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Characterization of functional groups and crystal structure of activated carbon from ketapang fruit biomass using FTIR and XRD

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ABSTRACT

This study investigates the functional group characteristics and crystal structure of activated carbon derived from ketapang fruit waste (*Terminalia catappa*) chemical activation with $ZnCl_2$ at a pyrolysis temperature of 600°C in a nitrogen atmosphere. Characterization was performed using Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). FTIR analysis confirmed the presence of O–H, C=C, C–H, and C–O functional groups, indicating the retention of oxygen-containing surface groups alongside the formation of aromatic carbon structures characteristic of activated carbon. XRD patterns exhibited broad diffraction peaks at $2\theta \approx 24° – 26°$ and $2\theta \approx 43° – 45°$, corresponding to the (002) and (100) crystallographic planes of carbon, confirming a predominantly amorphous structure with limited graphitic ordering. These findings demonstrate that ketapang fruit biomass is a promising precursor for producing activated carbon with well-developed surface chemistry and structural properties suitable for further development as a functional material.

Keywords: Activated carbon; FTIR; ketapang fruit; XRD; $ZnCl_2$

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INTRODUCTION

Activated carbon is a carbon-based material widely used in various applications. This is due to its porous structure and large surface area, which makes it suitable as a catalyst, adsorbent, energy storage, and electromagnetic wave absorber. Due to its extraordinary properties, such as complex surface chemistry, high specific surface area, good thermal and chemical stability, and strong mechanical resistance, activated carbon remains a highly attractive material for various applications [1]. According to [2], the use of activated carbon in industry has expanded to various important industries, such as food, pharmaceuticals, and refining processes in the petroleum and natural gas processing industry.

The use of biomass as a raw material for activated carbon is a major concern due to its abundant availability and environmentally friendly nature [3]. Various types of lignocellulosic biomass have been successfully converted into high-quality activated carbon, such as sugar palm fronds [4], empty sugar palm fruit bunches [5], and nipah palm fiber [6], demonstrating that local Indonesian biomass waste has great potential as an activated carbon precursor. Utilizing agricultural waste or fruit waste into activated carbon is a strategic step to add value to residues [7]. Ketapang fruit (*Terminalia catappa*) is a biomass with great potential as an activated carbon precursor due to its high lignocellulose content, consisting of cellulose, hemicellulose, and lignin, which is a good carbon source for the carbonization process. Furthermore, ketapang fruit has been considered an underutilized waste product, making its processing into activated carbon a

strategic effort to increase the added value of biomass waste while reducing environmental impacts. This rich lignocellulose content makes ketapang fruit potentially capable of producing activated carbon with a well-developed pore structure and high surface area [8].

Furthermore, the isolation of microcrystalline cellulose from ketapang leaves indicates that the plant contains an excellent natural polymer that can be converted into useful carbon through delignification and hydrolysis [9]. Chemical activation using agents such as $ZnCl_2$, KOH, or H_3PO_4 in the production process is crucial to trigger the formation of a large pore structure [10, 11]. Optimization of the type and concentration of chemical activators has been shown to significantly affect the electrochemical properties and pore characteristics of the resulting activated carbon [12].

The surface properties of carbon can be altered by this chemical activation, making it a supercapacitor electrode capable of storing large amounts of energy [13, 14]. Due to its renewable and sustainable nature, biomass-derived carbon is also beginning to be used in Li-ion batteries [15]. Structural and chemical characterization of activated carbon using FTIR and XRD is a standard method that has been widely applied to various biomasses, such as activated carbon from areca nut shells [16] and coffee grounds [17], both of which exhibit functional group patterns and amorphous structures consistent with lignocellulose-based activated carbon. Morphological and XRD methods for describing crystal structures have become standard, especially when used in conjunction with High Energy Milling (HEM) to produce nanometer-sized particles having specific crystal structures that can improve the functional performance of materials [18].

RESEARCH METHODS

Ketapang fruit biomass was used as the primary raw material in this study to produce activated carbon. The ketapang fruit was first cleaned of dirt and dried in the sun until a constant mass was achieved. Next, the sample was crushed using a hammer mill and crusher/grinder, and sieved through a 100-mesh sieve to obtain a powder with a uniform particle size.

A total of 30 grams of ketapang powder was chemically activated using 30 grams of $ZnCl_2$ (1:1 impregnation ratio) dissolved in distilled water, then stirred until homogeneous for 2 hours. The chemical activation process with $ZnCl_2$ aims to enhance the development of pore structures in the resulting carbon material through dehydration and pore formation during the carbonization process.

Figure 1. Activated carbon production process.

After the chemical activation process was completed, the sample was dried in an oven at 105°C for 24 hours before carbonization. The carbonization process was carried out using a

furnace tube at 600°C with a heating rate of 10°C/minute and a constant flow of nitrogen gas for 2 hours. This aimed to increase the carbon's purity and prevent oxidation during the process. The resulting carbon was then washed alternately with 0.1 M HCl solution and distilled water, followed by 0.1 M NaOH solution until the pH was neutral (pH = 7). Then, it was dried in an oven at 105°C for 12 hours. The process of preparing activated carbon from ketapang fruit biomass is shown in Figure 1.

RESULTS AND DISCUSSION

FTIR Analysis

Activated carbon functional group characterization was performed using FTIR in the wavenumber range of 400 – 4000 cm^{-1}. The FTIR spectrum of activated carbon carbonized at 600°C is shown in Figure 2.

