

Synthesis of activated carbon based on palm shells with ZnCl_2 activator and FeCl_3 doping

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ABSTRACT

Biomass-based activated carbon is a sustainable material with strong potential for adsorption and energy storage applications. This study aims to evaluate the physical characteristics of activated carbon derived from oil palm shell biomass, synthesized through preparation stages, chemical activation using ZnCl_2 with FeCl_3 doping (0.1 M), and carbonization at high temperatures under an inert atmosphere. Characterization was conducted using Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). The results indicate the presence of oxygen-containing functional groups ($-\text{OH}$, $\text{C}=\text{O}$, $\text{C}-\text{O}$) as well as $\text{Fe}-\text{O}$ bonds, and a turbostratic amorphous carbon structure. Therefore, the produced activated carbon exhibits favorable characteristics as a functional carbon material and shows strong potential for further applications.

Keywords: Activated carbon; FTIR; FeCl_3 ; oil palm shell; XRD

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INTRODUCTION

Activated carbon is a porous carbonaceous material widely used across various industries, including water treatment, pollutant adsorption, catalysis, and energy storage. This is due to its large specific surface area and exceptional adsorption capacity. The high demand for activated carbon has spurred research into biomass waste as an abundant, inexpensive, and sustainable alternative raw material. In line with the principles of a circular economy, biomass can be utilized to transform industrial waste into high-value materials [1,2].

As one of the world's largest palm oil producers, Indonesia generates a significant quantity of palm kernel shells annually. These shells possess a high lignocellulosic content—comprising cellulose, hemicellulose, and lignin—making them a highly promising raw material for the production of high-quality activated carbon [3,4]. Utilizing palm kernel shell waste for activated carbon offers economic value and

supports environmentally friendly industrial waste management and the circular economy [2].

Chemical activation using ZnCl_2 has proven effective in developing a porous structure and increasing the specific surface area of activated carbon through dehydration and pore-formation mechanisms during the carbonization process [1,5]. Combining ZnCl_2 activation with FeCl_3 doping is intended to introduce oxygen-rich functional groups onto the carbon surface while simultaneously enhancing the material's electrical conductivity. Consequently, this chemical activation approach is expected to yield activated carbon with more optimal structural characteristics [6]. The addition of transition metal dopants, such as FeCl_3 , can increase the number of active sites on the carbon surface, which can facilitate various types of adsorption processes and energy storage applications [6,7].

The identification of surface functional groups and the determination of the crystalline structure of activated carbon materials can be carried out using standard characterization

methods, namely Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) analysis [6,8]. The objective of this research is to synthesize activated carbon from oil palm shells using ZnCl_2 as an activating agent and 0.1 M FeCl_3 as a dopant. The physical characteristics are analyzed via FTIR and XRD testing to determine the quality and potential of the resulting carbon material.

LITERATURE REVIEW

Biomass

Biomass refers to organic material derived from living organisms—whether animals or plants—that can serve as a source of renewable energy and as a raw material for functional carbon [2]. Due to its high content of cellulose, hemicellulose, and lignin, lignocellulosic biomass is highly significant for the production of activated carbon. Cellulose and hemicellulose contribute to the formation of a porous structure during thermal decomposition, while lignin aids in the development of a stable aromatic carbon structure during the carbonization process [9]. Rice husks [10], sawdust [11], palm kernel shells [4], and various other types of biomass have been utilized as raw materials for activated carbon [7,11,12]. Biomass—often sourced from agricultural and industrial waste—represents an economical and sustainable resource for large-scale activated carbon production. Furthermore, the use of biomass as a raw material aligns with the concept of waste valorization, supporting the implementation of a circular economy and the reduction of carbon emissions [2].

Palm Kernel Shells

Palm kernel shells (from *Elaeis guineensis*) are hard, solid waste materials generated when fresh oil palm fruit bunches are processed into crude palm oil (CPO). As a by-product of the palm oil processing industry, these shells are produced in Indonesia—the world's largest palm oil producer—in quantities reaching millions of tons annually [13]. Palm kernel shells

are exceptionally well-suited for the production of high-quality activated carbon due to their dense lignocellulosic structure and high carbon content; they typically contain 22% – 29% cellulose, 20% – 25% hemicellulose, and 35% – 45% lignin [4,13]. According to Baby et al. [4], activated carbon synthesized from palm kernel shells—also known as palm kernel endocarp—possesses a large specific surface area and exhibits exceptional adsorption capacity when treating water contaminated with heavy metals. Palm kernel shells have significant potential as functional electrochemical materials due to their favorable physical properties, accessibility, low cost, and year-round availability [3].

