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Porosity and Pore Volume Analysis of EFB Fiber: Physical Characterization and Effect of Thermal Treatment

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Abstract
This research looked at the shape and physical properties of oil palm empty fruit bunch (EFB) fibre heated to 160°C, mainly looking at the number of pores and their size. The results showed that the EFB sample had a porosity of 68.98% with a pore volume reaching 40.69 cm³ from a total volume of 58.99 cm³. This indicates that most material consists of void spaces or pores, which have potential applications in porous materials such as filtration and adsorption. The microscopic pictures showed that the pores were not spread out evenly, with dense areas being spread out. The density distribution also revealed that the material's structure was not uniform, with larger pores and denser areas concentrating at smaller diameters. This study concluded that thermal treatment at 160°C significantly increased the porosity of the EFB material while maintaining a reasonably dense solid area. These findings provide valuable insights into using biomass waste for applications requiring porous properties and mechanical strength, especially in the energy storage, filtration, and adsorption industries.

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1. Introduction
Biomass waste as an alternative raw material in various technological applications has become an exciting research topic in recent decades. One of the biomass wastes with excellent potential is oil palm empty fruit bunches (EFB), a by-product of the palm oil industry. EFB is often discarded or burned, which causes environmental problems. However, the physical and chemical characteristics of EFB indicate that this material can be utilized as a porous material in technological applications such as energy storage, filtration, and adsorption (Wadchasit et al. 2021; Erdiwansyah et al. 2023; Gani et al. 2023, 2024a, b). Thermal treatment is one method to change biomass materials' physical and morphological properties, including EFB (Abdullah et al. 2019; Ibrahim et al. 2019; Isworo et al. 2020). This process can increase porosity and create a more heterogeneous structure, potentially improving the desired material properties in

technological applications (Ishizaki et al. 2013; Wu et al. 2019; Johnston et al. 2024). One of the crucial parameters affected by thermal treatment is porosity, which is the amount of space or pores in the material. Materials with high porosity are highly desirable in applications requiring high permeability, such as filtration systems or adsorbents.

This study focuses on the physical characterization of EFB fibres after thermal treatment at 160°C. The main objectives of this study are to analyze the changes in porosity and pore volume after heat treatment and to explore the potential of EFB as a porous material. In addition, the material's density distribution and morphological structure after heat treatment were analyzed using a microscope, providing deeper insight into the distribution of pores and dense areas in the material structure. The results of this study are expected to provide helpful information for the utilization of EFB in various technological applications, especially in energy storage, filtration, and adsorption (Ayinla et al. 2019; Erdiwansyah et al. 2021, 2022). This study shows that EFB has a porosity of 68.98% after thermal treatment, with a pore volume reaching 40.69 cm^3 of a total volume of 58.99 cm^3. In addition, the uneven pore distribution and heterogeneity of the resulting material structure indicate that EFB maintains its mechanical strength even though most of the material is porous. These findings suggest that EFB has significant potential as a porous material with high mechanical strength and permeability. This study is unique because it looks at the physical and morphological changes of EFB fibre after being heated at 160°C. The main changes are related to the material becoming more porous and having a more even density distribution. This study not only provides quantitative data on porosity reaching 68.98% but also reveals the heterogeneity of the material structure, which shows a balance between large porous areas and more focused dense areas. This provides new insights into the potential of EFB as a multifunctional material that combines high permeability with sufficient mechanical strength, which has not been widely discussed in previous literature. In this way, the study makes an immense contribution to the progress of biomass-based applications, especially the need for materials with high porous properties for infiltration, adsorption, and energy storage. It also adds to the conversation about using biomass waste more effectively and in a way that creates value.

2. Methodology

Fig. 1 shows the processing of empty palm oil waste to produce biocoke solid fuel. However, it is first crushed into a finer powder before being produced into biocoke. The first stage shows the EFB, which is still in its whole form. Then, the EFB is cut using scissors to make it easier to process. After being cut, the EFB is put into a blender and crushed into a powder or powder form. The last step shows the final result in fibre powder collected in a container, which may be used for further purposes such as producing biocoke fuel.

