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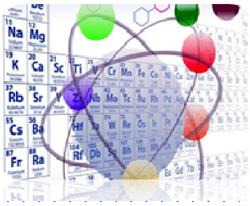

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Potential of Bituminous Coal to Become Nanocarbon Using the Hydrothermal Method

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ABSTRACT

This research investigates the potential of bituminous coal from Sawahlunto, Indonesia, as a raw material for nanocarbon synthesis using a hydrothermal method. The study aims to enhance the economic value of coal by exploring its transformation into nanocarbon. The process involves stages of preparation, carbonization, and hydrothermal at varying temperatures (120°C, 150°C, and 180°C) durations (2 and 4 hours). Fourier Transform Infrared (FT-IR) spectroscopy, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM) were employed to characterize the resulting nanocarbon. The hydrothermal process improved the crystallinity of coal from 51% to 57% and reduced its particle size to 47.71 nm. SEM and TEM analyses revealed a more uniform and refined morphology in the nanocarbon compared to raw coal. The study underscores the effectiveness of the hydrothermal method in optimizing the physical and structural properties of coal-derived nanocarbon, paving the way for its potential application in various industries.

Keywords: Bituminous coal, Nanocarbon, Hydrothermal method, Crystallinity, Advanced matrial.

1. INTRODUCTION

Coal is one of the world's most abundant and versatile natural resources, serving as a primary energy source and raw material for various industrial processes. Indonesia, as one of the largest coal producers, has an estimated coal resource of 207.54 billion tons, primarily comprising surface and subsurface reserves[1]. However, the current utilization of Indonesian coal remains suboptimal, with most of it directed toward energy production, particularly in coal-fired power plants. Only a small portion is allocated for non-energy purposes, necessitating innovative approaches to diversify and enhance its usage[2].

Bituminous coal, a high-carbon-content material, holds great potential as a precursor for advanced materials, including nanocarbon[3]. Nanocarbon materials, such as graphene, carbon nanotubes (CNT), and carbon nanofibers, have garnered significant attention due to their unique properties, including high electrical conductivity, thermal stability, and exceptional mechanical strength[4]. These characteristics make nanocarbon ideal for applications in electronics, energy storage, composites, and various advanced technologies[5].

The development of nanotechnology has enabled the transformation of coal into high-value nanocarbon materials, providing a sustainable pathway to utilize this abundant resource[6]. Among various methods, the hydrothermal process offers distinct advantages, such as lower energy consumption, environmentally friendly procedures, and the ability to tailor nanocarbon properties by controlling reaction parameters[7].

This study focuses on exploring the potential of bituminous coal from Sawahlunto, Indonesia, as a raw material for nanocarbon production through the hydrothermal method. The research aims to optimize the conditions for nanocarbon synthesis, including temperature and reaction time, and evaluate the resulting product's physical and chemical characteristics. By addressing these objectives, this research contributes to the sustainable utilization of Indonesian coal, enhancing its economic value and supporting technological advancements in various industries[5].

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The materials used in this study included bituminous coal sourced from Sawahlunto, West Sumatra, aquadest (distilled water), and ethanol. The equipment comprised a ball mill (Retch PM100) for grinding, a 325-mesh sieve for particle size uniformity, an oven for drying, a furnace for carbonization, an ultrasonicator for exfoliating graphite, and a hydrothermal reactor for nanocarbon synthesis. Characterization tools included Fourier Transform Infrared (FT-IR) spectroscopy for functional group analysis, X-Ray Diffraction (XRD) for determining crystallinity and particle size, Scanning Electron Microscopy (SEM) for surface morphology analysis, and Transmission Electron Microscopy (TEM) for microstructural observation.

2.2. Preparation of Graphite from Sawahlunto Coal

The procedure began with coal preparation, where the raw coal was crushed using a ball mill for 4 hours and then sieved to achieve a uniform particle size of 325 mesh. The sieved powder was dried in an oven at 105–110°C for 24 hours to remove moisture.