Activated Carbon

Activated carbon is a porous carbonaceous material characterized by a very high specific surface area (500 – 3000 m^2/g) and a controllable pore structure, ranging from micropores (< 2 nm) and mesopores (2 – 5 nm) to macropores (> 50 nm) [6]. The production of activated carbon typically involves two main stages: first, the raw material is carbonized at high temperatures in an inert atmosphere; subsequently, physical or chemical activation is applied to enhance the pore structure [2,5]. Chemical activation—using agents such as ZnCl_2 , KOH, or H_3PO_4 —generally yields activated carbon with a larger specific surface area compared to physical activation using water vapor or CO_2 [1]; [11]. ZnCl_2 degrades lignocellulose and inhibits tar formation, which facilitates the development of a more extensive pore structure during the carbonization process [5,9]. In a study by Gueye et al. [6], hierarchical porous activated carbon was synthesized from biomass via a two-step FeCl_3/KOH activation process for supercapacitor applications. The findings indicate that doping with FeCl_3 contributes to the incorporation of oxygen-rich functional groups onto the carbon surface. Biomass-derived activated carbons have been widely studied as adsorbents, supercapacitor electrodes, and other functional materials due to their superior physical properties and low production costs [7,10,14].

FTIR

FTIR is a spectroscopic technique used to identify chemical functional groups in a material based on the absorption patterns of infrared radiation at specific wavenumbers [6]. Since each functional group possesses a distinct vibrational frequency, an FTIR spectrum can provide comprehensive information regarding the chemical composition present on the surface of an activated carbon material. FTIR analysis reveals characteristic absorption peaks within specific wavenumber ranges for biomass-based activated carbon. A broad peak between $3200 - 3600\text{ cm}^{-1}$ is attributed to the (O–H) stretching vibration of hydroxyl groups or adsorbed water; a peak at $1600 - 1700\text{ cm}^{-1}$ corresponds to the (C=O) stretching vibration of carbonyl groups; a peak at $1000 - 1200\text{ cm}^{-1}$ is associated with the (C–O) stretching vibration of ether and alcohol groups; and a peak in the $500 - 700\text{ cm}^{-1}$ range can be linked to metal-oxygen bonds, such as Fe–O, resulting from the FeCl_3 doping process [6]. The surface of activated carbon contains oxygen-containing functional groups that are highly significant, as they enhance electrolyte wettability and pseudocapacitive activity—facilitated by reversible Faradaic reactions—thereby improving the electrochemical performance of the material [6,15].

XRD

The XRD characterization method relies on X-ray diffraction patterns and is based on Bragg's law ($n\lambda = 2d \sin\theta$), which relates the X-ray wavelength, the diffraction angle, and the interplanar spacing (d-spacing) [6]. For biomass-derived carbon materials, XRD patterns typically exhibit two broad peaks. The (002) peak appears around $2\theta = 20^\circ - 26^\circ$ and is associated with the interlayer spacing of graphene-like sheets, while the (100) peak appears around $2\theta = 43^\circ - 44^\circ$, reflecting the in-plane structural order of the graphene layers [6,8]. The broadness and low intensity of the diffraction peaks indicate that carbon materials with a low degree of crystallinity possess amorphous

and turbostratic characteristics. A similar semi-crystalline pattern—featuring two peaks at these positions—was observed in activated carbon derived from sugar palm (*Arenga pinnata*) petioles, as identified by Putri and Farma [8]. With a larger d-spacing compared to that of commercial graphite ($3.35\text{ \AA}$), the carbon structure is more expanded and exhibits higher porosity—a characteristic that is advantageous for energy storage and various adsorption applications [6,16].

RESEARCH METHODS

Materials and Equipment

Palm kernel shells are used as the biomass carbon source in this study. Zinc chloride (ZnCl_2) serves as an activating agent, while iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) is used as a dopant. Distilled water is used as a solvent during the mixing and washing processes.

