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Preparation and Characterization of Biochar from Coconut Shells with The Addition of Cu Metal as a Heterogeneous Catalyst

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ABSTRACT

Biochar is a carbon-rich solid produced by pyrolyzing biomass in the absence of oxygen. This study prepared and characterized coconut shell biochar modified with copper (Cu) as a heterogeneous catalyst. Coconut shells were selected for their high carbon content and porous structure. Biochar was produced by pyrolysis at 500 °C for 2 h under nitrogen, activated with KOH (1:4 w/w), and calcined at 500 °C. Copper was introduced by impregnating the activated biochar with a $Cu(NO_3)_2{\cdot}6H_2O$ solution, stirring for 5 h, followed by calcination at 500 °C. Characterization using FTIR, XRD, SEM-EDX, and BET identified functional groups, crystal structure, morphology, elemental composition, and surface area. Results indicate that Cu addition enhances functional group intensity, sharpens XRD peaks, increases surface area, and promotes more uniform pore distribution. These changes suggest improved catalytic potential for biomass conversion, particularly in steam reforming applications.

Keywords: biochar, coconut shell, copper catalyst, KOH activation, steam reforming

1. INTRODUCTION

Indonesia is the world's largest coconut producer, with most of the production coming from smallholder plantations. In 2020, Indonesian coconut production reached 2,811,954 tons across a plantation area of 3,396,776 ha.[1] This high coconut production is directly proportional to the amount of coconut shell waste produced. Generally, coconut shells are discarded or burned by farmers to use as fertilizer, but this process produces CO_2 and CH_4 gases, which negatively impact the environment.[2] Furthermore, the coconut processing industry, which produces oil, health products, energy, and food, contributes to the increasing volume of coconut shell waste. Therefore, effective management solutions are needed to prevent environmental pollution. One possible alternative is utilizing this waste as an alternative energy source through a thermochemical process, which not only reduces environmental impact but also increases its economic value.[3]

Processing coconut shell waste into value-added products such as biochar is a potential solution. Biochar is a solid carbon material produced through the pyrolysis of biomass at high temperatures with little or no oxygen.[4] This material has a porous structure, is chemically stable, has a high surface area, and contains various

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active functional groups[5,6], making it suitable for use as a pollutant adsorbent, catalyst support, and soil conditioner.[7,8] The biomass pyrolysis process produces three main products: biochar, bio-oil, and gas. Biochar is widely used for gas absorption,[9] soil improvement,[10] and wastewater purification.[11]

To maximize the performance of biochar in environmental and energy applications, an activation process is required to improve its physicochemical properties, such as porosity and the number of active sites. One effective method is chemical activation using a strong base such as potassium hydroxide (KOH). Activation with KOH can increase the specific surface area, enlarge the pore volume and distribution, and generate active functional groups that enhance the biochar's adsorption capacity.[10.11]

In heterogeneous catalysis, the addition of active metals to carbon supports is a common strategy to improve catalyst performance.[12] Copper (Cu) is an attractive choice due to its relatively low cost, high activity in reforming and hydrogenation reactions, and good selectivity for hydrogen production from biomass-based materials.[13,14] Furthermore, Cu exhibits high catalytic activity in methanol synthesis, water-gas shift reactions, and steam reforming at medium temperatures.[15] The addition of Cu to biochar has the potential to enhance catalytic activity by improving metal dispersion and enhancing metal-support interactions.[16]

Despite the significant potential of coconut shells and Cu metal, research systematically examining the preparation and characterization of Cu-modified coconut shell biochar for catalyst applications is still limited. Therefore, this study aimed to prepare and characterize coconut shell biochar activated with KOH and impregnated with Cu metal through a wet impregnation method followed by calcination. Characterization was performed using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscopy–Energy Dispersive X-ray Spectroscopy (SEM-EDX), and Brunauer–Emmett–Teller (BET) surface area analysis.[17,18] The results of this study are expected to contribute to the development of efficient, economical, and environmentally friendly carbon-based catalysts for biomass conversion applications, particularly in steam reforming processes.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The materials used in this study were coconut shell, HCl, metal ($Cu(NO_3)_2.3H_2O$), distilled water, KOH, filter paper, and universal pH. The equipment used were beakers, measuring cylinders, Erlenmeyer flasks, mortar and pestle, 200 mesh sieve, reflux apparatus, spatula, analytical balance, oven, hot plate, grinder, Fourier Transform Infrared (FTIR), X-ray diffraction (XRD), and Brunauer-Emmett-Teller (BET).

