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Synthesis and Characterization of Pelletized Coke with Tar Impregnation from Carbonization of Palm Kernel Shells as Nickel Laterite Reductant

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Abstract. The nickel laterite beneficiation policy supports the domestic stainless and alloy steel industries. However, reliance on imported coke for smelting remains a major challenge. The main challenge is the dependence on imports of reductant coke for the smelting process. Palm kernel shell (PKS) is a promising alternative that aligns with the carbon-neutral concept, but its pyrolyzed carbon shows low calorific value and mechanical strength for metallurgical use. This study aims to produce biocoke by converting PKS into carbon–carbon (C/C) composites using its carbon and tar products to enhance mechanical and thermoplastic properties. This study aims to produce biocoke by converting PKS into carbon–carbon (C/C) composites using its carbon and tar products to enhance mechanical and thermoplastic properties. Initial KOH activation (impregnation mass ratio 0.5) created a porous structure for tar deposition. Briquetting (30×30 mm) followed by co-carbonization (250 °C, 1 h) was conducted at various char-to-tar mass ratios. The best condition was achieved at a tar ratio of 1:2, producing composites with high fixed carbon (88.17%), low volatile matter, and compressive strength suitable for metallurgical applications. SEM analysis confirmed uniform carbon fiber distribution within the matrix. The final product also met ASTM D3173 standards with a heating value of 7,328 kcal/kg. XRD analysis of limonite ore reduction using this biocoke showed a decrease in geothite and lizardite phases and the formation of metallic phases such as Fe-Ni alloy, FeS, and wustite, especially at 900 °C and 1100 °C. These results indicate effective metallothermic reduction. C/C composite from PKS offers a sustainable, high-performance alternative to commercial coke for nickel laterite reduction, fulfilling both energy and environmental considerations.

Keywords: Biocoke, Carbon-carbon Composite, Co-carbonization, Laterite Reduction, Palm Kernel Shell, Tar

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INTRODUCTION

Indonesia's steel industry plays a crucial role in national development, with domestic demand for steel rising due to infrastructure, manufacturing, and construction growth. To meet this demand, the government has implemented various policies and initiatives, including the National Steel Industry Strategy, targeting a production increase to 10 million tons annually by 2025 (Ministry of Energy and Mineral Resources of the Republic of Indonesia, 2020). Domestic mineral processing, particularly nickel, is key to reducing reliance on steel imports and supporting local manufacturing. As the world's largest nickel producer, Indonesia benefits from high-grade, low-sulfur nickel reserves, making it a strategic player in the global nickel and steel industries (Agung and Adi, 2022).

The steel industry faces significant challenges in the smelting process due to the need for large amounts of carbon reductants, specifically coke. It has been reported that producing one ton of liquid steel (hot metal) requires approximately 525 kg of coke (Mochizuki *et al.*, 2016). Coke plays a crucial role in supplying energy and acting as a reducing agent for oxides, with good gas permeability in the reduction reaction, allowing metal and slag to melt (Nomura, 2017). However, commercial coke is currently only produced from coking coal, which Indonesia must import due to limited domestic reserves (Manurung *et al.*, 2024). In recent years, the availability of metallurgical coal in producing countries has significantly declined, leading to a shortage of supply in the international market and a sharp increase in coke prices. Moreover, environmental concerns are pushing for reductions in carbon emissions from coal-based steel smelting processes, as this process is responsible for producing around 1.83 tons of CO2 per ton of crude steel (Suriapparao and Tejasvi, 2022). These circumstances urgently call for research and development of alternative reductants to reduce national dependence on coal in nickel laterite smelting processes.

Utilization of palm kernel shell (PKS) is a suitable candidate for further utilization as a coke reductor feedstock because it has a high lignocellulose content (40-60% dry weight), has a hard structure and the calorific value of the raw material is higher than some other domestic biomass (>4000 cal/g) (Yuliusman and Nafisah, 2021). The slow carbonization technique, with low reaction temperatures and slow heating rates (2-10 °C), can produce high-quality activated carbon. This process tends to create a more organized and homogeneous pore structure with chemically stable carbon (Adrados *et al.*, 2016). However, the calorific value and carbon yield are generally low due to the dominance of tar production.