Fig. 1. EFB waste process to become biocoke fuel

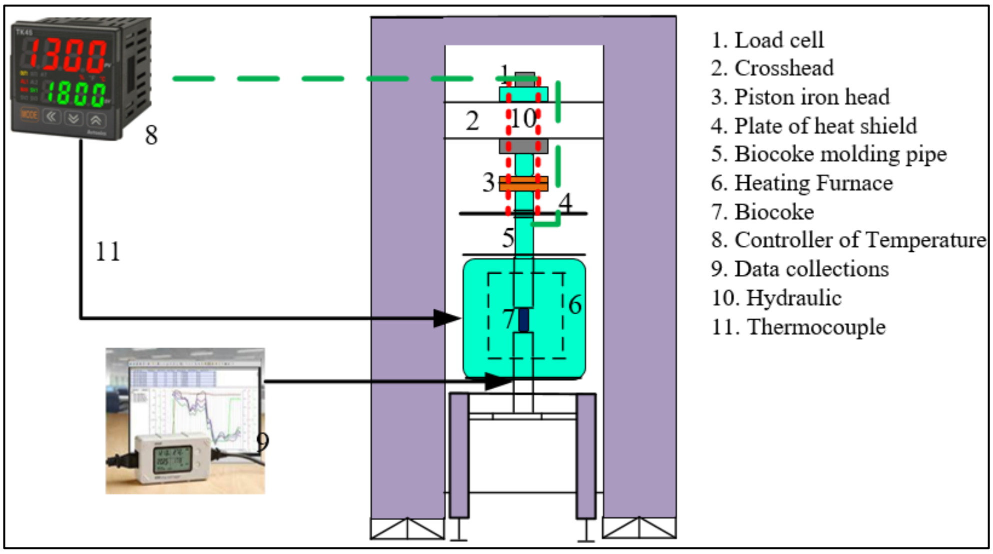

Fig. 2. Schematic diagram

The instrument or system utilized for the heating and compression process in the production of biocoke is depicted in schematic form in Fig. 2. Several primary components make up this system. To begin with, there is a load cell (1) and a crosshead (2) on the top, both of which are utilized to measure the resistance to compression. The iron piston (3) oversees pressing the biocoke material through a biocoke mould pipe (5) that is heat-shielded by a heat shield plate (4). The mould pipe is placed inside a heating furnace (6), which regulates the temperature to ensure the mould is heated appropriately. A temperature controller (8) determines the temperature at which the biocoke material (7) is heated in the furnace. A data collection device (9), used to collect data on the compression process, is then used to capture the temperature and compression process data. In addition, the system is outfitted with a thermocouple (11) that can correctly measure the temperature inside the furnace and a hydraulic device (10), which supplies power for the compression process.

2.1 Hardness Measurement

Following the application of pressure and heating, the biocoke samples were individually subjected to an individual measurement of their hardness using an apparatus that measures Hardness Shore D. This was done after the samples had been subjected to pressing and heating. During this procedure, a durometer is utilized to ascertain the degree to which a specific indentation penetrates the material. The Shore D scale is usually used to accurately measure biocoke and other hard materials. Data on its hardness is collected to explore how the process parameters influence the mechanical properties of the biocoke.

2.2 SEM-EDS Analysis

The samples were put through many complex tests before being looked at with energy-dispersive X-ray spectroscopy (EDS) to find out what elements were present and scanning electron microscopy (SEM) to see the microstructure. Hardness is related to surface morphology and elemental distribution, and these methods can be used to identify manufacturing processes correctly. Hardness is a parameter that can be measured. Electron microscopy with energy dispersive spectroscopy for scanning electron microscopy... SEM, or scanning electron microscopy, is a technique that involves shooting high-energy electrons in a very narrow beam to examine the surface of a material. The signals produced due to the interaction of these electrons with the atoms in the sample can show differences in topography, composition, and other features. Suppose we are aware of the energy of the electrons as well as the angle of the objective aperture. We can determine the lowest resolution (d) a scanning electron microscope (SEM) can achieve in an image using Equivalent Eq. 1.

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

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.

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

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, referred to as EDS, is a method to ascertain a sample's X-ray energy and intensity when an electron beam contacts it. Each element's X-ray "signature" is one of a kind, and to identify and measure the sample component, it is possible to use this signature. The energy of the photons that are created is what dictates this "signature" or characteristic. When doing quantitative analysis using EDS, it is common practice to employ semiquantitative quantification equations, such as the ZAF or Ÿ(ρz) formula, to rectify the data gathered and obtain more accurate element concentrations in the sample. The atomic number (Z), absorbance (A), and fluorescence (F) are all factors that are taken into consideration by these calculations (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.