2.3. Optimization of Graphite Carbonization from Sawahlunto Coal

The graphite powder derived from Sawahlunto coal, which initially exhibited amorphous properties, was subjected to a carbonization process to enhance its crystallinity. The carbonization was conducted at incremental temperatures of 200°C, 400°C, and 600°C, with each temperature maintained for 15 minutes. For each condition, 50 grams of graphite powder was weighed and placed into porcelain crucibles, which were then heated in a furnace. This process facilitated the removal of volatile components, including hydrogen, oxygen, and amorphous hydrocarbons, through pyrolytic decomposition. As these volatile elements were eliminated, the carbon atoms formed stronger interatomic bonds, leading to the development of a more crystalline graphite structure.

The carbonized graphite was subsequently subjected to sonication using an ultrasonic device to exfoliate and refine its pore structure. Sonication selectively removed particles adhered to the surface, improving the material's quality. The optimized carbonization process played a critical role in preparing graphite as a precursor material for hydrothermal nanocarbon synthesis, ensuring higher crystallinity and structural uniformity in the final product.

2.5 Hydrothermal Synthesis of Nanocarbon

For nanocarbon synthesis, 10 g of graphite powder was mixed with water at a 1:10 (m/v) ratio and subjected to hydrothermal treatment at varying temperatures (120°C, 150°C, and 180°C) for 2 and 4 hours. The resulting product was then processed using an ultrasonicator to produce nanocarbon.

2.6 Characterization of Nanocarbon

Characterization was performed to evaluate the properties of the nanocarbon. FT-IR spectroscopy identified functional groups, while XRD analysis measured crystallinity, interlayer spacing, and particle size using the Debye-Scherrer equation. SEM and TEM analyses revealed the surface morphology and microstructural properties of the samples. The experimental parameters were systematically varied, with six experimental runs involving different combinations of temperature and reaction time, as shown in the design: 120°C, 150°C, and 180°C for 2 and 4 hours.

3. RESULTS AND DISCUSSION

3,1 Synthesis of Nanocarbon from Sawahlunto Coal

The synthesis of nanocarbon from Sawahlunto coal was carried out in several stages: preparation, carbonization, and hydrothermal treatment.

In the preparation stage, large chunks of coal were reduced in size and then heated at 125°C for 24 hours to remove moisture. The coal chunks were subsequently carbonized in stages at temperatures ranging from 200°C to 600°C. The carbonization process served to reduce the amorphous properties of the coal's graphite, enhancing its crystalline nature, thereby making it more similar to crystalline graphite, which is a precursor material for producing graphene oxide and graphene[9].

During carbonization, volatile substances such as hydrogen, oxygen, and unorganized hydrocarbons were released due to the pyrolytic decomposition of coal. This process also facilitated the removal of amorphous aliphatic hydrocarbons, causing the carbon atoms to accumulate and form interatomic bonds, thereby creating a crystalline structure with elementary graphite crystals. Approximately 80% of the carbon content was retained at temperatures between 400°C and 600°C[10].

The final stage was the hydrothermal treatment, conducted at varying temperatures of 120°C, 150°C, and 180°C, with reaction times of 2 and 4 hours. This process produced graphene oxide (GO), which was subsequently subjected to ultrasonication using an ultrasonic device to create nano-sized GO or nanocarbon.

In principle, this method produces carbon by altering solubility, melting crystalline parts, accelerating physicochemical interactions, facilitating acid/base or ionic reactions, and precipitating carbon material[11]. This approach effectively transforms Sawahlunto coal into nanocarbon with potential applications in advanced material science.

3.2. Characterization of Functional Groups Using Fourier Transform Infrared (FT-IR)

Fourier Transform Infrared (FT-IR) analysis was performed to identify the presence of functional groups in Sawahlunto coal and the graphite produced at various carbonization temperatures. The FT-IR spectra for coal and nanocarbon are shown in Figures 4.1 and the identified functional groups are summarized in Table 4.1.