Carbon derived from PKS also has lower mechanical strength compared to coal, making it less rigid during compression and more prone to breakage during reduction in rotary kilns. While PKS carbon lacks thermoplasticity due to its high oxygen content, the tar itself exhibits good thermoplastic properties. To enhance the properties of PKS carbon, a carbon/tar composite can be created, which improves both carbon yield and the chemical and mechanical properties of the material (Hariyadi *et al.*, 2020; Mochizuki *et al.*, 2018b). In this context, *carbon-carbon (C/C) composites* refer to materials composed entirely of carbon, both in the reinforcing phase and the matrix. Specifically, C/C in this study refers to composites formed from tar-derived carbon deposited during thermal treatment, acting as both matrix and reinforcement (Abbaspour *et al*., 2025).

Further research has been conducted on depositing carbon from tar decomposition into porous iron ore, with significant changes in surface area observed after tar impregnation at 600 °C (Mochizuki *et al*., 2018b; Zhao *et al*., 2025). Activated carbon from PKS using KOH as an activator during carbonization at 600 °C has produced mesoporous carbon with a surface area of 658 m^2/g, highlighting its potential for tar deposition (Hariyadi *et al*., 2024). Briquetting using polymer adhesives such as polyvinyl acetate has also shown improvement in calorific value and mechanical properties, though this method is less economical on a larger scale. This research seeks to develop a circular production concept for C/C composite coke derived from PKS carbonization, with feasibility testing for laterite nickel ore reduction. Key parameters to evaluate include the effects of alkali activation on pore size, carbon-tar impregnation ratios, and co-carbonization operating conditions.

MATERIALS AND METHODS

Sample

PKS pieces with particle sizes ranging from 550 to 750 µm were used in this study. The PKS samples were sourced from a local palm oil mill in Kalimantan Timur and were prepared according to the procedures outlined earlier. The proximate and ultimate analyses of the PKS samples, detailing their composition and properties, are presented in Table 1.

Alkali Activation

Alkali activation is performed using a one-step method with KOH. The dry PKS samples are mixed with KOH solution (1.8 M) at different mass ratios (1:0.25, 1:0.5, and 1:1), stirred for 6 hours, and left at room temperature for 18 hours, resulting in a total activation time of 24 hours. After activation, the samples are separated from the filtrate and dried at 110 °C for 3 hours.

Table 1. Analyses of PKS used in this study.

Parameter	Unit	Value	Method
Proximate Analysis			
Moisture content (M)	wt%-as received	9.37	ASTM D3173
Volatile matter (VM)	wt%-adb	67.76	ASTM D3173
Fixed carbon (FC)	wt%-adb	21.19	by difference
Ash content (AC)	wt%-adb	1.68	ASTM D3173
Ultimate Analysis			
C	wt%-daf	46.65	ASTM D5373
H	wt%-daf	5.17	ASTM D5373
N	wt%-daf	<0.1	ASTM D5373
S	wt%-daf	<0.1	ASTM D4239
O	wt%-daf	48.16	by difference
Gross calorific value (GCV)	cal/g	4,698	ASTM D3173

Carbonization of PKS

Carbonization of PKS was conducted with a pilot retort furnace quartz reactor equipped with a condenser downstream of the reactor. It features an airtight seal to prevent air entry and a gas outlet for collecting carbonization gases and trapping the oils. After approximately 500 g of the dried sample was charged in the reactor, the sample was heated at a rate of 5 °C/min to 600 °C for 2 hours. The char was recovered after cooling the reactor to an ambient temperature. The char sample from carbonization is then washed with hot water and filtered to separate from the residue. Washing is repeated until the pH of the filtrate is neutral. The neutral char was oven-dried for 6 hours.

Preparation of Composite C/C

The briquetting process was carried out using char and oils (as received tar) samples produced from the best variation in the carbonization process. The char sample was then homogenized with carbonization tar as an adhesive with a binder ratio of 1-5% w/w (mass ratio). The mixture was then molded into 30 x 30 mm cylindrical briquettes. The samples were then left for curing for 12 hours so that the carbon particles could stick together well. Then the co-carbonization process was carried out at a temperature variation of 250 °C for 1 hour with a heating rate of 5 °C/min. The pore structure of the samples before and after CC preparation was measured by the N_2 adsorption method. The surface morphology of the particles before and after co-carbonization was also confirmed through SEM Analysis. The indirect tensile strength of the coke was calculated by Eq. (1)

$$f = \frac{2P}{\pi d l} \tag{1}$$

where, f is the indirect tensile strength (MPa), P is the failure load (N), and d and l are the sample diameter and thickness (mm), respectively. The proximate and GCV of the biocoke were also analyzed to find composites that could meet commercial standards.

