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Characterization of Porosity and Pore Volume in EFB Samples through Physical and Morphological Parameters

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Abstract
This study analyses the physical and morphological characteristics of oil palm empty fruit bunch (EFB) fibre samples heated at 150°C. Measurements of parameters such as total volume, solid volume, integral volume, and pore volume were carried out to determine the porosity properties of the samples. Based on the measurement results, the total volume of the sample was recorded at 48.28 cm^3, with a solid volume of 10.18 cm^3, an integral volume of 16.47 cm^3, and a pore volume of 38.09 cm^3, resulting in a porosity of 78.90%. The morphological images of the samples support these results by showing significant pore distribution. These findings provide important insights into the structure and potential use of EFB fibres in porous material applications, especially in the context of sustainability and biomass waste utilization. This study also contributes to further understanding of the thermal and physical characteristics of natural materials at high temperatures.

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1. Introduction
Oil palm empty fruit bunches (EFB) are one of the abundant biomass wastes from the palm oil industry. In Indonesia, EFB is produced in large quantities yearly, along with the increasing production of crude palm oil (CPO). EFB is increasingly in demand as an alternative raw material in various industrial applications, mainly because of its sustainable and environmentally friendly nature. In recent decades, research related to EFB has snowballed, especially in terms of using EFB as a porous material for energy storage, absorption, and building materials (Erdiwansyah et al. 2023; Gani et al. 2023a, 2024a, b; Oliveira et al. 2024). The physical characteristics of EFBs, such as porosity and pore volume, play an essential role in their applications in various fields (Parshetti et al. 2013; Zaini et al. 2017; Ruksathamcharoen et al. 2019). Porosity, which describes the ratio between the pore volume and the total volume of a material, is one of the main factors in determining a material's ability to absorb liquids or gases (Lian et al. 2011; Gani et al. 2023b; Sánchez-Mendieta et al. 2024). Significant changes

in the microstructure of EFBs can occur at high temperatures, including increased porosity that allows for broader use in porous material technology applications.

This study was conducted to understand the effect of high temperature on the porosity and pore volume characteristics of EFB. EFB samples were heated at 150°C and then measured for parameters such as total volume, solid volume, integral volume, and pore volume. The measurement results showed that the pore volume reached 38.09 cm^3 with a porosity of 78.90%, indicating that high temperature increases the material's capacity to maintain ample pore space. Research through morphological analysis showing the distribution of pores in EFB samples after heating has recently been reported in the study (Mahidin et al. 2020; Erdiwansyah et al. 2022, 2024). Modern imaging technology can analyse this distribution visually, strengthening the quantitative findings from volume measurements. The images also show the internal structure of the fibres becoming more organized after thermal treatment, which has the potential to increase the structural stability of EFB. Studying thermal modification of natural materials such as EFB is critical in developing new environmentally friendly materials. Many studies have shown that biomass such as EFB can be used as a substitute in various industrial applications, from construction materials to water absorption media or hazardous chemicals (Zakaria et al. 2016; Onoja et al. 2019; Saadon et al. 2022). Heating at a specific temperature can modify the physical and chemical properties of the material, which contributes to improving its performance.

Therefore, this study aims to provide further insight into how thermal treatment of EFB can affect the porosity and pore volume properties. The results can be used to consider broader applications of EFB in high-tech porous materials. This study also contributes significantly to understanding the potential of biomass waste utilization for industrial sustainability. The novelty of this paper lies in the in-depth analysis of the effect of high temperature on the porosity and pore volume of oil palm empty fruit bunch (EFB) fibre, which has not been widely explored in previous studies. This study offers new insights into the physical modifications that occur in EFB when heated at 150°C, especially in increasing porosity up to 78.90% and significant changes in internal structure. In addition, using morphological imaging methods to identify the pore distribution visually provides a new dimension in characterizing biomass-based materials. These results make using EFB as a porous material more beneficial and give us new ways to use it in more prominent areas, like energy storage and cleaning up the environment. These are essential steps toward making biomass waste-based industries more sustainable.