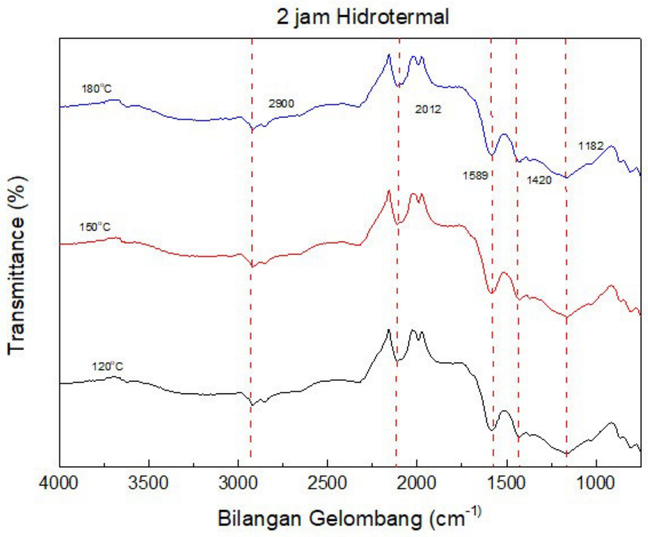


Figure 1. FT-IR Spectrum of Coal-Based Nanocarbon at a 2-Hour Variation

Table 1. Functional Groups in Coal and Nanocarbon with a 2-Hour Variation

Sample	Wavelength (cm^{-1})	Transmittance (%)	Functional Group
Coal	3650–3250	37.91–28.97	Hydrogen bond (-OH), water content (H_2O)
	2911	24.17	Saturated aliphatic group (C-H), asymmetric
	2841	25.12	Methoxy ($-OCH_3$)
	1432	23.80	Methyl group ($-CH_3$), asymmetric
Nanocarbon 120°C	3623	70.83	Alcohol and hydroxy compound
	2109.7	74.87	Acetylenic group (-C≡C-)
	2922.2	66.78	Saturated aliphatic group (C-H), asymmetric
	1431.3	60.17	Methyl group ($-CH_3$), asymmetric
Nanocarbon 150°C	3602	72.96	Alcohol and hydroxy compound
	2117	76.42	Acetylenic group (-C≡C-)
	2922.2	67.94	Saturated aliphatic group (C-H), asymmetric
	1431.3	61.93	Methyl group ($-CH_3$), asymmetric
Nanocarbon 180°C	3637	73.20	Alcohol and hydroxy compound
	2117	76.37	Acetylenic group (-C≡C-)
	2918	70.85	Saturated aliphatic group (C-H), asymmetric
	1432	62.11	Methyl group ($-CH_3$), asymmetric

*[12]

In Figure 1, as highlighted in Table 1, it is evident that the transmittance values for the temperature variations of 120°C, 150°C, and 180°C over 2 hours differ at each wavenumber. This indicates that at a temperature variation of 180°C, the highest loss of amorphous molecules occurred compared to the other temperature variations. Transmittance reflects the amount of infrared radiation transmitted through a molecule. The higher the transmittance value, the fewer molecules absorb the infrared radiation[13]. Therefore, it can be concluded that nanocarbon produced at 180°C is the best.