Reduction Test for Laterite Ore

The performance of biocoke was carried out to reduce laterite nickel ore using the roasting method. 15 %wt of meshed CC 1:2 composites and 10 % wt of Na_2SO_4 (Merck) were fully combined with 150 µm of nickel laterite ore. For the reduction process, pellets with a diameter of 10 to 15 mm were created and placed in a muffle furnace. The holding duration was 120 minutes, while the reduction temperature ranged from 700°C to 1100°C with a heating rate of 5 °C/min. XRD patterns were recorded using a diffractometer (Pan Analytical Powder X-Pert) under the following conditions: radiation Cu Kα, tube current, and voltage of 20 mA and 40 kV, respectively, the scanning range of 10° to 80°, step size of 0.017°, and scanning speed of 10.16°/s.

RESULT AND DISCUSSION

Effect of Alkali Activation on Pore Structure

The carbonization reaction mechanism is very complex and can vary depending on the selection of the reaction temperature. Initial optimization has been carried out by this research team previously through the study of the effect of temperature on the quality of charcoal produced in the temperature range of 500 - 800 °C (Hariyadi *et al*., 2024), referring also to several previous studies (Lobato-Peralta *et al*., 2025; Ngo *et al*., 2024; Xu *et al*., 2024). The best results were obtained at carbonization time at 600 °C for 2 hours based on the highest FC value (>70%) and the calorific value of the char produced (>6,000 cal/g). To increase the porosity of char particles to provide a high particle surface area for carbon deposition during the preparation of CC, we conducted an impregnation method using KOH for char. The one-step process-based activation method was chosen to reduce energy consumption, minimize process waste, and enhance materials efficiency.

During the carbonization process, the reaction between KOH and carbon initially occurs as a solid-solid reaction, which then progresses into a solid-liquid reaction. The redox reaction between carbon and KOH, along with other potential reactions, is illustrated in equations (2) to (6). The activation temperature significantly influences the reaction mechanism. Initially, KOH is converted to K_2O at 400°C through dehydration reaction (Eq. (2)). This is followed by the water-gas reaction (Eq. (3)) and the water-gas shift reaction (Eq. (4)) which occur simultaneously with the Boudouard reaction (Eq. (5)). In the Eq. (6), the formation of potassium carbonate (K_2CO_3) results from the reaction of K_2O with CO_2. At carbonization temperatures above 500°C, a large amount of metallic potassium is generated (Eq. (7) and Eq. (8)). Metallic potassium is mobile. It penetrates the carbon structure of the carbon matrix during activation at temperatures above 500°C. After removal of K metal due to neutralization, the carbon structure cannot revert to its original form, leading to the formation of a highly porous carbon material. The overall reaction between carbon and KOH that occurs during activation is shown in Eq. (9).

$$2\,\mathrm{KOH} \rightarrow \mathrm{K_2O} + \mathrm{H_2O} \tag{2}$$
(dehydration)

$$\mathrm{C} + \mathrm{H_2O} \rightarrow \mathrm{CO} + \mathrm{H_2} \tag{3}$$
(water-gas reaction)

$$\mathrm{CO} + \mathrm{H_2O} \rightarrow \mathrm{CO_2} + \mathrm{H_2} \tag{4}$$
(water-gas shift reaction)

$$\mathrm{C} + \mathrm{CO_2} \rightarrow 2\,\mathrm{CO} \tag{5}$$
(boudouard reaction)

$$\mathrm{K_2O} + \mathrm{CO_2} \rightarrow \mathrm{K_2CO_3} \tag{6}$$
(carbonate formation)

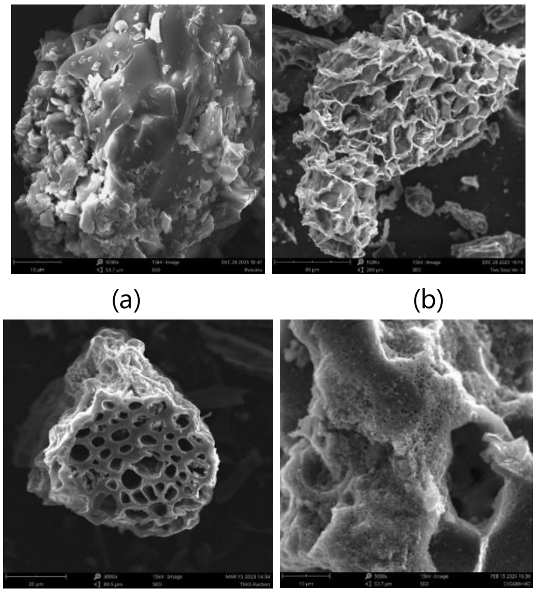
$$\mathrm{K_2O} + \mathrm{H_2} \rightarrow 2\,\mathrm{K} + \mathrm{H_2O} \tag{7}$$
(reduction by hydrogen)