2. Methodology

Fig. 1 shows how natural materials are processed from oil palm fruit bunches to become biocoke fuel products. The process begins with oil palm fruit whose fibres are removed from the bunches (**Fig. 1(a)**). After that, the fibres are cut into smaller parts (**Fig. 1 (b)**). Next, the cut fibres are put into a blender to be ground (**Fig. 3(c)**). The result of this process is a solid biocoke fuel product, as shown in **Fig. 1(d)**, which is used as fuel.

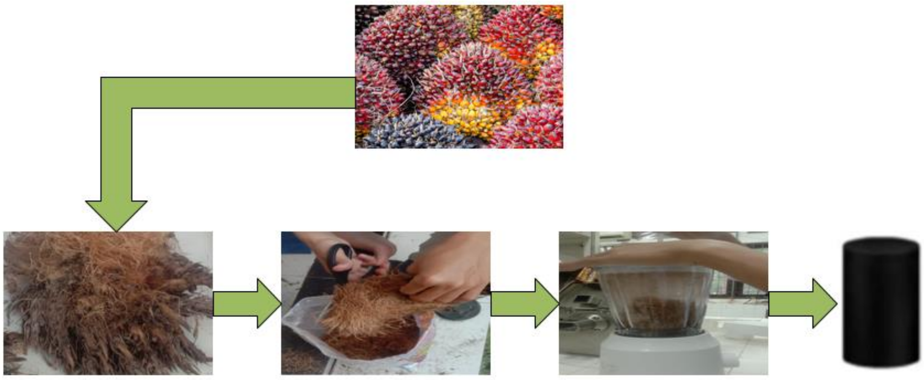

Fig. 1. EFB waste process to become biocoke fuel

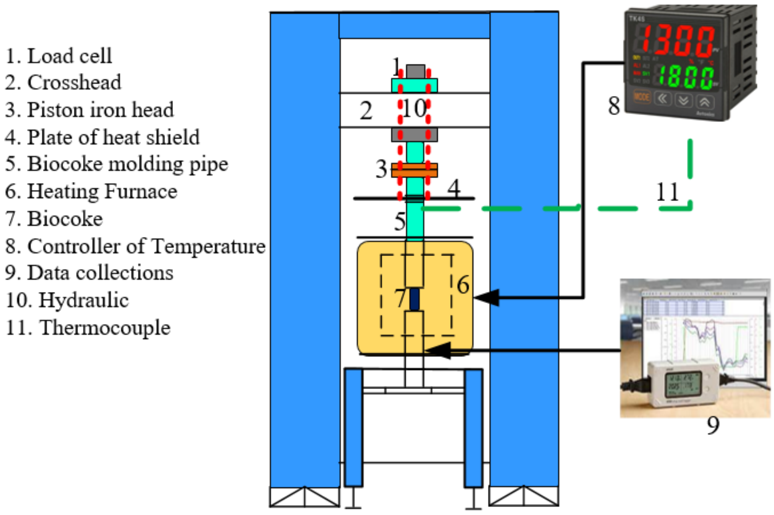

Fig. 2. Schematic diagram

Fig. 2 shows a schematic of the tool or system used for the heating and compression process in making biocoke. This system consists of several main components. First, there is a load cell (1) and a crosshead (2) on the top, which are used to measure the compression force. An iron piston (3) presses the biocoke material through a biocoke mould pipe (5), which is protected by a heat shield plate (4). The mould pipe is inserted into a heating furnace (6), which controls the temperature to ensure proper heating. The biocoke material (7) is heated in the furnace to a temperature set by a temperature controller (8), and temperature and compression process data are collected using a data collection device (9). The system is also equipped with a hydraulic device (10), which provides power for the compression process, and a thermocouple (11) to accurately measure the temperature inside the furnace.

2.1 Hardness Measurement

After being subjected to pressing and heating, the biocoke samples were each subjected to an individual measurement of their hardness using equipment that measures Hardness Shore D. Within this process, a durometer is utilised to determine the extent to which a particular indentation penetrates the material. The Shore D scale is frequently utilised to measure biocoke and other hard materials. Data on its hardness is collected to investigate how the process parameters influence the mechanical properties of the biocoke.

