

Synthesis of activated carbon based on palm oil fruits with FTIR and XRD characterization

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ABSTRACT

Activated carbon derived from oil palm frond biomass was successfully synthesized via chemical activation using KOH, followed by ZnO doping. This study aims to evaluate the structural and functional characteristics of the synthesized material. The synthesis process involved impregnation, drying, and carbonization under a nitrogen atmosphere. X-ray diffraction (XRD) analysis revealed the formation of crystalline ZnO with a hexagonal (wurtzite) structure, indicated by diffraction peaks at $2\theta \approx 31.66^\circ$, 34.32° , and 36.14° , along with a broad peak corresponding to amorphous carbon. Fourier transform infrared (FTIR) analysis identified functional groups such as O–H, C=O/C=C, and C–O, as well as a Zn–O vibration band at approximately 484 cm^{-1} . These findings confirm the successful formation of a composite material consisting of amorphous activated carbon integrated with crystalline ZnO, which shows potential for adsorption and catalytic applications.

Keywords: Activated carbon; FTIR; oil palm fronds; XRD; ZnO

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INTRODUCTION

With the growing demand for environmentally friendly and sustainable materials, the use of biomass as a raw material for functional materials is growing. Indonesia, as one of the world's largest palm oil producers, produces large amounts of biomass waste, which has the potential to be an abundant source of carbon for conversion into high-value activated carbon [1].

Activated carbon is a porous material with a high surface area and is widely used in various fields, such as adsorption, energy storage, and environmental treatment. The characteristics of activated carbon are strongly influenced by the type of raw material and the activation method used. Chemical activation using potassium hydroxide (KOH) is known to be effective in increasing the porosity and surface area of carbon through dehydration and the formation

of a microporous structure, making it one of the most widely used methods in biomass-based activated carbon synthesis.

In addition to the activation process, modification of activated carbon through the addition of inorganic materials such as zinc oxide (ZnO) is also gaining attention in the development of advanced materials. ZnO is a semiconductor material with good optical and catalytic properties, potentially enhancing the functional properties of activated carbon, particularly in terms of material stability and surface interactions [2]. The addition of ZnO is expected to increase the number of active sites and improve the structural characteristics of activated carbon.

Although various studies have reported the synthesis of biomass-based activated carbon and its modification using inorganic materials, studies specifically examining the combination of chemical activation using KOH and ZnO

doping on oil palm frond biomass are still limited. Furthermore, studies examining the relationship between crystal structure, carbon amorphousness, and the presence of surface functional groups on material characteristics also require further development.

Material characterization is a crucial step in understanding the physical and chemical properties of the resulting activated carbon. X-ray diffraction (XRD) was used to identify the crystal structure, carbon amorphousness, and the presence of ZnO phases in the material. Meanwhile, Fourier transform infrared spectroscopy (FTIR) was used to identify functional groups on the activated carbon surface, such as hydroxyl ($-OH$) and carbonyl ($C=O$) groups, as well as other groups that play a role in the adsorption process [3].

Based on this description, this study aimed to synthesize activated carbon from oil palm frond biomass through chemical activation using KOH and ZnO doping, and to analyze the material's structural and functional group characteristics using XRD and FTIR. This research is expected to contribute to the development of sustainable biomass-based carbon materials with high added value.

LITERATURE REVIEW

Oil Palm Frond Biomass-Based Activated Carbon

Lignocellulose is known as a primary source for the development of carbon-based materials due to its abundant availability, relatively low cost, and environmentally friendly properties. One potential biomass is oil palm frond (OPF), which is produced in large quantities from the maintenance and harvesting of oil palm plantations [1].

Oil palm fronds are composed primarily of cellulose (42.05%), hemicellulose (32.07%), and lignin (20.08%), although the composition can vary among lignocellulosic biomass [4, 5]. These three components play a crucial role in the formation of the carbon structure during the carbonization process, where complex

interactions between lignocellulosic components influence the conversion process and pore formation [6, 7]. The relatively high carbon content makes OPF a suitable raw material for activated carbon production [1]. Furthermore, the lignocellulosic structure provides an initial framework that can develop into a pore system after undergoing an activation process.

Utilization of oil palm fronds is currently limited, generally used as compost or left to degrade naturally in the field. Therefore, converting this biomass into activated carbon offers added value, both economically and environmentally [8].

Several studies have shown that carbon produced from oil palm fronds (OPF) has characteristics suitable for various applications, such as adsorption and energy storage. The resulting carbon structure can range from amorphous to semi-graphitic, with porosity development influenced by the activation method used [2]. This indicates that OPF serves not only as a carbon source but also as a precursor capable of producing materials with good functional properties.

