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CHARACTERIZATION OF YOUNG COCONUT SHELL CHARCOAL ACTIVATED BY NAOH FOR POTENTIAL USE AS A NATURAL ADSORBENT

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ABSTRACT

Young coconut shell is ideal for adsorption applications due to the high content of hemicellulose, lignin, and cellulose, coupled with the porous structure and significant activated carbon content. Therefore, this study aimed to examine the characterization and potential of coconut shell charcoal as a natural adsorbent. To produce charcoal, young coconut shells were pyrolyzed at 400 °C. About 0.1 N of Sodium Hydroxide (NaOH) solution was used for the chemical activation process to increase pore volume and surface area. XRD (X-ray diffraction), FTIR (Fourier Transform Infrared Spectroscopy), and SEM (Scanning Electron Microscopy) were selected to characterize coconut shell-activated carbon. The results showed that based on XRD analysis, charcoal had an amorphous structure with minimal crystallinity. SEM results showed significant pore development after activation. FTIR analysis indicated the presence of -OH, C=O, and C-O functional groups. In conclusion, coconut shell charcoal has the potential to be an effective and inexpensive adsorbent to reduce environmental pollution.

Keywords: Adsorption, Characterization, Mercury, XRD, SEM, FTIR, Young Coconut Shell Charcoal.

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INTRODUCTION

The increasing environmental pollution, specifically from industrial activities, is raising public concerns. Therefore, sustainable solutions are needed to treat polluted air and water.[1]Environmental pollution treatment has been widely studied by applying certain methods, such as cation exchange from coconut fiber [2], electrocoagulation [3], heterogeneous photocatalysis with TiO_2 [4], absorption with *Aza222* polymer 5 and others. Several challenges arise in the application of this technology, including low removal efficiency, excessive use of chemicals, and lack of selectivity. [6] Adsorption and membranes are separation methods that are often applied. These two methods often show a high level of selectivity and achieve a high removal rate. [6] Adsorption is one of the most effective methods for removing contaminants, including heavy metals, dyes, and organic pollutants. [7] However, the most important factor in determining the efficiency and sustainability of the process is the selection of adsorbent materials. [6] The most frequently used adsorbent material is activated carbon, which has been proven to be effective in the adsorption process. [8] The high surface area and large pore structure allow for effective adsorption. [9] However, the cost required for the production of activated carbon with high carbon and low inorganic materials is relatively high. [10] The replacement of adsorbent raw materials with biomass has become the focus of many studies, specifically in terms of cost, modifiability, and environmental friendliness. [11] Biomass-based activated carbon is more in demand because of its abundant availability and easy accessibility. [10] According to previous reports, biomass from agricultural waste has not been optimally processed, and some is even thrown away. [12] Several high-quality adsorbents from biomass waste have been developed, including cocoa husk [13], rice husk [14], corn cob [15], walnut shell [9], coconut pith [16], and coconut shell. [10] Among various agricultural wastes, coconut shell shows significant potential as a source of high-quality activated carbon. [10] Most studies focus on old coconut shell waste [17], while young coconut shell waste is a resource that has not been optimally

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utilized, despite being abundant in tropical countries such as Indonesia. [18] Young coconuts are usually consumed as drinks, and the unused parts produce waste in the form of fiber and shell, which are generally disposed of in landfills. [12] Shell contains 46.10% cellulose, 28.25% lignin, and 25.65% hemicellulose, a high biomass content that can be a heavy metal adsorbent. [19] Proper characterization of these properties is very important to determine the feasibility as an adsorbent in environmental applications. [12] Making adsorbents from shell waste can reduce environmental pollution and support the zero-waste program. [19] The Activation process is very important to improve the adsorption capacity of raw biomass by increasing the surface area, pore volume, and chemical reactivity. [14] NaOH (sodium hydroxide) is commonly selected as an activation agent because of the chemical properties of carbon-based adsorbents, which are effective in increasing porosity. [10] It not only removes dirt but also has functional groups with oxygen content, which increases adsorption performance. [20] The selection of NaOH activation combination with young coconut shell is considered capable of developing environmentally friendly adsorbents to prevent pollution at a low cost. This study contributes to the utilization of waste by using shell waste to overcome economic and environmental problems. This study examined the properties of activated charcoal from coconut shell, including physical, chemical, and morphological to determine its potential as a natural adsorbent. XRD (X-ray diffraction) analysis was carried out to analyze the effect of activators and detect the crystal structure of activated charcoal. The functional group analysis of the sample applied FTIR (Fourier Transform Infrared Spectroscopy), while the elemental composition and surface morphology were determined using Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX). The analysis results provide additional insight into how to convert agricultural waste into a natural but high-value adsorbent, improving sustainable practices by selecting renewable natural resources. In addition, this study is appropriate to the growing trend towards green technology in environmental management and directly supports Sustainable Development Goal (SDG) 6: Clean Water and Sanitation, by offering innovative, low-cost, and eco-friendly solutions for water treatment and pollution reduction.