2.2 SEM-EDS Analysis

After complicated testing, the samples were analysed using energy-dispersive X-ray spectroscopy (EDS) to identify the elemental composition and scanning electron microscopy (SEM) to visualise the microstructure. Surface morphology and elemental distribution are related to hardness, and these methods can be used to identify manufacturing processes accurately. They are scanning Electron Microscopy with Energy Dispersive Spectroscopy. Scanning electron microscopy (SEM) scans the surface of a sample by firing high-energy electrons in a very narrow beam. The interaction of these electrons with atoms in the sample produces signals that can reveal variations in topography, composition, and other characteristics. Suppose we know the electron energy and the objective aperture angle. We can use Eq. 1 to calculate the lowest resolution (d) a scanning electron microscope (SEM) can achieve in an image.

$$d \approx \frac{1.22\lambda}{2\sin(\alpha)} \tag{1}$$

Where λ is the electron wavelength (which depends on the electron energy) and α is half the objective aperture angle. The electron wavelength can be calculated using (Eq.2) based on the de Broglie formula.

$$\lambda = \frac{h}{\sqrt{2m_e V(1+\frac{eV}{2mc^2})}} \tag{2}$$

Where h h is Planck's constant, m_e is the electron mass, V is the accelerating voltage, e is the electron charge, and c is the speed of light. Electron diffraction spectroscopy, often known as EDS, is a technique that determines the X-ray energy and intensity generated by a sample when an electron beam strikes it. Each element has its own unique X-ray "signature" that can be used to identify and quantify the sample component. This "signature" is determined by the energy of the produced photons. To rectify the data that has been acquired and obtain more accurate element concentrations in the sample, quantitative analysis using EDS frequently utilises semiquantitative quantification formulae such as the ZAF or Ϋ(ρz) formula. These formulas take into consideration the effects of atomic number (Z), absorption (A), and fluorescence (F) (Mishima et al. 1991).

a. Z (Atomic Number): The chance of electrons interacting with the sample is affected by this factor.
b. A (Absorption): This function adjusts the intensity of the detected signal based on the amount of X-rays absorbed by the sample.
c. F (Fluorescence): Considering the rise in particular intensity brought about by X-ray fluorescence by other sample components is essential.

It is necessary to represent the interactions between electrons and samples and the interactions between X-rays and samples to derive the ZAF or ϕ(ρz) formula. Additionally, the analytical software included with the EDS equipment is frequently utilised to carry out this operation. When used in conjunctions, each approach reveals information about the sample's minute structure and elemental composition that would have been impossible to determine if only one had been utilised.

2.3 Porosity Value Analysis

Analysis of the porosity value of a biocoke sample can reveal several qualities, including the capacity to hold and move fluids inside the sample, among other characteristics. The proportion of a material is the proportion of space (pores) out of the total volume of the material. Quantifying and assessing the porosity of the biocoke samples that were transferred using scanning electron microscopy is possible by utilising the equation (3-5) (Malvern 1969; Gibson 2007).

$$V_{solid} = \int_{y_{min}}^{y_{max}} \int_{x_{min}}^{x_{max}} f(x,y)dxdy \quad (3)$$
$$V_{total} = f_{max}\,(x_{max} - x_{min})(y_{max} - y_{min}) \quad (4)$$
$$\emptyset = \frac{V_{pori}}{V_{total}} = \frac{(V_{total} - V_{solid})}{V_{total}} = 1 - \frac{V_{solid}}{V_{total}} \quad (5)$$

3. Result & Discussion

Based on the results of data analysis in **Table 1**, the sample of oil palm empty fruit bunch (EFB) fibre heated at a temperature of 150°C showed significant physical characteristics. The H max value of 59,000 and H min of 6,800 indicate the range of mechanical strength of this material after thermal treatment. The coordinate parameters X (1,149) and Y (805) indicate the structure and dimensional distribution of the EFB sample tested. The total volume of the material reached 48.28 cm^3, where most of the volume was in the form of pores, which was 38.09 cm^3, equivalent to a porosity of 0.79, or around 79%. This shows that most of the volume of the material is not solid but consists of empty spaces or pores, which contribute to the porous properties of the material.