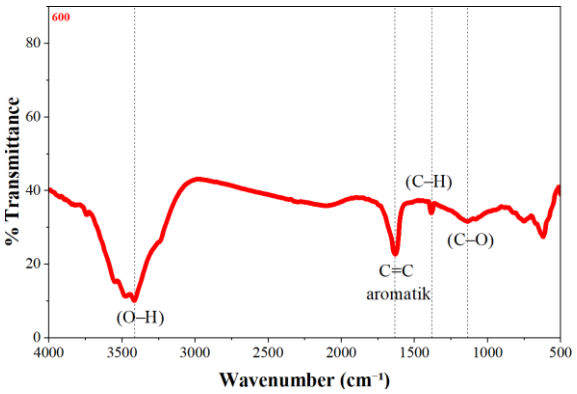

Figure 2. FTIR spectrum.

The FTIR spectrum shows a dominant peak at 3416 cm^{-1}, indicating stretching vibrations of the hydroxyl (O–H) group. The presence of this group is crucial because Terminalia catappa is known to contain cellulose and natural polymers rich in oxygen functionality [9]. The carbonization process at 600°C does not completely remove this group, meaning the activated carbon retains its hydrophilic properties, which are beneficial for applications in liquid media. This persistent O–H group pattern is also consistent with findings in other biomass-based activated carbons such as areca nut shells [16] and coffee grounds [17], indicating that hydroxyl groups are a common characteristic of lignocellulosic activated carbons.

The absorption band at 1633.5 cm^{-1} represents the aromatic C=C stretching vibration, which is a key indicator of successful carbon network formation [11]. Further analysis at wavenumbers 1380.8 cm^{-1} (C–H) and 1136.2 cm^{-1} (C–O) indicates the presence of residual bonds from lignocellulosic components. This is consistent with findings that variations in the chemical characteristics of the seeds and shells of ketapang fruit significantly influence the retention of functional groups during thermal degradation [8].

Technically, the abundance of oxygen functional groups acts as active sites that increase the material's surface reactivity. Surface modifications produced by chemical activation have been shown to optimize adsorption interactions through hydrogen bonding and π–π interactions [10, 19]. Ketapang fruit activated carbon has great potential for use as a conventional adsorbent and as a useful component in water purification systems contaminated with metal ions due to its complex surface chemistry profile [7].

XRD Analysis

Activated carbon crystal structure characterization was performed using X-ray Diffraction (XRD). The XRD diffraction pattern of activated carbon carbonized at 600°C is shown in Figure 3.

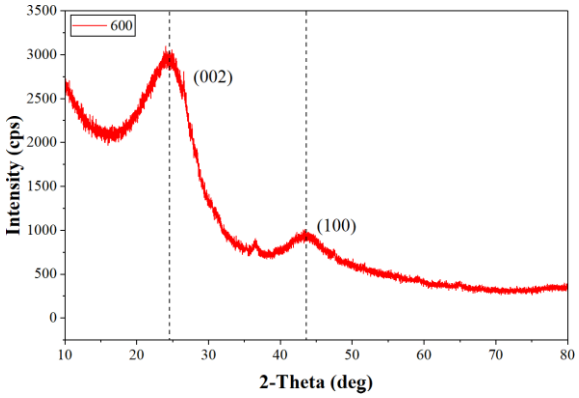

Figure 3. XRD spectrum.

The results of the crystal structure characterization using XRD are shown in Figure 3. The resulting diffraction pattern shows two broad peaks at angles $2\theta \approx 24° – 26°$ and $2\theta \approx 43° – 45°$, which correspond to the (002) and (100) crystal planes, respectively. The appearance of these significantly broad peaks confirms that the material is dominated by an amorphous carbon structure with short-range order [14, 15]. This diffraction pattern, with two broad peaks on the (002) and (100) planes, is a characteristic of lignocellulosic biomass-based activated carbon, which has been consistently reported for activated carbon from sugar palm fronds [4] and empty sugar palm fruit bunches [5], both of which were carbonized at similar temperature ranges.

Although the amorphous phase dominates, the presence of the peak on the (002) plane indicates the formation of partial graphitic domains. This amorphous structure, with numerous crystal defects, offers mechanical and electrochemical advantages. In energy storage applications, this structure facilitates ion diffusion into the carbon pores [13]. Furthermore, shifts in the diffraction peaks can occur due to the use of intensive activation or milling techniques. This is a result of the reduction in crystallite size to the nanometer scale [18].

This structural analysis indicates that the microarchitecture of the ketapang fruit activated carbon is stable. This material is highly competitive due to its large surface area and graphitic domains, which support thermal stability [1]. These results support the position of Terminalia catappa-based activated carbon as an ideal choice for electrode materials in supercapacitors, Li-ion batteries, or electromagnetic wave absorbers that require a specific crystal structure [18, 20].

CONCLUSION

Activated carbon obtained from ketapang fruit biomass through a carbonization process at 600°C and chemical activation with $ZnCl_2$ exhibits unique structural and chemical characteristics. FTIR analysis results indicate the presence of O–H, C=C, C–H, and C–O functional groups, indicating the presence of oxygen groups and the formation of aromatic carbon structures on the material's surface.

XRD analysis results indicate that the resulting activated carbon has a structure dominated by an amorphous phase, with diffraction peaks on the (002) and (100) planes indicating the formation of graphitic domains.

Ketapang fruit-based activated carbon has an amorphous structure and the presence of surface functional groups that support various material interaction mechanisms, thus having the potential to be developed as a functional material.

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