2.2. Coconut Shell Biomass Preparation

Coconut shells collected from the market are cleaned of fibers attached to their surfaces. They are then washed under running water and dried in the sun for 24 hours. The dried coconut shells are shredded and dried again in an oven at 110 °C for 12 hours.

2.3. Coconut Shell Pyrolysis

A total of 150 grams of coconut shells were placed into the reactor and then pyrolyzed at 500°C for 2 hours with nitrogen gas added. The resulting biochar was then crushed using a grinder, sieved using a 200-mesh sieve, and characterized using FTIR, XRD, and XRF.

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2.4. Activation Using Potassium Hydroxide (KOH)

A total of 20 grams of coconut shell biochar was washed with HCl solution, then rinsed with distilled water until pH 7. Then, it was activated with a base activator of 2M Potassium Hydroxide (KOH) with a ratio of 1:4 (w/w) for 2 hours. Then it was neutralized using distilled water to pH 7 and dried in an oven at 110°C for 2 hours. Next, it was calcined at 500°C for 2 hours.

2.5. impregnation using metal ($Cu(NO_3)_2.3H_2O$)

A total 10 grams of biochar activation product was then mixed with 150 mL of 1%, 3%, 5% solution ($Cu(NO_3)_2.3H_2O$) in a beaker glass, after which it was refluxed while stirring with a magnetic stirrer at a temperature of 80°C for 5 hours. Then the results of the reflux process were dried in an oven at a temperature of 110°C for 24 hours. Then it was calcined at a temperature of 500°C for 2 hours with nitrogen gas flowing.

2.6. XRD characterization

The Blank biochar, activated biochar, and biochar impregnated with Cu metal were characterized using XRD to determine their structure.

2.7. FTIR characterization

The obtained blank biochar, activated biochar, and biochar impregnated with Cu were characterized using FTIR to determine their functional groups and chemical properties.

2.8 BET Characterization

The obtained blank biochar and Cu-impregnated biochar were characterized using BET to determine the surface area, pore volume and average pore diameter.

3. RESULTS AND DISCUSSION

Coconut shells are a potential biomass source for biochar catalyst production. Coconut shells are used as a base material for charcoal production due to their excellent thermal diffusion properties due to their high cellulose and lignin content.[3]

Biochar is produced through pyrolysis, which involves heating biomass (in this case, coconut shells) at high temperatures without oxygen. This process produces biochar with a large pore structure and a wide surface area. KOH was chosen as the activator because it can control the acidity of the coconut shell carbon powder, thereby opening the pores. After activation, KOH is expected to increase the micropore and mesoporous structures in the carbon electrode.

The wet impregnation process with $Cu(NO_3)_2\cdot 6H_2O$ solution produced materials with varying Cu content of 1%, 3%, and 5%. After impregnation, the materials were dried and calcined to produce chemically stable Cu-modified biochar.

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3.1. Analysis of Characterization Results

3.1.1 Analysis Using XRD

X-ray diffraction (XRD) is to distinguish between crystalline and amorphous materials. The principle of X-ray diffraction is the diffraction of X-ray waves that undergo scattering after colliding with crystal atoms. The resulting diffraction pattern displays a crystalline structure. Diffraction pattern analysis can determine the parameters of the lattice, crystal size, and crystal phase identification. The type of material can be determined by comparing the results of X-ray diffraction with a catalog of diffraction results of various materials.[6]

The degree of crystallinity is obtained from the measurement of X-ray Diffraction, which is used to determine the crystallinity properties (crystalline or amorphous) of a material, the lattice parameters and the distance between atoms in the crystal plane. The degree of crystallinity is the level of regularity of the structure of a material.