Furthermore, the solid volume value recorded at 10.18 cm^3 indicates the solid part of the sample that provides structural strength to the material. The integral volume of 16.47 cm^3 indicates the total volume of an integral part of the sample that includes essential elements to maintain material stability. The porosity of 78.90% suggests that this material has the potential to be a porous material with high storage capacity. This characteristic is vital for applications such as absorbent or energy storage materials, where large pore spaces can increase the adsorption capacity or holding capacity of liquids or gases.

Table 1. Results of porosity analysis of EFB samples at a temperature of 150°C.

Parameter	Sample of EFB (150°C)
H max	59000
H min	6800
X	1149
Y	805
Volume Total	48.28
Volume Solid	10.18
Volume Integral	16.47
Volume Pori	38.09
Porosity	0.79
Percentage	78.90

The analysis results show the microscopic structure of the oil palm empty fruit bunch (EFB) fibre sample after 150°C temperature treatment. **Fig. 3** shows the morphology of the material with quite significant pore variations. The diameter scale on the horizontal axis, which ranges from 0 to 1000 nm, shows that this material has a porous structure on the nanometer scale. The material's density, shown on the vertical axis (0 to 800 nm), provides an overview of the material's distribution of particles or fibres after the thermal treatment process. The colours in the image, with graduation from black to white associated with the density value, indicate the differences in the hardness or compactness of the material in different areas. Darker coloured areas (close to the value of 6800) indicate large pores or less dense parts, while lighter areas (close to 59.000) indicate denser or more compact material parts. This aligns with the porosity values found in the previous table analysis, where the EFB porosity reaches 78.90%. This image provides a strong visualisation that this material consists of a significant porous structure.

The image's layered and irregular structure indicates the exfoliation and microdamage the heat treatment may have caused. The thermal process at 150°C appears to affect the internal structure of the fibres, releasing inter-fibre bonds and creating larger pore spaces. This morphology is potentially advantageous in adsorbent or filtration applications, where the capacity to store and transmit substances through large pores is an important characteristic. The pore size distribution detected in this image shows a significant variation in pore size, ranging from very small to more critical. Larger pores can increase the material's permeability, while smaller pores can increase the adsorption capacity. When you mix these two kinds of pores, the heated EFB material can be used for many things, like as an adsorbent or as a lightweight composite material that balances strength and the ability to absorb.

