	Compounds	Composition (%) (b/b)
1	3.162	2,2 - dimethoxybutane (C ₆ H ₁₄ O ₂)	0.50
2	3.291	1 - Hydroxy - 2 - butanone (C ₄ H ₈ O ₂)	1.80
3	6.721	Nonane (C ₉ H ₂₀)	1.64
4	8.849	Phenol (C ₆ H ₆ O)	2.07
5	9.277	Dodecane (C ₁₂ H ₂₆)	1.34
6	11.233	2(3H) - Furanone, Dihydro - 4 - Hydroxy (C ₄ H ₆ O ₃)	0.57
7	11.378	Tridecane (C ₁₃ H ₂₈)	1.85
8	12.953	Naphthalene (C ₁₀ H ₈)	0.56
9	13.173	Dodecane (C ₁₂ H ₂₆)	2.15
10	14.772	Pentadecane (C ₁₅ H ₃₂)	3.08
11	16.234	Pentadecane (C ₁₅ H ₃₂)	2.96
12	17.099	Nonadecane (C ₁₉ H ₄₀)	0.45
13	17.597	Pentadecane (C ₁₅ H ₃₂)	2.64
14	17.917	Methyl laurate (C ₁₃ H ₂₆ O ₂)	0.71
15	18.893	Phenol (C ₆ H ₆ O)	22.66
16	19.480	Pentadecane, 2,6,10 - Trimethyl (C ₁₈ H ₃₈)	0.37
17	20.086	Nonadecane (C ₁₉ H ₄₀)	2.67
18	20.161	Phytane (C ₂₀ H ₄₂)	1.29
19	20.384	Methyl myristate (C ₁₅ H ₃₀ O ₂)	1.38
20	21.234	Nonadecane (C ₁₉ H ₄₀)	2.26
21	22.325	Nonadecane (C ₁₉ H ₄₀)	2.09
22	22.607	Methyl palmitate (C ₁₇ H ₃₄ O ₂)	19.73
23	23.071	Isobutyl phthalate (C ₁₆ H ₂ O ₄)	0.52
24	23.366	n-Eicosane (C ₂₀ H ₄₂)	1.87
25	24.350	Methyl linoleate (C ₁₉ H ₃₄ O ₂)	3.97
26	24.400	Methyl octadec - 9 - enoate (C ₁₉ H ₃₆ O ₂)	17.05
27	24.631	Methyl stearate (C ₁₉ H ₃₈ O ₂)	1.70
28	25.311	n - Nonacosane (C ₂₉ H ₆₀)	1.03
29	26.220	n - Tetratetracontane (C ₄₄ H ₉₀)	0.84
30	27.093	n - Nonacosane (C ₂₉ H ₆₀)	0.49
		Total	100

Table 7

Percentage of fuel type in bio-oil based on carbon chain [54].

Fuel Oil	Chemical Formula	Percentage (%)
Gasolin	C ₅ - C ₁₀	29.23
Kerosin	C ₁₁ - C ₁₃	6.05
Diesel	C ₁₄ - C ₁₉	60.87

cm⁻¹ and there was a stretching vibration until the fourth absorption peak of 1,915.9 cm⁻¹. From GC-MS analysis of bio-oil, it is known that the efficiency in process and quality of the product, and also integrating the microwave pyrolysis system with other process to increase the overall efficiency. Microwave assisted pyrolysis is expected to be a new alternative to producing biochar that could be commercialized. Based on the analysis, it was determined that the produced biochar fulfills the required standards and is suitable for commercial use, such as improving soil fertility and serving as a renewable energy source. The commercial approach to biochar produced on a bench scale has found a common ground. This approach is designed to obtain commercial biochar to be used as a solid fuel in various industrial applications, such as use in industrial furnaces for gasification and syngas production, to bio-oil production. Evidently, the main parameters such as high heating value, density, and C/N ratio have met commercial standards. The production of commercial biochar through a combination of pelletization and microwave-assisted EFB pyrolysis processes is expected to be an initial study in integrating more efficient processing of oil palm empty fruit bunches. Further regulations and standards are needed to develop commercial standard biochar on an industrial scale, especially regarding product quality and safety. The selection of raw materials, pre-treatment

stages, frequency, time, and power of the pyrolysis process, reactor design, temperature control, and automation systems must be further highlighted. Although this technology is promising, the initial equipment investment may be higher than traditional pyrolysis technology. However, reduced operating costs from energy efficiency can offset these costs in the long term.

CRedit authorship contribution statement

Rivaldi Sidabutar: Methodology, Conceptualization. **Irvan:** Validation, Supervision, Validation, Supervision. **Bambang Trisakti:** Methodology, Conceptualization. **Noersukma Dwi Gusty:** Formal analysis, Data curation. **Mhd. Rivaldi Syahputra:** Visualization, Data curation. **Bagus Wahyu Saputra:** Visualization, Investigation. **Vikram Alexander:** Formal analysis, Conceptualization. **Hiroyuki Daimon:** Supervision, Methodology. **Peer Mohamed Abdul:** Validation, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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