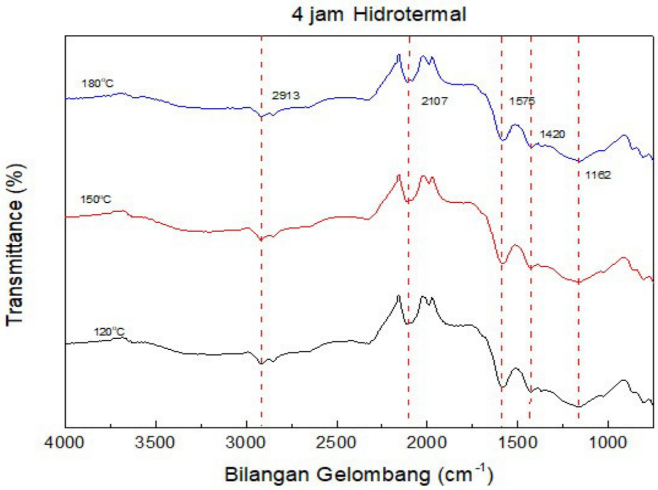


Figure 2. FT-IR Spectrum of Coal-Based Nanocarbon at a 2-Hour Variation

Table 2. Functional Groups in Coal and Nanocarbon with 4-Hour Variations

Sample	Wavenumber (cm⁻¹)	Functional Group	Observation
Coal	3650–3250	Hydrogen bond (-OH), water content (H_2O)	Broad absorption, indicating water content
	2911	Saturated aliphatic methylene (-CH)	Indicates asymmetric C-H bonds
	2841	Methoxy ($-OCH_3$)	Absorption typical of methoxy groups
	1432	Methyl ($-CH_3$), asymmetric	Indicates methyl group bonding
Nanocarbon 120°C	3623	Alcohol and hydroxy compound	Strong absorption indicates hydroxy presence
	2109.7	Alkyne (-C≡C-)	Characteristic of acetylene groups
	2922.2	Saturated aliphatic methylene (-CH)	Asymmetric C-H bond absorption
	1431.3	Methyl ($-CH_3$), asymmetric	Indicates methyl groups
Nanocarbon 150°C	3602	Alcohol and hydroxy compound	Increased intensity of hydroxy compounds
	2117.1	Alkyne (-C≡C-)	Absorption characteristic of alkyne groups
	2922.2	Saturated aliphatic methylene (-CH)	Strong asymmetric C-H bond absorption
	1431.3	Methyl ($-CH_3$), asymmetric	Methyl group bonding clearly observed
Nanocarbon 180°C	3637	Alcohol and hydroxy compound	Strong absorption indicates hydroxy presence
	2117	Alkyne (-C≡C-)	Prominent alkyne absorption
	2918	Saturated aliphatic methylene (-CH)	Strong asymmetric C-H bond
	1432	Methyl ($-CH_3$), asymmetric	Indicates methyl group bonding

*[12]

In Figure 2, as highlighted in Table 2, it is evident that the transmittance values for the temperature variations of 120°C, 150°C, and 180°C over 4 hours differ at each wavenumber. This indicates that at a temperature variation of 180°C, the highest loss of amorphous molecules occurred compared to the other temperature variations. Therefore, it is concluded that nanocarbon produced at 180°C is the best.

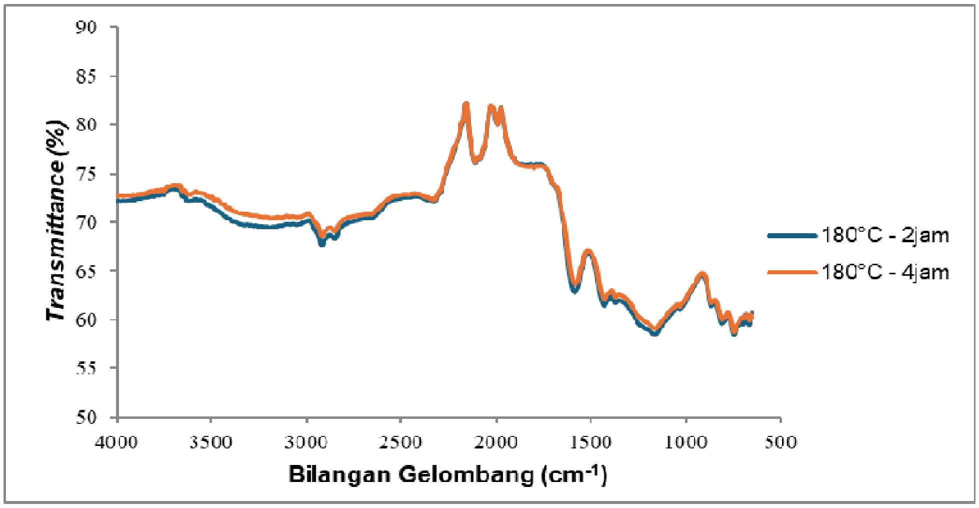


Figure 3. Shows A Comparison Of The FT-IR Spectra Of Nanocarbon Synthesized At A Hydrothermal Temperature Of 180°C For 2 Hours And 4 Hours.

It can be observed that the spectrum of nanocarbon synthesized for 4 hours has higher transmittance, as indicated by the FT-IR spectrum being positioned above the spectrum of nanocarbon synthesized for 2 hours. Transmittance reflects the amount of infrared radiation transmitted through a molecule, where higher transmittance values indicate fewer molecules absorbing the infrared radiation[13].

Thus, nanocarbon synthesized for 4 hours experienced a greater loss of amorphous components, resulting in a better nanocarbon structure. Consequently, in this study, nanocarbon obtained after 4 hours is determined to be the best. This nanocarbon will be further characterized in terms of its crystallinity, morphology, and microstructure.