EXPERIMENTAL

Raw Material Preparation

The materials used include young coconut shell waste, Sodium Hydroxide (Sigma-Aldrich), Mercury (II) Chloride (Sigma-Aldrich), hydrochloric acid (Sigma-Aldrich), and distilled water.

Making Young Coconut Shell Charcoal in Powder Form

Young coconut shell waste was collected, washed, and cleaned of dirt, then dried in the sun for 5 days. The drying process was continued using an oven at 105ºC for 3 hours. The sample was crushed to a size of 2-3 mm for the pyrolysis stage at a temperature of 400ºC for 3 hours, which produced liquid smoke, tar, and charcoal, as shown in Fig.-1. A detailed procedure for charcoal production can be found elsewhere.[18] Subsequently, charcoal was crushed with a grinder and sieved to a size of 100 mesh.

Synthesis of Activated Carbon

The sample was activated with 0.1 N NaOH for 24 hours at a sample and activator ratio of 1:4, then washed with 0.5 M hydrochloric acid (HCl) and distilled water until the pH reached 7. Following activation, the material was dried at 105ºC for 1 hour.

Characterization

XRD analysis was performed using a Shimadzu XRD 7000 diffractometer with an accelerating voltage of 40 kV and a current of 30 mA. The scanning diffractometer recorded in the 2θ range from 10 to 55. SEM-EDX characterization was performed using an FEI Inspect S50 to identify the morphology and elemental percentage of activated carbon. Meanwhile, FTIR characterization was performed using an IR Tacer-100 to identify the transmission spectrum of the sample and functional groups with a wavelength measurement range of 700 to 4000 cm^{-1} measured the sample surface area, pore size, pore distribution, and surface volume.

Charcoal production using the pyrolysis reactor in Fig.-1 was carried out by heating young coconut shells at a temperature of around 400°C in conditions without oxygen. The raw materials were placed into the main reactor, and during heating, the biomass decomposed into three main products, namely liquid smoke, tar, and charcoal. The gas and steam produced were then channelled through pipes to the spiral condenser,

where the gas was cooled to produce condensate in the form of liquid smoke and tar, collected in a container. The remaining solids, undecomposed in the reactor, became charcoal, which was collected at the bottom and was ready to be used.

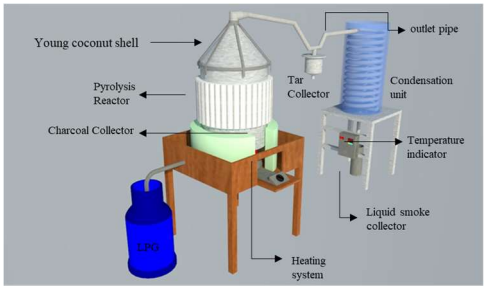


Fig-1: Charcoal Production Process

RESULTS AND DISCUSSION

XRD Analysis

XRD characterization was conducted to obtain the diffraction pattern for young coconut shell charcoal. The sample was analyzed using Origin Pro Software to obtain the sample phase, and the results are shown in Fig.-2. There are two diffraction patterns found, namely, young coconut shell charcoal samples before (YCS) and after (YCSAC) activation. Compared to YCSAC, the diffraction pattern of YCS showed sharper and clearer crystallinity peaks. This was indicated by the several high-intensity peaks, namely ~28°, ~38°, and ~45° around the angle of 2θ; hence, the degree of crystallinity was higher. The diffraction pattern of YCSAC showed a uniform intensity distribution and wider peaks, specifically in the range of 2θ (15° - 30°), indicating the dominant nature of amorphous characteristics. The decreased peak intensity in YCSAC samples is characteristic of activated carbon materials due to changes in the material structure after modification or activation. [21] Due to chemical or thermal decomposition process that disrupts the crystal regularity, this activation produces a more amorphous structure, thereby increasing the surface area and porosity. This enhanced porosity is particularly valuable for water filtration applications, aligning with Sustainable Development Goal 6: Clean Water and Sanitation, which emphasizes the development of affordable and efficient technologies for improving water quality. Sujiono *et al.* (2022)[10] showed that chemical activation with NaOH on carbon materials caused significant changes in the crystal structure because the material is more amorphous, leading to increased adsorption properties. YCSAC also experienced activation in the form of a decrease in the crystallinity peak and an increase in amorphous components. These results are from previous studies where the crystal structure becomes more amorphous when chemical or physical activation occurs, with a loss of sharp diffraction peaks on XRD. [17] Emahi *et al.* (2019) [8] also found a similar pattern to YCSAC, where thermal activation of activated carbon from biomass waste produced an amorphous structure with a low peak at 2θ = 20–30°. Anshary *et al.* (2022) [19] found that after activation, the XRD diffraction pattern of activated carbon from young coconut shell waste showed a high level of amorphosity.