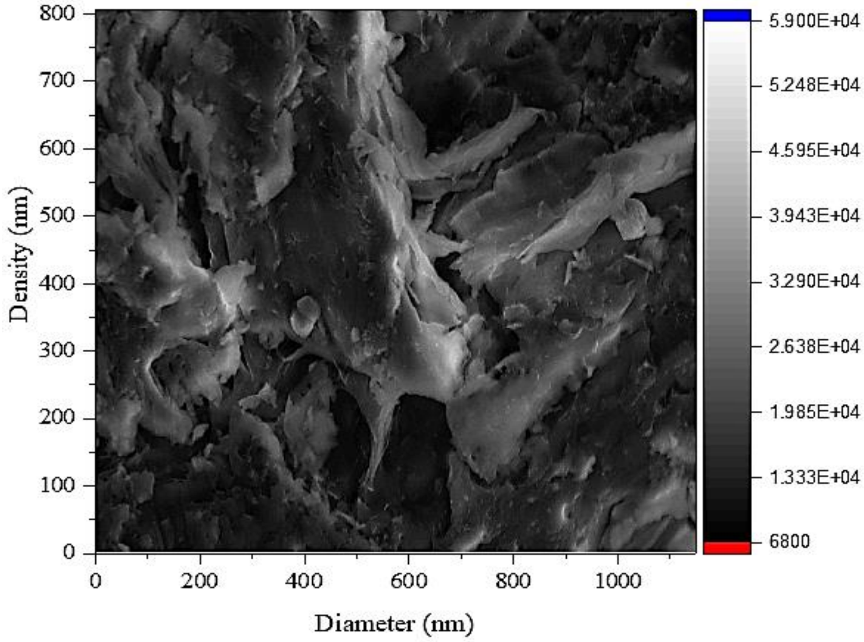

Fig. 3. Results of material morphology analysis with pore variations

Fig. 4 shows the density distribution of oil palm empty fruit bunch (EFB) fibre material heated at 150°C in the form of a colour map. On the horizontal axis, the image shows the range of pore diameters from 0 to more than 1000 nm, while the vertical axis shows the density of the material up to 800 nm. The colour scale on the right side indicates the variation in material density, with blue indicating areas of low density (values around 6,800). In contrast, red and yellow indicate regions of high density (values close to 59,000). The image's dominant blue and green colours indicate that most material structures are relatively low to medium density, which correlates with a reasonably ample pore space. This aligns with the material's high porosity characteristics (78.90%) in **Table 1**. The green zones scattered in several areas indicate a higher density, where there may be a denser material structure, such as fibres not significantly damaged by heating.

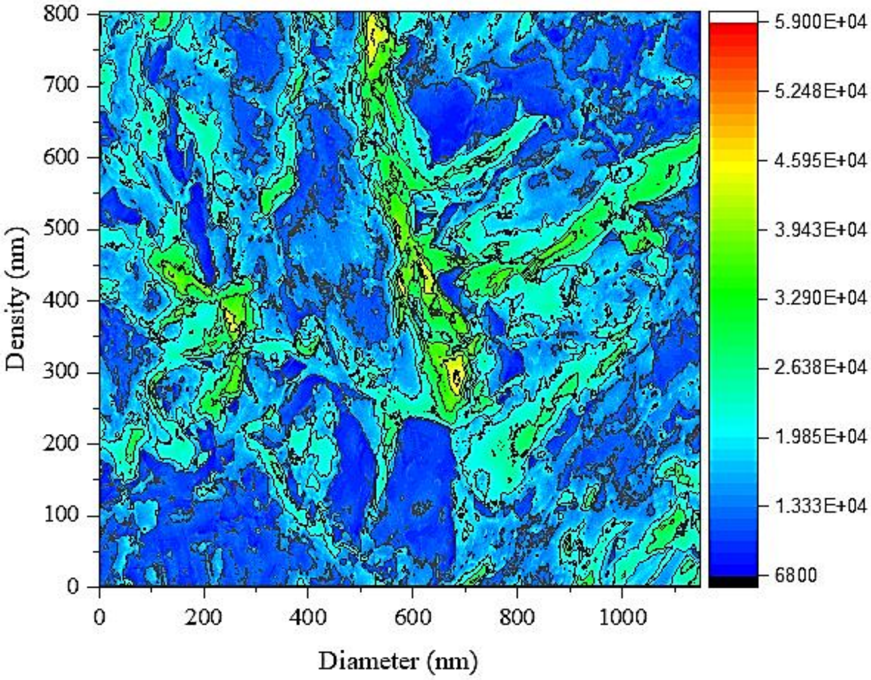

Fig. 4. Density distribution of EFB fibre material

In addition, this colour distribution pattern shows that although the material's porosity is quite large, there is significant variation in the size and distribution of the pores. Blue zones indicate large pores, which allow high permeability for fluids or gases. In contrast, zones with yellow or red colours indicate denser areas where the material is potentially more resistant to mechanical deformation or pressure. This combination of porous and dense areas provides an idea of the heterogeneity of the material, which is vital in determining the application of this material. Overall, this image provides visual information about the heterogeneity of the EFB material structure after thermal treatment.