To derive the ZAF or ϕ(ρz) formula, it is imperative to accurately depict the interactions between electrons and samples and between X-rays and samples. In addition, the analytical software provided with the EDS equipment is typically utilized to carry out this process. When used in concert with the others, every method exposes information about the sample's minute structure and elemental makeup that would have been impossible to ascertain if only one of the methods had been utilized.

2.3 Porosity Value Analysis

Examining the porosity value of a biocoke sample can reveal several properties, including the ability to both retain and transfer fluids inside the sample, amongst other qualities. When compared to the entire volume of a substance, the proportion of a material is the proportion of space (pores) that that material contains. Using the equation (3-5), it is possible to quantify and evaluate the porosity of the biocoke samples that were transferred using scanning electron microscopy. This can be done by utilizing the equation. (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

The analysis results in **Table 1** are samples of oil palm empty fruit bunches (EFB) heated at a temperature of 160°C, which shows results that reflect significant changes in their physical characteristics. The H max value of 65,600 and H min of 2,200 indicate an extensive range of mechanical strength, indicating that the material maintains good structural integrity even after heating. The X and Y parameters recorded values of 1,153 and 807, respectively, which provide information related to the dimensions or distribution of the sample components after thermal treatment. The total volume of the material was recorded at 58.99 cm^3, with a pore volume of 40.69 cm^3 and a solid volume of 18.29 cm^3. These data indicate that most of the material consists of pores with a porosity percentage of 68.98%. This means that about 69% of the total volume of the material is space or pores, indicating that the heat treatment has produced a material with significant porous properties. This characteristic is essential in material applications that require high permeability, such as absorbents or materials for filtration. An integral volume of 20.34 cm^3 indicates the distribution of solid material within the sample, which is essential for maintaining strength and structural stability despite its high porosity.

Table 1. EFB samples were subjected to a porosity study at a temperature of 160ºC.

Parameter	Sample of EFB (160ºC)
H max	65600
H min	2200
X	1153
Y	807
Volume Total	58.99
Volume Solid	18.29
Volume Integral	20.34
Volume Pori	40.69
Porosytas	0.69
Persentase	68.98

Fig. 1 shows the microscopic structure of oil palm empty fruit bunch (EFB) fibre after being heated at 160°C, with the diameter scale on the horizontal axis and the density on the vertical axis. The colour scale on the right side shows the variation of density, where red indicates low density (the lowest value is 2,200), and blue indicates high density (the highest value is 65,600). This structure illustrates the distribution of pores and solid materials in the sample after thermal treatment. The darker areas in the image indicate pores or voids within the EFB structure, which is consistent with the porosity measurement result of 68.98%. The lighter-coloured zones, especially in the prominent round areas, indicate the presence of denser material. This distribution of pores and dense areas suggests that thermal treatment causes the material to become more heterogeneous, with large pores and dense regions remaining in the form of a more focused structure.