$$\mathrm{K_2O} + 2\,\mathrm{C} \leftrightarrow 2\,\mathrm{K} + 3\,\mathrm{CO} \tag{8}$$
(reduction by hydrogen)

$$6\,\mathrm{KOH} + 2\,\mathrm{C} \rightarrow 2\,\mathrm{K} + 3\,\mathrm{H_2} + 2\,\mathrm{K_2CO_3} \tag{9}$$
(overall reaction)

The microstructure and morphology of the activated carbon were observed using SEM as shown in Figure 1. Based on the test results, the cell size and cell distribution appear heterogeneous and form a clear pore structure. In the SEM test, electrons are fired at the carbon sample, and the scattered electrons and secondary electrons emitted by the sample will form an image. The stages of the carbonization process, chemical activation using KOH, and physical activation help destroy the cell walls of the palm kernel shell so that the pore lattice becomes weak, which causes the pores to open. The pore opening is visible and can be observed from the test results using SEM.

At an impregnation ratio of 0.25, the SEM images show a carbon surface that begins to show pore formation, but the pore structure is not yet very developed. The pores that are formed tend to be uneven and are still dominated by a relatively smooth surface with little scraping. This indicates that at this ratio, the KOH activator is not yet fully effective in creating an optimal micropore structure, so the specific surface area is still limited. With an increase in the impregnation ratio to 0.5, a significant increase in pore formation was observed. In SEM, the carbon surface shows a more even distribution of pores and micropores, and mesopores begin to form. This more open structure indicates that the higher amount of KOH accelerates the activation reaction, so the carbon is more eroded, and more voids are formed. At this ratio, there is a good balance between pore formation and carbon structure stability (Husain *et al.*, 2023). At an impregnation ratio of 1.0, SEM shows a highly eroded carbon surface with many pores formed, both on the micro and mesopore scale. The surface structure is rougher, and there is deeper erosion, indicating that at higher KOH concentrations, the chemical reaction between KOH and carbon is more aggressive. Despite the increased porosity, excess KOH can cause over-erosion, potentially reducing the mechanical strength of the activated charcoal (Triana *et al.*, 2025). At this point, although the surface area increases, attention is needed to the potential for excessive degradation of the carbon structure.



(a) (b)

(a) (b)

Fig. 1: SEM Surface Morphology of Char Particles (a) Char without KOH Activation; (b) Char with 0.25 ratio KOH activation; (c) Char with 0.5 ratio KOH activation and (d) Char with 1.0 ratio KOH activation

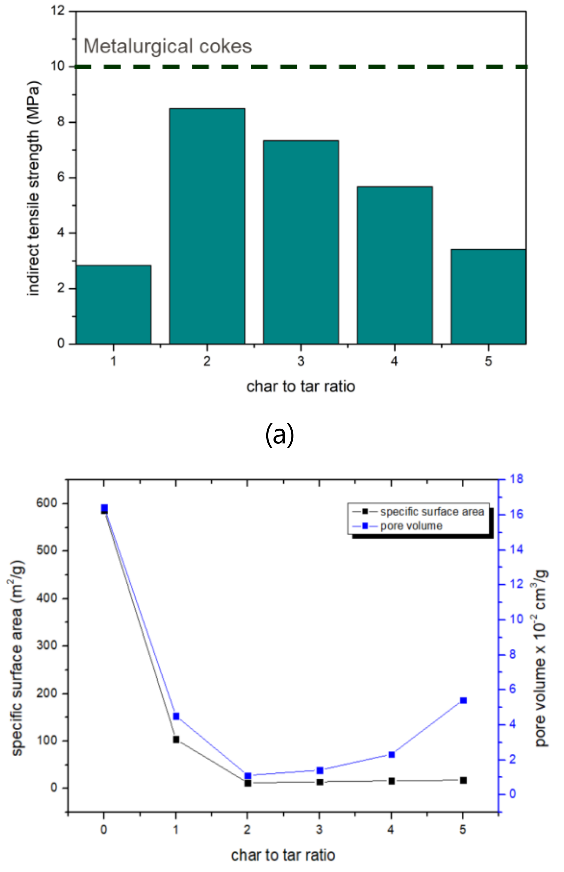
Effect of Char-Oils Ratio for Preparation of C/C Composite

The ratio of char to tar (or oils) plays a critical role in determining the properties of the resultant carbon-carbon (C/C) composite. In this study, the ratios explored ranged from 1:1 to 1:5, and the indirect tensile strength was used as a key metric for evaluating composite performance.