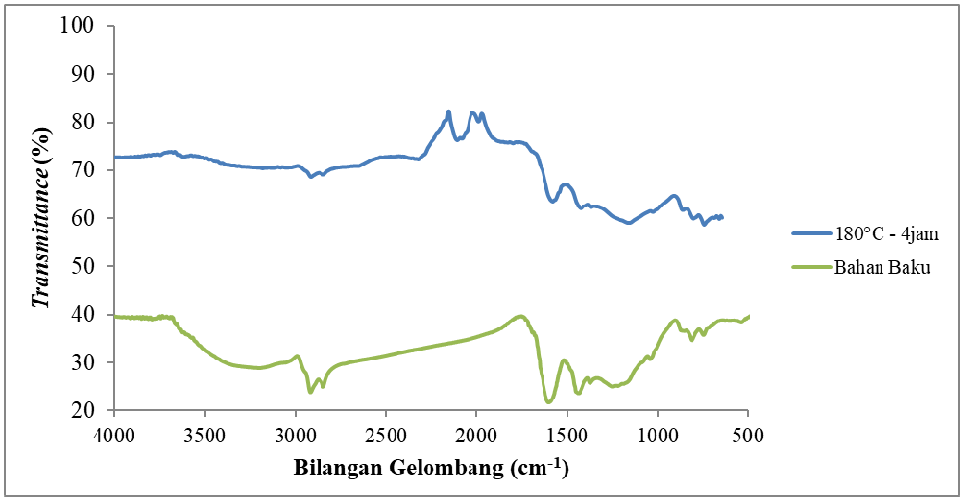


Figure 4. FT-IR Spectrum of Produced Nanocarbon and Coal

In the FT-IR analysis procedure, the sample is exposed to infrared (IR) radiation. The IR radiation affects the vibration of atoms in a molecule within the sample, resulting in the absorption and/or transmission of specific energy levels. This makes FT-IR useful for identifying specific molecular vibrations present in the sample[14].

It can thus be concluded that the hydrothermal method successfully transformed coal into nanocarbon, as evidenced by the loss or reduction of amorphous bonds in the coal, demonstrated through FT-IR analysis.

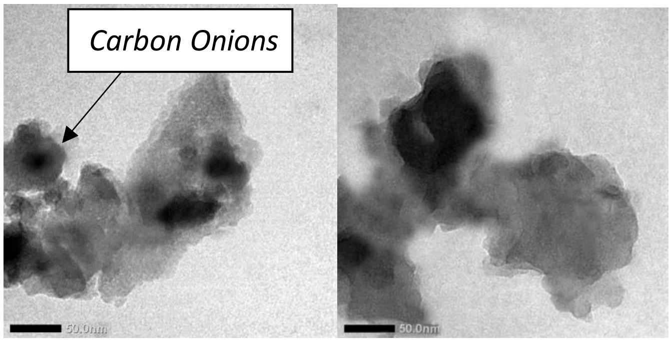
3.3. Electron Microscopy (EM) Characteristics

Electron Microscopy (EM) is a technique used to examine the morphology of a material. Morphological information, including the shape and size of catalysts and the resulting products, is essential for characterizing the nanocarbon produced. EM techniques are primarily divided into Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). Both techniques operate on the principle of scanning a material's surface with an electron beam, capturing the reflected electrons, and displaying the image on a cathode-ray tube. The resulting image represents the surface morphology of the specimen.

SEM provides an electron microscopy resolution of approximately 10 nm, making it suitable for observing larger-scale surface features. TEM, on the other hand, offers higher resolution, allowing for detailed visualization of structural characteristics. At low magnifications, TEM images display contrast due to electron absorption by the material, influenced by the thickness and composition of the material. At higher magnifications, TEM reveals detailed data about the crystal structure and other fine features.