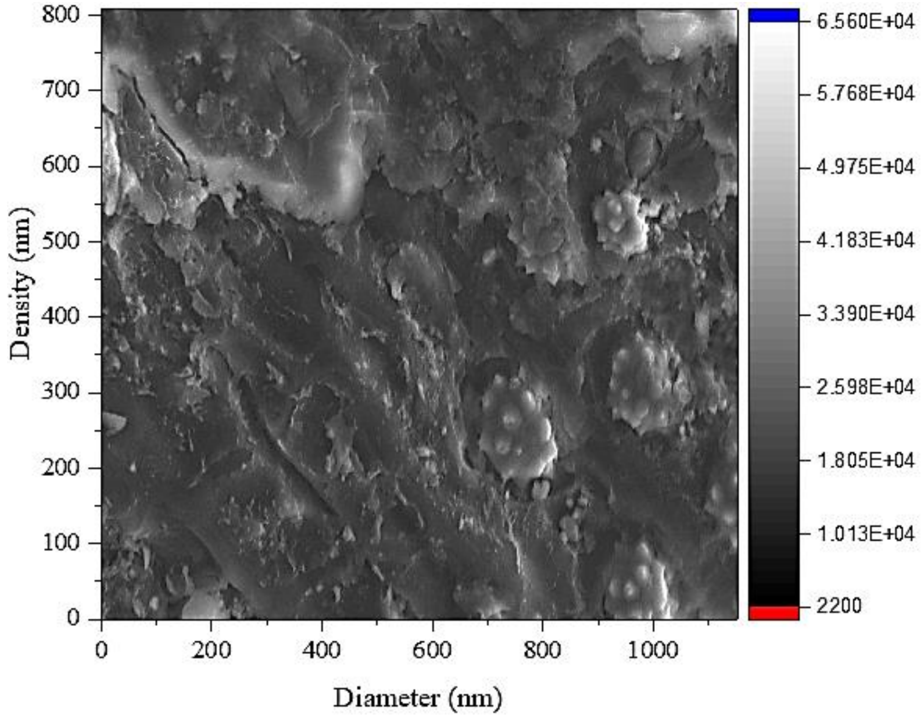

Fig. 1. Microscopic structures in EFB samples

At diameters around 500-800 nm, prominent cluster-like formations are seen, indicating the presence of denser, more concentrated material in certain areas. Heating may not significantly impact these areas, maintaining the material's mechanical integrity. This is important for applications where a combination of strength and porosity is required, such as filters or adsorbents. This image shows that although the EFB has been heat treated, the material maintains a diverse distribution between large pores and dense structures. This combination indicates that this material has potential for various technological applications that require porous materials with high permeability but still have mechanical resistance in certain areas.

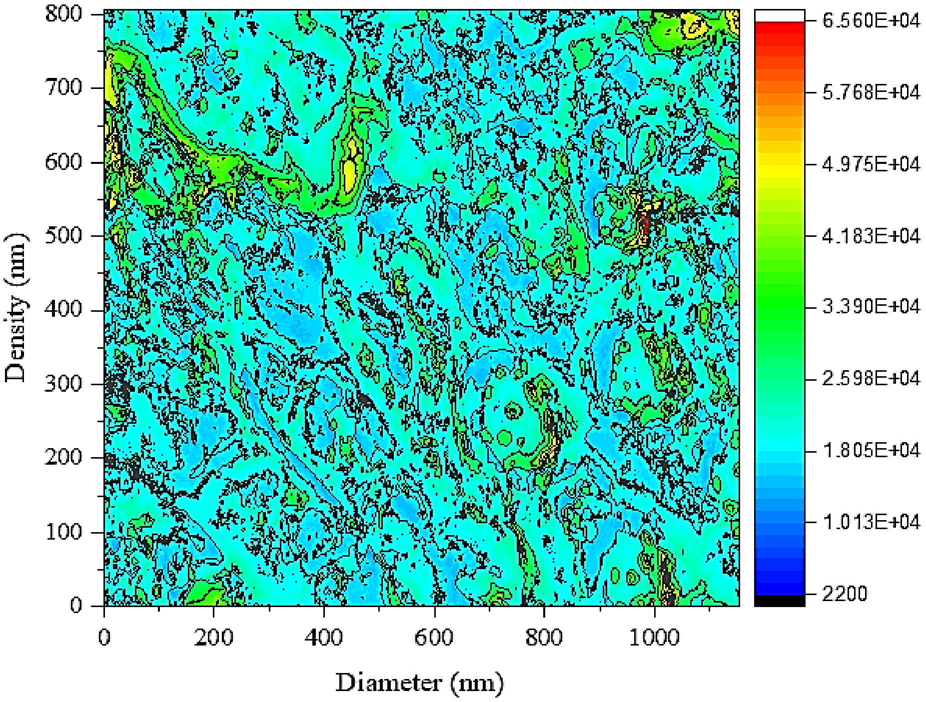

Fig. 2. Distribution for EFB Sample

Fig. 2 shows the density distribution of oil palm empty fruit bunch (EFB) fibre material heated at 160°C. The density (vertical axis) and pore diameter (horizontal axis) are displayed in the nanometer (nm) scale. The colour scale on the right side shows the density values, with blue depicting the lowest density region (around 2,200) and red depicting the highest density region (around 65,600). This figure provides a detailed visualization of the variation in material structure after heat treatment. The dominant blue and green colour distribution indicates that most of the material has a low to medium density, which supports the observation that this material is highly porous. The pores shown in blue in various areas of the image indicate the presence of significant void spaces, supporting the high porosity percentage (68.98%) measured in the previous table data. This shows that the material has undergone structural changes due to heating, creating significant pore spaces essential for porous material applications such as absorbers or filters.

On the other hand, some small areas are yellow to red, indicating the presence of the high-density regions. These areas serve as points of solid mass concentration in the material, which heating may not significantly affect. The distribution of these high-density areas tends to be scattered and not focused in one place, indicating that the material's structure remains quite heterogeneous even through the heating process. Overall, this figure demonstrates that the EFB structure, after heating, still has variations in the distribution of pores and density. This variation reflects the material's multifunctional nature, where the combination of high porosity and randomly distributed local density allows the material to function in various applications, from adsorbent materials to filtration media. This combination of porous and dense areas balances the permeability and mechanical strength of the material.