The co-carbonization process at low temperature (250 ^{o}C) successfully facilitates the deposition of tar onto the char, significantly altering its structural properties. This is evident from the results of BET surface area analysis, which show a dramatic reduction in both surface area and pore volume. The data indicate that the tar effectively fills the pores of the char, resulting in a denser and less porous material (Mochizuki *et al.*, 2024). Approximately 2 nm tar molecules [i.e., low molecular composition aromatic compounds (molecular size: approximately 1.0-3.0 nm)] are loaded into ~4 nm pores of Char (Abe *et al.*, 2018).

Furthermore, pore size measurements reveal that the majority of pores are nearly eliminated, suggesting that the co-carbonization process creates a compact carbon matrix with minimal voids (Yuuki Mochizuki and Naoto Tsubouchi, 2022). This transformation enhances the composite's mechanical properties, as observed in the improved indirect tensile strength.

The impact of the char: tar ratio on the indirect tensile strength of the C/C composite reveals a clear trend shown in Figure 2. At a ratio of 1:1, the tensile strength is relatively low, measured at 2.8 MPa; the composite exhibits high char content, which provides a rigid carbon structure. However, excessive char might lead to poor binding due to insufficient tar to act as a matrix material, resulting in lower tensile strength (Mochizuki *et al.*, 2016). The structure could also be more porous and less uniform, limiting the load-bearing capacity.



(a)

(b)

Fig 2. Physical characteristic of CC Composite as char to tar ratio (a) indirect tensile strength; (b) pore characteristic

As the ratio increases to 1:2, the tensile strength reaches its peak at 8.5 MPa, suggesting an optimal balance between the structural contribution of char and the binding capability of tar. However, further increases in tar content, such as at 1:3 (7.3 MPa), 1:4 (5.6 MPa), and 1:5 (3.4 MPa), result in a gradual decline in tensile strength. During co-carbonization, excessive tar might also result in the formation of volatile by-products, increasing porosity, and reducing the overall mechanical integrity of the composite (Mochizuki *et al.*, 2018). These factors could explain the decline in tensile strength at higher tar proportions. The performance at a 1:2 ratio, being close to commercial coke, suggests that this ratio achieves a similar microstructure and density, crucial for high tensile strength.

Proximate and Gross Calorific Value of CC Composites

Figure 3 illustrates the proximate analysis of four materials: raw char, wet composite (CC) with a 1:2 ratio, final composite (CC) after co-carbonization with a 1:2 ratio, and commercial coke. The parameters analyzed include moisture (M), volatile matter (VM), ash content (AC), and fixed carbon (FC), providing insights into the transformation of char into a bio-coke alternative.

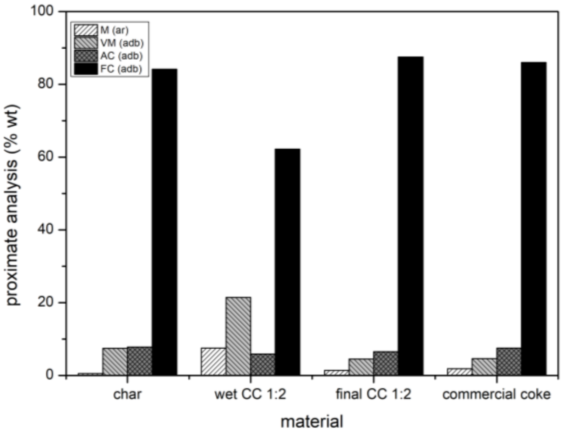


Fig. 3 Proximate analysis of bio-coke product. M: Moisture, VM: Volatile Matter, AC: Ash Content, FC: Fixed Carbon

The transformation process begins with raw char, which is mixed with a binder at a 1:2 ratio to form wet composite briquettes. At this stage, the wet composite retains higher moisture content and some volatile matter compared to the final product because the addition of tar, which has high amounts of organic compounds (Flores *et al.*, 2017). During co-carbonization, these wet composites undergo thermal decomposition in a controlled atmosphere. This process facilitates the release of volatile matter through devolatilization and the reduction of moisture. Consequently, the fixed carbon (FC) content significantly increases, as observed in the final composite. The co-carbonization also contributes to the stabilization of the material, reducing its ash content (AC) and enhancing its structural integrity, thus improving its suitability as a bio-coke (Zhang *et al.*, 2024).