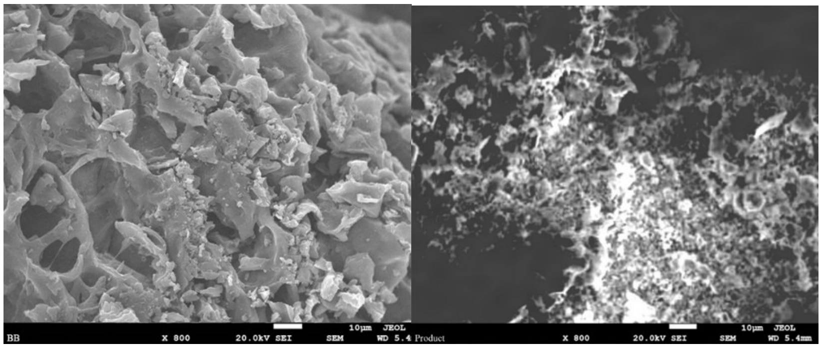
TEM generates images in either dark field (DF) or bright field (BF) modes using a focused electron beam for imaging. A BF or DF image is formed when a specific diffraction beam is used while the objective aperture blocks other diffraction beams from entering the recording system. SEM, on the other hand, scans a sample surface using an electron beam (5–50 kV) with magnifications ranging from 20x to 50,000x and a resolution of approximately 5 nm. SEM can provide 3D-like effects for samples with deep areas or highly irregular structures. However, its resolution limits SEM's ability to resolve crystals smaller than 5 nm.

Through SEM and TEM analyses, the morphology, size, and structural characteristics of both raw coal and the resulting nanocarbon can be observed. TEM is particularly advantageous for examining fine crystalline structures, while SEM is suitable for larger-scale surface morphology and distribution analysis.



(a) (b)

Figure 5. TEM Results: (a) Coal, (b) Nanocarbon



(a) (b)

Figure 6. SEM Results: (a) Coal, (b) Nanocarbon

The images above show the TEM results for coal and nanocarbon at a temperature of 180°C for 4 hours.

Based on its morphology in Figure 5 (a), it can be observed that coal graphite does not yet display graphene sheets, as evidenced by the presence of numerous stacked layers. This indicates that coal graphite can be further exfoliated to produce graphene-like nanocarbon[15]. However, Carbon Onion Quasi-Spheres, concentric graphite layers resembling spheres with a size of approximately 50 nm, were detected. These results align with previous research by Purwandari (2020)[16], which reported no graphene sheets under similar conditions but observed stacked layers. Similarly, Ribka (2012)[2] identified Carbon Onion Quasi-Spheres, albeit with different particle sizes.

In contrast, Figure 5 (b) clearly shows that nanocarbon exhibits a morphology with varying sheet transparency. This may be attributed to the number of layers within the nanocarbon structure, with some parts of the coal graphite remaining unoxidized. TEM analysis reveals that the sheet morphology consists of multiple partially oxidized graphite oxide layers[16]. These findings are consistent with Purwandari (2020), who also reported the presence of sheets with different transparencies, indicating graphene sheets approximately 50 nm in size after hydrothermal treatment of coal.

The SEM results in Figure 6 (a) for coal at 800x magnification reveal an uneven morphology. The surface shows clumps or aggregates of coal particles with irregular sizes. Similar morphological features were reported by Purwandari et al. (2020), who also studied the synthesis of graphene oxide from Sawahlunto coal. Compared to nanocarbon in Figure 6 (b), it is evident that nanocarbon morphology at 800x magnification is much smoother and more uniform than that of coal. The nanocarbon morphology from Sawahlunto coal appears smooth and entangled. This may be due to the reconstruction of new carbon chemical bonds caused by oxidation, leading to lattice defects from the formation of alkoxy groups[14].

3.4. Crystallinity Characterization Using X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis was conducted to determine the crystallinity degree of Sawahlunto coal and the resulting nanocarbon. The results are presented in Figure 7. The carbon structure analysis was performed on coal and nanocarbon in powdered form, screened through a 325-mesh sieve, using a Shimadzu 7000 instrument. The conditions employed included a copper (Cu) radiation source, energy of 40 kV, current of 30 mA, scanning speed of 2°/minute, data recorded every 0.02°, and a scanning angle range between 7° and 70°.