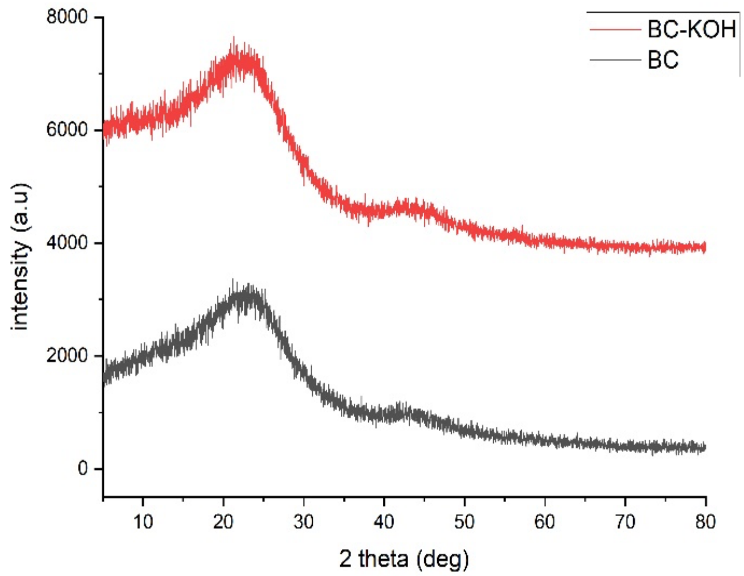

Figure 1. XRD patterns of various biochars and KOH activation of biochar

Based on the results of characterization with 2-theta (deg) with a peak width of 23°, which is a characteristic of amorphous or semi-crystalline materials. It shows a partially organized structure of amorphous or carbon graphite. This indicates the existence of small domains with a low level of aromatic regularity. The absence of sharp peaks indicates that the biochar does not contain significant amounts of crystalline minerals, or that the minerals have been decomposed during the pyrolysis process. Likewise, even after being activated, the structure of bichar still shows an amorphous structure.

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Table 1. XRD data of biochar and KOH activation on biochar

Sampel	Peak 2θ (°)	Crystalline Phase	Crystallite Size (nm)	Information
Biochar	23.19, 43.04	Amorf	0.605	Weak peaks, indicating irregular carbon structure
KOH Activated Biochar	23.2, 43.4	Graphitic carbon	0.604	Activation increases porosity and slightly sharps peaks.

The diffractogram differences between Biochar and Biochar-KOH look almost the same. In the biochar diffractogram, Biochar-KOH produces diffraction peaks that are not sharp. Figure 4.4 shows that XRD analysis shows significant differences between biochar and KOH-activated biochar. Biochar and biochar-KOH show diffraction patterns that are not sharp, indicating an amorphous structure with characteristic peaks at 2θ around 23° and 43° which reflect the turbostratic (amorphous) carbon structure. The calcination process further increases the intensity and sharpness of the peaks, indicating increased crystallinity and stabilization of the carbon structure through aromatization and removal of functional groups.

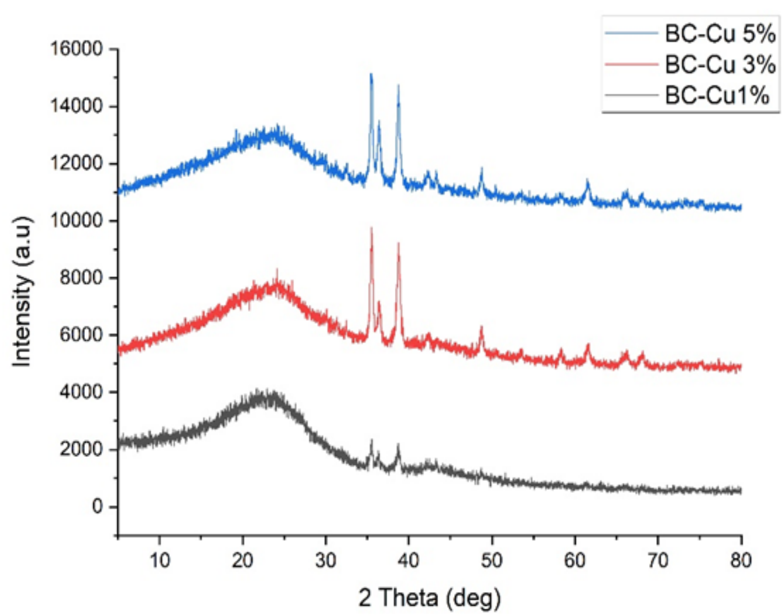

Figure 2. XRD patterns of various biochars impregnated with Cu metal at variations of 1%, 3%, and 5%

Table 2. XRD data of biochars impregnated with Cu metal at variations of 1%, 3%, and 5%

Sampel	Peak 2θ (°)	Crystalline Phase	Crystallite Size (nm)	Information
BC-Cu 1%	35.5, 38.7	CuO, Cu_2O	28.767	Sharp peaks indicate the formation of Cu nanoparticles.
BC-Cu 3%	35.4, 38.6	CuO, Cu_2O	24.608	Increased intensity and crystallinity of metal
BC-Cu 5%	35.3, 38.5	CuO, Cu_2O	29.472	Very sharp peaks, forming larger crystals