Thus, oil palm fronds can be categorized as a promising raw material for activated carbon synthesis. The combination of high carbon content, a lignocellulosic structure that supports pore formation, and abundant availability make it relevant to sustainable waste utilization concepts.

Chemical Activation with KOH in Activated Carbon Synthesis

Chemical activation is a method widely used in activated carbon synthesis because it can produce materials with a high surface area and more controlled pore distribution compared to processes without activation. Potassium hydroxide (KOH) is often used due to its effectiveness in forming micropores and improving the carbon's surface properties [2].

In oil palm frond biomass, the use of KOH has been shown to increase the porosity and surface area of activated carbon, especially

when performed at high activation temperatures [2]. This activation process involves chemical reactions and etching mechanisms in the carbon matrix, causing the less stable amorphous components to decompose, creating small cavities that develop into pores.

The role of KOH is not limited to pore formation but also contributes to the development of the carbon's internal structure. This process produces a large number of micropores that serve as primary sites for the adsorption of ions and molecules [9]. Furthermore, appropriate activation conditions can also facilitate the formation of mesopores, which act as diffusion pathways to the micropores [9].

The simultaneous presence of micropores and mesopores significantly influences the performance of activated carbon. Micropores contribute to the specific surface area and adsorption capacity, while mesopores support diffusion processes toward the interior of the material. Therefore, hierarchical pore structures generally provide better performance than carbon dominated by micropores [9, 10].

In addition to affecting the pore structure, activation using KOH can also alter the surface chemistry of the carbon. Several studies have reported that increasing the number of oxygen functional groups contributes to improved hydrophilic properties (wettability) and interactions with electrolytes or adsorbates [10]. Thus, the quality of activated carbon is determined by a combination of surface area, pore size distribution, and surface chemical characteristics [10].

In oil palm frond biomass, KOH is considered more effective than other activators in producing activated carbon with a high surface area and better pore distribution. This makes KOH a primary choice for the synthesis of OPF-based activated carbon [2].

ZnO Doping in Biomass-Based Activated Carbon

Activated carbon development focuses not only on increasing the surface area and pore

structure but also on modifying the surface properties through a doping process. One material frequently used as a dopant is zinc oxide (ZnO), which has semiconducting properties, good chemical stability, and high surface activity [11].

The addition of ZnO to an activated carbon matrix aims to increase the number of active sites and strengthen the interaction between the material and surrounding ions or molecules. The presence of ZnO can also improve the material's electrochemical properties, as it supports charge transfer and enriches reaction centers on the carbon surface [12].

Furthermore, ZnO doping can affect the pore structure of activated carbon. Several studies have shown that the presence of ZnO results in a more open structure, thereby increasing pore accessibility and facilitating the diffusion of molecules into the material [13]. This is important because the performance of activated carbon is determined not only by the number of pores but also by the efficiency of transport to the active sites.

In terms of characterization, the presence of ZnO in activated carbon material can be identified using X-ray diffraction (XRD), which shows characteristic peaks in the hexagonal (wurtzite) crystal structure. This structure is an indicator of the success of the doping process and indicates that the ZnO remains stable throughout the synthesis process [12].

Thus, ZnO doping is an effective approach to improving the functional properties of biomass-based activated carbon. The combination of the porous carbon structure and the active properties of ZnO produces a composite material with superior performance compared to unmodified carbon.

Characterization of Activated Carbon Structure and Functional Groups

Characterization is a crucial step in understanding the physical and chemical properties of the resulting activated carbon. Two commonly used techniques are X-ray diffraction (XRD) and Fourier transform

infrared spectroscopy (FTIR), which provide information regarding the crystal structure and surface functional groups.

XRD analysis is used to determine the crystal structure and degree of regularity of the carbon. Biomass-based activated carbon generally exhibits an amorphous structure characterized by a broad peak in the 2θ region around $20^\circ - 25^\circ$, which corresponds to the (002) plane of carbon [14]. This amorphous structure favors pore formation and increased surface area.

Furthermore, the presence of ZnO in carbon composites can be identified through characteristic diffraction peaks indicating a hexagonal (wurtzite) crystal structure. The appearance of these peaks indicates that ZnO was successfully dispersed within the carbon matrix and retained its crystalline structure [12].

Meanwhile, FTIR analysis was used to identify functional groups on the surface of activated carbon. Common functional groups include hydroxyl ($-\text{OH}$), carbonyl ($\text{C}=\text{O}$), and ether ($\text{C}-\text{O}$), which originate from lignocellulosic components and the activation process [15]. These groups play a role in enhancing the interaction between activated carbon and surrounding molecules or ions.