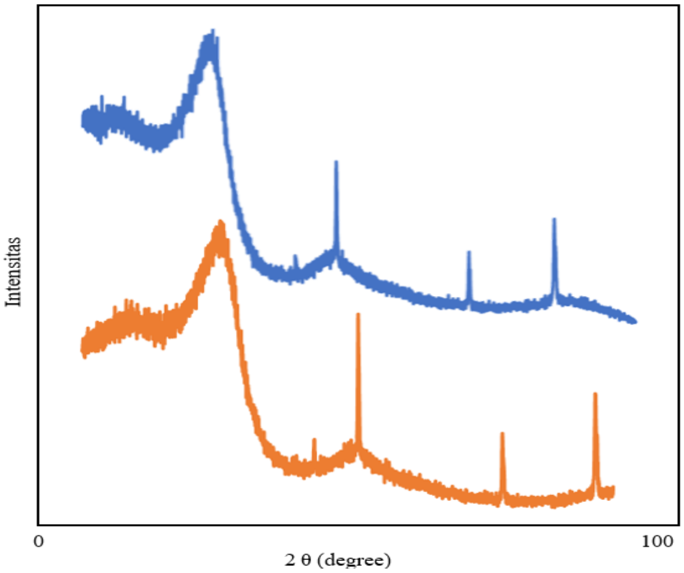


Figure 7. XRD Spectrum of Coal and Nanocarbon

According to Saikia et al. (2009), the parameters analyzed were the degree of crystallinity (X), the interlayer distance of aromatic carbon (d_{002}), the aromatic layer height (Lc), the aromatic layer width (La), and the number of nanocarbon layers (N). These parameters were calculated using Bragg's and Scherrer's formulas.

Table 3. Crystallinity Parameters of Coal and Nanocarbon

Sample	Peak 2θ (°)	θ (°)	Minimum Interlayer Distance (d_{002}, nm)	Aromatic Layer Height (Lc, nm)	Crystallinity Degree (X, %)
Coal	23.22	11.63	0.381	0.139	51.173
Nanocarbon	25.02	12.51	0.355	0.140	57.688

From Table 3, it is shown that the carbonization process applied to Sawahlunto coal did not result in significant changes. The coal exhibited a crystallinity degree of 51% with a minimum interlayer distance of 0.13993 nm. Meanwhile, the nanocarbon showed a crystallinity degree of 57% with a minimum interlayer distance of 0.14039 nm.

This indicates an increase in crystallinity and minimum interlayer spacing in the nanocarbon obtained. The hydrothermal treatment resulted in an increase in crystal size and the elimination of defects due to the rearrangement of carbon atom structures within the carbon material. The hydrothermal method also led to an increase in the height of aromatic layers (Lc), contributing to the growth of crystal planes and the formation of crystal layers. Amorphous carbon structures were transformed into semi-crystalline carbon

From the XRD spectrum (Figure 3.7), it can be observed that Sawahlunto coal contains carbon, as evidenced by the peak at 2θ = 23.22°, reinforced by another peak at 2θ = 43.98°. These 2θ peaks are characteristic of graphite[11].

In addition to determining the degree of crystallinity, XRD analysis also aims to measure the particle diameter of the resulting nanocarbon. A similar determination of nanocarbon particle diameter was conducted in a study by Purwandari (2020) using the Debye-Scherrer equation as follows:

$$D = \frac{K\lambda}{\beta \cos\theta}$$

where:

D = Particle diameter (nm)

K = Scherer constant (0,9)

λ = X-ray wavelength (0,1540 nm)

β = Full Width at Half Maximum (FWHM)

θ = Diffraction angle

Based on calculations, the particle diameter of nanocarbon was found to be 47.71 nm. This result aligns with TEM analysis, which showed that the nanocarbon particle diameter was approximately 50 nm. In contrast, the coal sample used in this study, which passed through a 325-mesh sieve (44,000 nm), demonstrated that the hydrothermal process effectively reduced the coal particle size to the nanoscale.

4. CONCLUSION

This study demonstrated the potential of Sawahlunto coal as a precursor for nanocarbon synthesis using the hydrothermal method. FT-IR analysis confirmed the transformation into nanocarbon, indicated by reduced amorphous bonds and the presence of nanocarbon-specific functional groups. SEM and TEM analyses showed morphological changes, with nanocarbon exhibiting smoother surfaces and partially oxidized graphite oxide sheets, compared to the irregular morphology of raw coal. XRD analysis revealed an increase in crystallinity from 51% to 57% and reduced particle size from 44,000 nm to approximately 50 nm. Optimal synthesis conditions (180°C, 4 hours) produced high-quality nanocarbon for advanced material applications.

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