XRD diffractograms of Cu-modified biochar (1%, 3%, 5%) showed a broad amorphous peak at 2θ ≈ 22°, typical of amorphous carbon. With increasing Cu content, crystalline peaks appeared at 2θ ≈ 43.3°, 50.4°, and 74.1°, corresponding to the (111), (200), and (220) planes of Cu metal (JCPDS 04-0836), indicating the presence of CuO/Cu_2O. At 1% Cu, the Cu peak intensity was low, while at 3% and 5% Cu the intensity increased significantly, indicating higher crystallinity and dispersion of Cu. The average crystallite size of 28.767–29.472 nm indicated the formation of nanoparticles. The decrease in the carbon peak intensity indicated structural disruption due to metal deposition. Overall, Cu impregnation increased crystallinity and produced active metal phases that have the potential to improve the catalytic performance of biochar.

3.1.2 Analysis Using FTIR

Fourier Transformed Infrared (FTIR) is one of the instrument units that can be used to detect functional groups, identify compounds and analyze mixtures from the analyzed sample without damaging the sample. In the infrared region on the electromagnetic wave spectrum it starts from wavelengths of 14000 cm^{-1} to 10^{-1}. FTIR can also be used to detect functional clusters.[1] Revealed that the FTIR detector showed a band that widened in the region of 1200–1500 cm^{-1} which identified the presence of C-H and C-O groups. The appearance of the absorption peak at the wave number of 2510.46 cm^{-1} which forms the O-H functional group.

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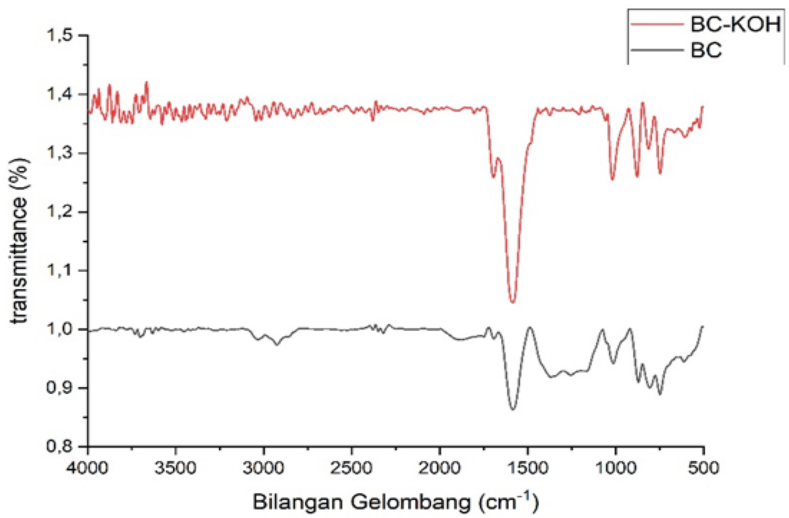

Figure 3. FTIR spectrum patterns of various biochars and KOH activation of biochar.

In this study, the FTIR spectrum was interpreted at wavenumbers of 4000–500 cm⁻1. In FTIR, the y-axis indicates absorbance or percentage transmittance, which measures the number of frequencies passing through the compound (not absorbed), while the x-axis indicates the wavenumber (cm⁻1). This study used the FTIR spectrum in the wavenumber range of 4000–500 cm^{-1} to identify functional groups in biochar before and after activation.

Unactivated coconut shell biochar exhibited absorption bands of O–H at 3435 cm^{-1}, aliphatic C–H at 2925 cm^{-1}, aromatic C=O or C=C at 1630 cm^{-1}, and polar C–O groups at 1100 cm^{-1}, which have the potential to interact chemically with metals. Biochar activation with KOH causes a shift and decrease in the intensity of the –OH (~3400 cm^{-1}) and aromatic C=C (~1600 cm^{-1}) bands, indicating a reduction in hydroxyl groups and modification of the aromatic carbon structure. The C–O band (~1030–1100 cm^{-1}) shifts and becomes sharper, indicating an increase in functional oxygen groups such as phenols or lactones. These changes indicate that KOH activation successfully modifies the biochar surface, reducing hydrophilic groups and increasing active groups (–CH_2, C=O) which are important for improving catalytic reactivity in the steam reforming process.