In materials modified with ZnO, FTIR can also demonstrate the presence of Zn–O bonds at low wavenumbers, which is an additional indicator of successful doping [12]. The combination of carbon functional groups and the ZnO phase contributes to improving the material's surface chemical properties.

Thus, the use of XRD and FTIR allows for a comprehensive analysis of the structure and surface properties of activated carbon, which can be linked to the material's performance in various applications.

RESEARCH METHODS

Materials and Equipment

Oil palm fronds were used as the biomass carbon source in this study. Potassium hydroxide (KOH) served as the activating

agent, while zinc oxide (ZnO) served as the dopant. Aquadest served as the solvent in the mixing and washing processes.

The equipment used included a hammer mill, grinder, 100-mesh sieve, oven, magnetic stirrer, and a tube furnace equipped with nitrogen (N_2) gas flow for the carbonization process.

Activated Carbon Synthesis and ZnO Doping Process

The dried oil palm fronds were first reduced in size using a hammer mill. The material was then further ground with a grinder to obtain a fine powder. To obtain a uniform particle size distribution, the powder was sieved through a 100-mesh sieve.

A total of 30 g of biomass powder was then mixed with a KOH solution dissolved in distilled water at a mass ratio of 1:1. In the modified sample, ZnO was added with a carbon-to-ZnO ratio of 1:0.5. The resulting mixture was then homogenized using a magnetic stirrer for approximately 2 hours until a uniform suspension was obtained.

After the homogenization stage, the mixture was dried before entering the carbonization process. Carbonization was carried out using a tube furnace at 800°C for 3.5 hours under a nitrogen (N_2) atmosphere. This stage included a 1.5-hour preheating (ramp-up) followed by a 2-hour holding time. During this process, KOH served as a chemical activation agent, contributing to the formation of the pore structure, while ZnO was dispersed into the carbon matrix. After the carbonization process was complete, the sample was cooled naturally to room temperature.

The carbonized product was then washed with distilled water until it reached a near-neutral pH to remove any remaining activator or byproducts. The final stage involved re-drying to obtain dry powdered activated carbon.

Material Characterization

Characterization was performed using X-ray diffraction (XRD) to analyze the crystal

structure, the degree of carbon amorphousness, and the presence of ZnO phases. Additionally, Fourier transform infrared spectroscopy (FTIR) was used to identify functional groups on the surface of activated carbon.

The overall research methodology for synthesizing activated carbon from oil palm fronds is shown in Figure 1.

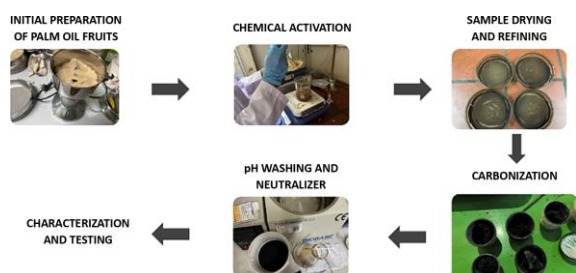


Figure 1. Research methodology for synthesizing activated carbon.

RESULTS AND DISCUSSION

Crystal Structure Characterization (XRD)

X-ray diffraction (XRD) characterization of a ZnO-doped activated carbon sample revealed a diffraction pattern reflecting the presence of two main phases: amorphous activated carbon and crystalline ZnO. The sample was coded PLS-0.5, where PLS stands for Palm Leaf Sample, and 0.5 indicates the doping ratio of ZnO to carbon.

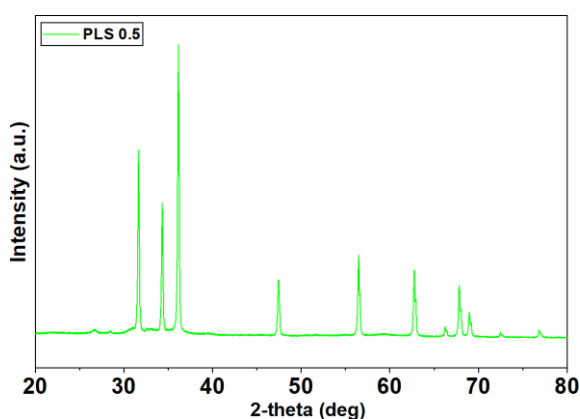


Figure 2. XRD diffraction pattern of ZnO-doped activated carbon (PLS-0.5).

The main diffraction peaks were observed at 2θ angles of 31.66° , 34.32° , and 36.14° . These three peaks are associated with the (100), (002),

and (101) crystal planes of ZnO, which has a hexagonal (wurtzite) structure. This diffraction pattern is consistent with JCPDS standard data No. 36-1451, which serves as a reference for the ZnO crystal phase with a hexagonal wurtzite structure [16, 17].