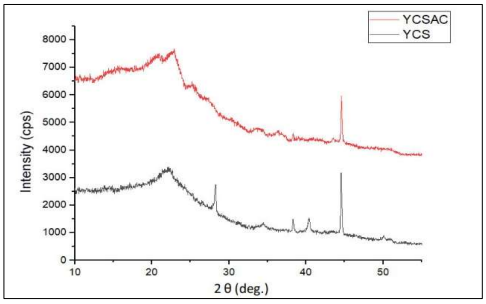
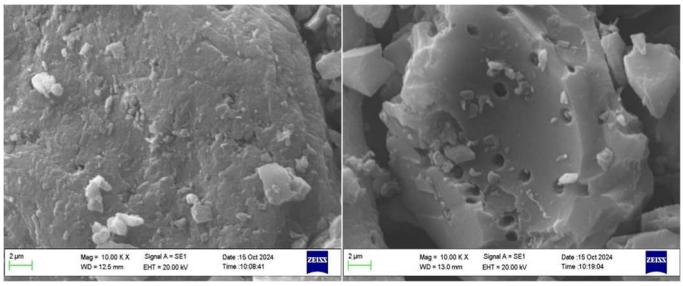


Fig.-2: XRD Diffraction Patterns before (YCS) and after (YCSAC) Activation

Compared to the YCS sample, the decrease in crystallinity and increase in amorphous character occurred due to activation or modification of the YCSAC sample. These changes are generally expected in the process of making adsorbent materials such as activated carbon, which focuses more on increasing porosity than crystallinity.

SEM-EDX Analysis

SEM was used to characterize the surface morphology of young coconut shell adsorbent before and after chemical activation using a magnification of 10.00 KX (10,000 times) as shown in Fig.-3 (a,b). Figure-3a shows a relatively denser structure with large and irregular particle agglomeration. This indicates that the material has a less dispersed structure, possibly indicating charcoal before activation, where porosity has not developed significantly. Particle agglomeration may be formed due to natural binding or the absence of an activation process that forms additional pores. [22]



(a) (b)

Fig.-3: Surface Morphology of YCS (a) and YCSAC (b) Samples

Figure-3b shows a surface with a more open texture as well as broken and coarse particles. A clearer porous structure is also visible, indicating characteristics of the material after the activation process, where there is an increase in porosity. Activation process usually produces a rougher surface as well as micro and macro pores, which can increase the total surface area. [23] This porosity is needed to improve the adsorption properties of activated carbon. The purpose of the charcoal activation process is to expand and open the pore structure of the material, as in Fig.-3b. Liu *et al.* (2020) [14] found that the increase in the material's ability to absorb substances such as metals occurred because activated carbon had a more porous and rougher surface. EDX characterization results in Table-1 show a significant change in elemental composition between YCS and YCSAC samples. To detect the elemental composition on the surface, EDX and SEM analyses were conducted. [24] The main elements detected in YCS were C (carbon) and O (oxygen), reflecting the lignocellulose content of the natural material. Meanwhile, increased carbon content in YCSAC was estimated to occur along with the loss of volatile elements during the activation process. The percentage of YCS carbon was around 68.09%-70.63%, while the percentage of YCSAC decreased to 63.58% after activation. The activation process causes some carbon to be lost (oxidized) as gas, resulting in a decrease. Both YCS and YCSAC samples experienced an increase in the oxygen of 29.25% and 34.39%, respectively. This shows that the activation process increases oxygen absorption in the pore structure of charcoal. The mineral content in both charcoal samples was relatively low, indicated by the very small amount of chlorine, magnesium, sodium, and potassium. The process of structural changes that occur due to the activation process is characterized by an increase in oxygen and a decrease in carbon. [25] The results were in line with the study conducted by Liu *et al.* (2020), [14] where a more porous structure was obtained using the SEM method, due to chemical or thermal activation of materials with carbon materials. Faisal *et al.* (2018) [20] also reported that the removal of organic components in activated carbon materials during activation was due to increased porosity, which expanded the effective surface area. According to Emahi *et al.* (2019), significant changes in surface morphology occurred due to the activation process, which developed micro- to mesopores as observed in YCSAC. This study provides additional information needed to determine characteristics and potential applications of charcoal, specifically derived from young coconut shell waste, in the field of air and water treatment.