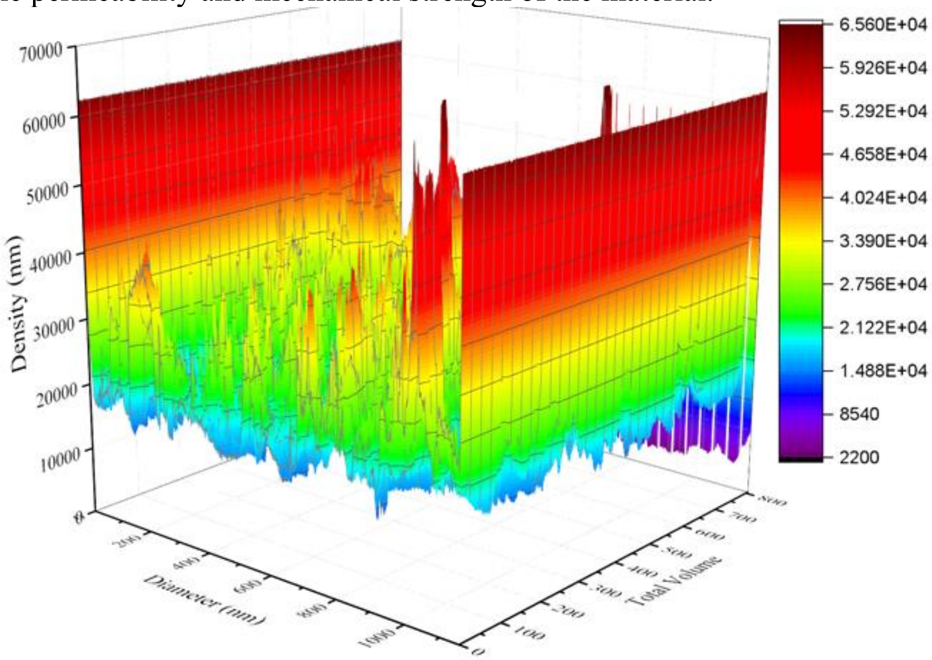

Fig. 3. EFB waste biocoke fuel density at 160°C.

Fig. 3 shows the three-dimensional distribution of the density of the EFB fibre material heated at 160°C. On the horizontal axis, there are two parameters, namely, the pore diameter and the total volume of the material. The vertical axis shows the density of the material (in nanometers). The colour scale on the right side of the figure indicates the variation in material density, with red indicating high density (up to 65,600 nm). In comparison, blue and purple indicate low density (around 2,200 nm). The colour distribution in this figure shows that most of the material has a high density, especially at smaller diameters, clearly visible in the red to yellow areas at the top of the graph. This area indicates that the dense material tends to be concentrated at small diameters, and its volume is relatively low. This dense structure suggests that a part of the material still maintains its integrity even after being thermally treated, so it has the potential to be a material that can maintain mechanical strength.

In contrast, the green, blue, and purple areas indicate a more extensive pore distribution in the material with a larger diameter. This reflects that the material has more voids or pores at a large diameter, following the recorded porosity results of 68.98%. This indicates that the EFB material, after thermal treatment, has a significant porous structure, which is vital for applications that require high permeability, such as adsorbents or filter media. Overall, this image shows the heterogeneity of the EFB material after heating, with variations between dense and porous areas. The high-density part is distributed in a smaller volume, while the high-porosity part appears larger. The combination of these two characteristics suggests that this material has multifunctional potential in various technological applications that require a balance between mechanical strength and permeability.

4. Conclusion

This study explores the physical and morphological characteristics of EFB fibre after thermal treatment at 160°C, focusing on analysing porosity and pore volume. Based on the results obtained, the EFB sample showed a relatively high porosity of 68.98%, with a pore volume reaching 40.69 cm^3 from a total volume of 58.99 cm^3. This indicates that most of the material consists of significant void spaces or pores, which have the potential for applications in porous materials, such as filtration and adsorption. The microscopic view visualizes the material's morphology after heating, showing a layered structure with an uneven distribution of pores but scattered dense areas. The visualized density distribution shows heterogeneity in the material structure, with large porous areas at larger diameters and dense regions concentrated at smaller diameters. This distribution supports the conclusion that the thermal treatment at 160°C resulted in a sizeable porous structure, while some parts of the material retained their strength. The three-dimensional graph further confirms that the high density at small diameters indicates that the material still has good mechanical strength even when heated.