In addition to the main peak, other peaks were detected at 2θ around 47.43° , 56.48° , and 62.75° , corresponding to the (102), (110), and (103) crystal planes. The presence of these peaks further confirms the formation of a crystalline ZnO phase in the sample [16].

On the other hand, a broad peak in the 2θ range around $26^\circ - 27^\circ$ is observed, indicating the presence of an amorphous carbon structure. This pattern is a common characteristic of biomass-based activated carbon, which generally has a low degree of crystal regularity [18].

The formation of a composite between ZnO and activated carbon also causes ZnO to be dispersed in the carbon matrix. This condition can affect the intensity and distribution of diffraction peaks, thus indicating an interaction between the ZnO and activated carbon phases in the composite material [19].

Overall, the XRD analysis results show that the successfully synthesized material is an activated carbon–ZnO composite with a combination of an amorphous carbon structure and a hexagonal (wurtzite) crystalline ZnO phase. These results indicate that the ZnO synthesis and doping process proceeded successfully.

Functional Group Characterization (FTIR)

The Fourier transform infrared spectroscopy (FTIR) analysis of the ZnO-doped activated carbon (PLS-0.5) sample revealed several absorption bands indicating the presence of several functional groups in the material.

The main absorption bands were detected at wavenumbers 3446.01 cm^{-1} , 2138.45 cm^{-1} , 1630.40 cm^{-1} , 1222.52 cm^{-1} , and 484.73 cm^{-1} .

The broad band at 3446.01 cm^{-1} indicates the presence of O–H stretching vibrations. These groups generally originate from hydroxyl

groups or water molecules adsorbed on the surface of activated carbon and are commonly found in biomass-based activated carbon [18].

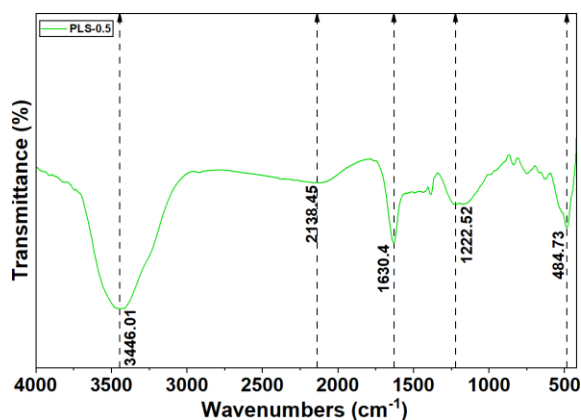


Figure 3. FTIR spectrum of ZnO-doped activated carbon (PLS-0.5).

The absorption band at 1630.40 cm^{-1} is associated with C=O or C=C stretching vibrations. The presence of these bands indicates the presence of aromatic structures or carbonyl groups in the activated carbon material [18].

The band at 1222.52 cm^{-1} is interpreted as C–O stretching vibrations, which are generally associated with alcohol, ether, or ester groups in the activated carbon structure [18].

Meanwhile, the band at 2138.45 cm^{-1} can be associated with vibrations of triple-bonded groups, such as C≡C or C≡N, which likely form during the carbonization process.

At low wavenumbers, at 484.73 cm^{-1} , an absorption band appears indicating Zn–O bond vibrations. This band is a characteristic feature of ZnO and is associated with the metal–oxygen bonding in the material's structure [20].

The presence of this Zn–O bond also confirms the XRD analysis results, which previously indicated the formation of a crystalline ZnO phase. Thus, the FTIR results provide additional evidence that ZnO has been successfully doped and integrated into the activated carbon matrix [19].

In general, the FTIR analysis results confirm the presence of characteristic functional groups in activated carbon and demonstrate the formation of Zn–O bonds, indicating the

successful integration of ZnO into the resulting composite material.

CONCLUSION

Synthesis of activated carbon from oil palm frond biomass through chemical activation using KOH combined with ZnO doping produces a composite material with unique structural characteristics. X-ray diffraction (XRD) patterns indicate the presence of a hexagonal ZnO crystalline phase (wurtzite) at 2θ angles of approximately 31.66° , 34.32° , and 36.14° , dispersed in an amorphous carbon matrix. Identification using Fourier transform infrared (FTIR) shows the presence of O–H, C=O/C=C, and C–O functional groups, as well as a Zn–O band at approximately 484 cm^{-1} as an indicator of ZnO integration in the carbon structure. The combination of these properties indicates that the resulting material has the potential to be further developed in adsorption and catalysis applications.

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