Table-1: Elemental Composition of Activated Carbon from YCS and YCSAC

Elements	YCS	YCSAC
Carbon	68.09	63.58
Oxygen	29.25	34.39
Magnesium	0.20	0.12
Chlorine	0.52	-
Potassium	1.94	1.00

Sodium	-	0.74
Calcium	-	0.12
Silicon	-	0.06

FTIR (Fourier Transform Infrared Spectroscopy) Analysis

Figure-4 shows the FTIR spectrum illustrating characterization results of two coconut shell charcoal samples (YCS and YCSAC) before and after activation. In the spectrum, the description of the functional groups contained in the sample is provided in characteristic peaks. At around 3500–3200 cm^{-1}, an absorption peak was found, indicating the presence of hydroxyl groups (OH) of water or –OH groups from alcohol or phenol compounds.[26] This peak appears clearer in YCS than in YCSAC, suggesting that the carbon activation process has reduced the content of hydroxyl groups. At around 2900–2800 cm^{-1}, there was a small peak usually associated with aliphatic C-H stretching, which often appears in carbon-based materials. This peak decreased slightly in YCSAC, indicating changes in the aliphatic structure after carbon activation. [10,18] There was a peak corresponding to the carbonyl group (C=O) at around 1700 cm^{-1}, which originated from several compounds, including aldehyde, carboxylic acid, or ketone. [27] The peak intensity in YCSAC appeared smaller during the carbon activation process due to the presence of several decomposed carbonyl groups. A small peak at around 1600–1500 cm^{1} indicated the presence of an aromatic ring (C=C). This ring is commonly found in coconut shell material due to the content of lignin content as the basic material..[28] The peak appeared clearer in YCSAC, where an increase in the content of aromatic structures occurred in the carbon material due to the activation process.[10,19] A peak representing the C-O bond from the alcohol, ether, or ester group was found at around 1200–1000 cm^{-1}. [10] This peak also decreased in YCSAC due to the reduction of the oxygen group after the carbon activation process. [29,30]

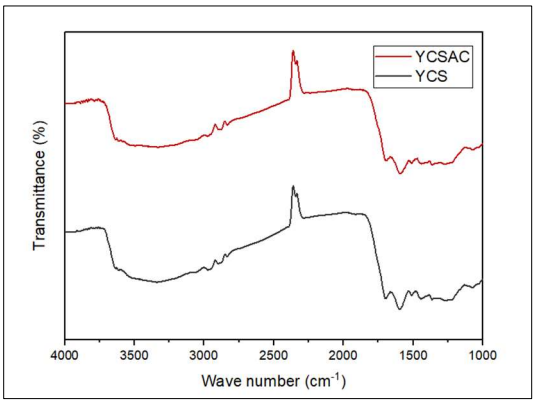


Fig.-4: FTIR Spectrum of Samples before (YCS) and after (YCSAC) Activation

Based on FTIR results, there was an increase in the aromatic structure (C=C) and changes in the arrangement of functional groups, including reduction of carbonyl, hydroxyl, and C-O groups due to carbon activation in YCSAC. This shows that carbon activation not only modifies the chemical composition of the surface but also changes the morphology of the material, thereby affecting the adsorptive properties.

CONCLUSION

In conclusion, this study successfully prepared effective activated carbon by applying NaOH activation agent from young coconut shell waste. Compared with the sample before activation, activated carbon has a more amorphous structure with changes in the crystal structure indicated by a decrease in peak intensity after applying the XRD method. Based on the SEM-EDX analysis results, the percentage of carbon in YCS (68.09%) decreased after activation (63.58%), while oxygen increased from 29.25% to 34.39%. The presence of functional groups such as hydroxyl, ether, and carbonyl during characterization analysis with FTIR showed the success of the activation process, indicated by an increase in the potential for material adsorption. In general, the results showed that activated carbon from young coconut shells has the potential to be an effective technology for absorbing heavy metals, such as mercury, in the wastewater treatment process. This contributes directly to the achievement of the United Nations SDG 6: Clean Water and Sanitation, which emphasizes the development of accessible, environmentally friendly technologies to

improve water quality, reduce pollution, and promote the safe reuse of wastewater, particularly in regions with limited access to advanced treatment infrastructure.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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