The FTIR spectrum of Cu-modified biochar (1%, 3%, 5%) showed a shift and change in the intensity of the absorption bands –OH (~3430–3400 cm^{-1}), aromatic C=C (~1600 cm^{-1}), and C–O (~1030–1100 cm^{-1}), indicating the interaction of surface functional groups with Cu^{2+} ions. At higher Cu concentrations, an enhancement of the band at 530–580 cm^{-1} appeared, indicating the formation of Cu–O bonds and the presence of CuO/Cu_2O. As the Cu concentration increased, the band shift became more pronounced and its intensity decreased, indicating more active groups –OH, C=O, and C–O were involved in metal binding. These changes confirmed the success of Cu impregnation and have the potential to improve the adsorptive and catalytic properties of biochar.

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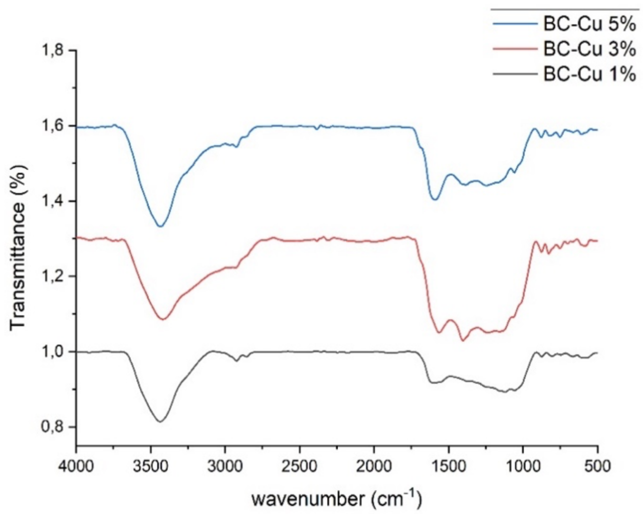

Figure 4. FTIR spectrum patterns of various biochars impregnated with Cu metal at variations of 1%, 3%, and 5%

3.1.3 Analysis Using BET

Characterization of surface area and pore structure using the Brunauer-Emmett-Teller (BET) method is of interest in evaluating the potential of biochar as an adsorbent material or catalyst support. In this study, copper (Cu) metal impregnation was carried out with varying concentrations of 1%, 3%, and 5% on biochar, then the surface texture parameters were analyzed using nitrogen gas at a temperature of 77.3 K.

Table 3. Comparison of surface area data, pore volume and average pore diameter of Biochar and Cu Metal impregnated biochar

Catalyst	Surface Area (m2/g)	Pore Volume (cm3/g)	Pore Diameter (nm)
Biochar	4,03	0,0174	17,33
BC-Cu 1%	257,3	0,1813	2,82
BC-Cu 3%	298,2	0,1912	2,56
BC-Cu 5%	393,9	0,2275	2,31

BET analysis showed that unmodified biochar had a very low surface area (4.03 m^2/g) and was dominated by macropores, thus limiting catalytic active sites. After Cu modification, the surface area and pore volume increased significantly, while the pore size decreased to the mesoporous range. BC-Cu 1% had 257.3 m^2/g, BC-Cu 3% reached 298.2 m^2/g, and BC-Cu 5% was the highest with 393.9 m^2/g and pore size of 2.31 nm. This increase in surface area and optimal pore distribution increased the number of active sites and maximized

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catalyst-reactant contact, thus accelerating the reaction and increasing product yield. Overall, Cu modification significantly increased the surface area and porosity of biochar compared to pure biochar. Biochar catalyst with 5% Cu metal modification excelled in producing the highest surface area to support heterogeneous catalyst performance.

4. CONCLUSION

Coconut shell biochar was successfully prepared through pyrolysis and KOH activation, then modified with copper (Cu) metal using a wet impregnation method. FTIR analysis showed strong interactions between Cu^{2+} ions with the –OH, C=O, and C–O functional groups on the biochar surface. XRD results revealed increased crystallinity and the formation of dispersed Cu, CuO, and Cu_2O phases on the surface, with an average crystallite size of 28–29 nm. BET analysis showed a significant increase in surface area from 4.03 m^2/g (initial biochar) to 393.9 m^2/g at 5% BC-Cu, with a decrease in pore size to the optimal mesoporous range for catalytic reactions. Overall, Cu modification improved the physicochemical characteristics of biochar, thus having high potential for use as a heterogeneous catalyst in biomass conversion and steam reforming processes.

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