In contrast, the drastic decrease in density at large diameters reflects a significant increase in porosity. This finding is essential in developing multifunctional materials where a combination of high mechanical strength and permeability is required. Overall, this study shows that thermal treatment of EFB at 160°C significantly increases the material's porosity while maintaining a sufficiently strong solid area. These findings provide valuable insights into using biomass waste for applications requiring porous properties and mechanical strength. Thus, EFB has excellent potential for use in various industries, especially in energy storage, filtration, and adsorption.

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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References

Abdullah CI, Azzahari AD, Rahman NMMA, et al. (2019) Optimizing treatment of oil palm-empty fruit bunch (OP-EFB) fiber: Chemical, thermal and physical properties of alkalized fibers. Fibers Polym 20:527–537

Ayinla RT, Dennis JO, Zaid HM, et al. (2019) A review of technical advances of recent palm bio-waste conversion to activated carbon for energy storage. J Clean Prod 229:1427–1442

Erdiwansyah, Gani A, MH N, et al. (2022) Policies and laws in the application of renewable energy Indonesia: A reviews. AIMS Energy 10:23–44. https://doi.org/10.3934/energy.2022002

Erdiwansyah, Gani A, Zaki M, et al. (2023) Analysis of technological developments and potential of biomass gasification as a viable industrial process: A review. Case Stud Chem Environ Eng 8:100439. https://doi.org/https://doi.org/10.1016/j.cscee.2023.100439

Erdiwansyah, Mahidin, Husin H, et al. (2021) A critical review of the integration of renewable energy sources with various technologies. Prot Control Mod Power Syst 6:3. https://doi.org/10.1186/s41601-021-00181-3

Gani A, Adisalamun, Arkan D MR, et al. (2023) Proximate and ultimate analysis of corncob biomass waste as raw material for biocoke fuel production. Case Stud Chem Environ Eng 8:100525. https://doi.org/https://doi.org/10.1016/j.cscee.2023.100525

Gani A, Erdiwansyah, Desvita H, et al. (2024a) Analysis of chemical compounds and energy value for biocoke fuel by FTIR and TGA. Case Stud Chem Environ Eng 100644. https://doi.org/https://doi.org/10.1016/j.cscee.2024.100644

Gani A, Erdiwansyah, Desvita H, et al. (2024b) Comparative analysis of HHV and LHV values of biocoke fuel from palm oil mill solid waste. Case Stud Chem Environ Eng 9:100581. https://doi.org/https://doi.org/10.1016/j.cscee.2023.100581

Gibson RF (2007) Principles of composite material mechanics. CRC press

Ibrahim Z, Ahmad M, Aziz AA, et al. (2019) Properties of chemically treated oil palm empty fruit bunch (EFB) fibres. J Adv Res Fluid Mech Therm Sci 57:57–68

Ishizaki K, Komarneni S, Nanko M (2013) Porous Materials: Process technology and applications. Springer science & business media

Isworo YY, Kim G-M, Jeong J-W, Jeon C-H (2020) Evaluation of torrefied empty fruit bunch (EFB) and kenaf combustion characteristics: Comparison study between EFB and kenaf based on microstructure analysis and thermogravimetric methods. Energy & Fuels 34:7094–7104

Johnston W, Godakawela J, Gatti C, et al. (2024) Fibro-porous materials: 3D-printed hybrid porous materials for multifunctional applications. Addit Manuf 94:104470. https://doi.org/https://doi.org/10.1016/j.addma.2024.104470

Malvern LE (1969) Introduction to the Mechanics of a Continuous Medium

Mishima Y, Yong Myong Hong, Suzuki T (1991) Determination of the γ solvus surface in Ni□Al□X ternary systems. Mater Sci Eng A 146:123–130. https://doi.org/https://doi.org/10.1016/0921-5093(91)90272-O

Wadchasit P, Suksong W, O-Thong S, Nuithitikul K (2021) Development of a novel reactor for simultaneous production of biogas from oil-palm empty fruit bunches (EFB) and palm oil mill effluents (POME). J Environ Chem Eng 9:105209. https://doi.org/https://doi.org/10.1016/j.jece.2021.105209

Wu J, Xu F, Li S, et al. (2019) Porous polymers as multifunctional material platforms toward task-specific applications. Adv Mater 31:1802922