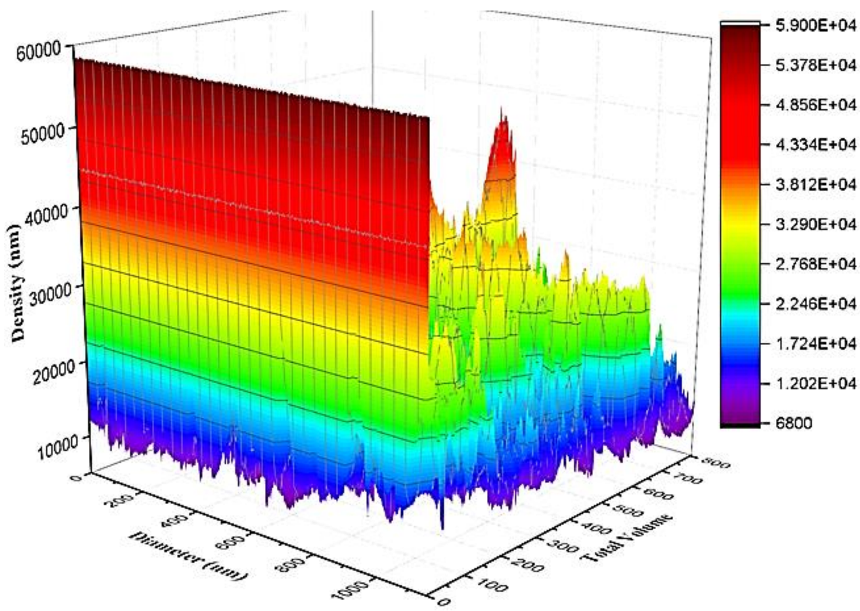

Fig. 5. Density dimensions of biocoke fuel from EFB waste at a temperature of 150°C

The pores with various sizes and varying density distributions indicate the potential use of this material in different technological applications, such as adsorbents, filtration, or composite materials. The complex density distribution also provides insight into how thermal treatment can change the material's microstructure, producing variations useful for specific applications that require a combination of porosity and mechanical strength.

Fig. 5 is a three-dimensional picture of how dense the EFB fibre material is when heated to 150°C. The parameters were calculated by measuring the material's diameter and total volume. On the horizontal (diameter) and vertical (total volume) axes, the image shows the development of the material structure. At the same time, the z-axis (upward) depicts the density of the material, expressed in nanometers (nm). The colours along the vertical axis reflect the variation in density values with a colour scale from blue to red, where blue indicates lower density, and red indicates very high density. This figure shows that most of the material has a high density concentrated in the area with a smaller diameter, marked in red on the upper left of the graph. This indicates the presence of a very dense part of the material in its microstructure, which may contribute to the structural strength of the material after thermal treatment. Along with the increase in diameter and total volume, there is a drastic decrease in density, which can be seen from the colour gradation that changes to green, blue, and purple. This indicates that pores begin to form with larger sizes and the voids in the structure increase.

The intermediate density regions (green and yellow) show a more uniform distribution of material but still show the presence of pore space that allows fluid interaction within the material. These regions indicate the potential use of EFB materials as adsorbents or energy storage, where larger pores increase the absorption capacity or liquid flow. The areas with sharply decreasing density (blue and purple) indicate that these materials have large pores that dominate at larger diameters, consistent with the high porosity characteristics found in the previous **Table 1**. This three-dimensional graph provides deep insight into the heterogeneity of the EFB material structure after heating, showing a combination of dense and porous areas. The varying distribution in this graph includes essential information about the material's physical properties that can be optimized for various applications. With high density at small diameters and large porosity at large diameters, this material has great potential in applications such as filters or adsorbents requiring strength and permeability.

4. Conclusion

This research investigated what happens to the physical and morphological properties of EFB fibre when it is heated to 150°C. Specifically, it looked at how the porosity and pore volume distribution change. Based on the measurement results, the EFB sample showed a very high porosity of 78.90%, with a pore volume of 38.09 cm^3 from a total volume of 48.28 cm^3. This increase in porosity indicates that thermal treatment at this temperature can significantly increase the pore space in the material, allowing its use as an efficient adsorbent. Morphological analysis with microscopic images shows an irregular layered structure, with large and small pores evenly distributed. These images confirm that heating at 150°C affects the pore volume and causes changes in the material's microstructure, making it more fragmented and porous. The three-dimensional graph further shows the material's density distribution, which shows a high density at small diameters and a drastic decrease in density at larger diameters, indicating a heterogeneous and porous structure. These findings provide important insights into the potential applications of EFB-based materials, especially in porous material technology such as filtration, energy storage, and liquid or gas adsorption. This research also opens up wider opportunities to utilise biomass waste as an environmentally friendly material, supporting industrial sustainability by developing new materials with high performance and good efficiency in various technological applications.

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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.

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