The final composite closely matches the proximate composition of commercial coke, particularly in terms of fixed carbon content, which exceeds 80% in both materials. This high FC content is critical for industrial applications such as metallurgy, where coke serves as a carbon source and structural support in blast furnaces (Surup *et al.*, 2022). The observed reduction in VM and AC during co-carbonization indicates an effective carbonization process, minimizing impurities and enhancing calorific value. The comparison underscores the effectiveness of co-carbonization in converting char-based composites into high-quality bio-coke, positioning it as a sustainable alternative to commercial coke in industries requiring carbon-intensive inputs. This process leverages waste or low-value materials like char, contributing to resource efficiency and environmental sustainability.

Reduction Behaviour of CC Composites for Laterite Ore

According to Figure 4, nickel is present as goetite-(Fe, Ni)OOH and olivine-$MgNiO_4Si$ in limonitic ore. Lizardite-$H_4Mg_3O_9Si_2$ and quartz-SiO_2 are the impurities. The phase changes observed during the reduction of limonitic nickel ore with carbon are primarily due to the thermochemical reactions occurring as the temperature increases. Wustite, ferronickel, quartz, iron sulfide, and magnesioferrite are all present, as seen in Fig. 4. There is no quartz peak at 700°C and 900°C, but when the temperature is elevated to 1100°C, a quartz peak is detected, albeit the wustite intensity is reduced (Sari et al., n.d.).

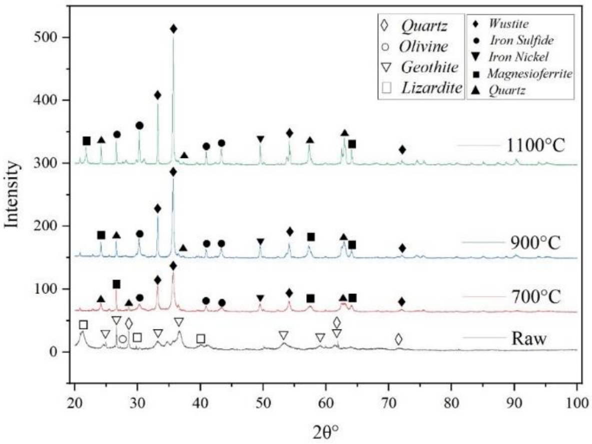


Fig 4. Reduction behaviour of CC composites

During the reduction process, carbon acts as a reducing agent, converting metal oxides into their metallic or lower oxidation state forms. Goethite (Fe, Ni)OOH decomposes at high temperatures, releasing water and forming iron oxides such as wustite (FeO). Olivine $MgNiO_4Si$ undergoes structural transformations, potentially releasing nickel and forming other silicates or oxides. At higher temperatures, the system may reach equilibrium conditions that favor the recrystallization or reformation of quartz. Additionally, some silicate phases formed at lower temperatures might decompose back into quartz (Suharno *et al.*, 2020).

CONCLUSION

The carbonized char can be enhanced using tar produced during the pyrolysis of woody biomass. This modified char-coke composite (CC) is created through a heat treatment process involving a mixture of char and tar recovered from sawdust pyrolysis. During CC preparation, the mesopores within the char are filled with carbonaceous materials derived from the tar. However, increasing the tar content negatively impacts the indirect tensile strength of the resultant coke. The study determined that the optimal conditions for producing high-strength coke with desirable proximate characteristics—comparable to commercial cokes—were achieved at a char-to-tar ratio of 1:2 and an appropriate preparation temperature.

This suggests that carefully controlling the composition and processing parameters can produce high-performance bio-coke suitable for industrial applications.

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NOMENCLATURE

f : Indirect tensile strength [Mpa]
d : diameter [m]
l : Thickness [m]
P : Failure load [N]
adb : as dried basis
arb : as received basis
AC : ash content [%wt]
FC : fixed carbon [%wt]
M : moisture content [%wt]
VM : volatile matter [%wt]
GCV : Gross calorific value [cal/g]

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