The equipment used includes a hammer mill, digital scale, oven, electric grinder, 100-mesh sieve, hot plate, glass beakers, magnetic stirrer, a tube furnace equipped with a nitrogen (N_2) gas flow system for the carbonization process, porcelain crucibles, litmus paper, 100 ml volumetric flasks, and a pipette filler and vacuum system for washing and neutralization.

Synthesis of Activated Carbon and Doping with $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

Palm kernel shells, having undergone washing and drying, are reduced in size using a hammer mill. The material is then pulverized using a grinder and passed through a 100-mesh sieve.

A 30 g portion of the biomass powder is mixed with ZnCl_2 (dissolved in distilled water) at a 1:1 ratio. For the modified samples, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ is added at a molar ratio of 0.1 (4 g) relative to the carbon/ ZnCl_2 mixture. The mixture is homogenized using a magnetic stirrer for 2 hours until a uniform suspension is obtained.

Following homogenization, the samples are dried prior to the carbonization process. Carbonization is carried out in a furnace at 800°C for 3.5 hours under a nitrogen (N_2) atmosphere.

During this process, ZnCl_2 acts as a chemical activating agent to facilitate the formation of a porous structure, while the $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ becomes dispersed within the carbon matrix. Once carbonized, the samples are cooled to room temperature.

The carbonized samples must be neutralized using distilled water to remove any residual activating agents or by-products. In the final stage, the samples are dried once more to obtain activated carbon in the form of a dry powder.

Material Characterization

Characterization is performed using XRD to analyze the degree of amorphousness of the carbon, its crystal structure, and the presence of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. Additionally, FTIR is used to identify the functional groups present on the surface of the activated carbon.

The overall workflow of the research methodology for the synthesis of activated carbon from oil palm shells is illustrated in Figure 1.



Figure 1. Research activity plan.

RESULTS AND DISCUSSION

FTIR Analysis of 0.1 Molar Palm Kernel Shell Samples

Figure 2 displays the FTIR spectrum of an activated carbon sample derived from palm kernel shells (PKS), prepared using ZnCl_2 as an activator and doped with 0.1 M FeCl_3 . The FTIR analysis reveals four primary absorption peaks at wavenumbers 3413.5 cm^{-1} , 1627.8 cm^{-1} , 1130.9 cm^{-1} , and 621 cm^{-1} . Each peak corresponds to specific functional groups on the synthesized activated carbon surface.

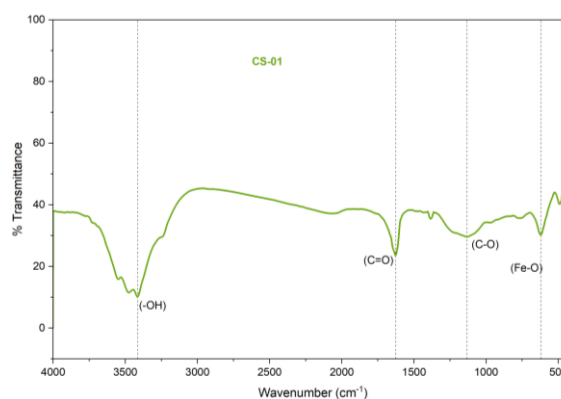


Figure 2. FTIR spectrum.

A broad, deep absorption peak at a wavenumber of 3413.5 cm^{-1} indicates the stretching vibration of $-\text{OH}$ (hydroxyl) groups. These peaks may originate from hydroxyl groups on the carbon surface—derived either from physically adsorbed water or from phenolic groups formed during the carbonization and chemical activation processes. In a study by Gueye et al. [6], a broad absorption band in the $3200 - 3600\text{ cm}^{-1}$ range was identified as a characteristic feature of alcohol or phenolic groups in biomass-based activated carbon activated with FeCl_3 . The presence of these intense $-\text{OH}$ groups is consistent with the current findings. On the surface of activated carbon, hydroxyl groups enhance the wettability of the carbon surface by various solvents and increase the surface reactivity of the resulting activated carbon material [6,14].

The absorption peak at a wavenumber of 1627.8 cm^{-1} corresponds to the stretching vibration of $\text{C}=\text{O}$ (carbonyl) groups. This may be attributed to ketone or carboxylate groups formed on the carbon surface as a result of oxidation occurring during the chemical activation process with ZnCl_2 . This finding is consistent with the characterization results for biomass-based activated carbon reported by Gueye et al. [6] and Maslahat et al. [14]. On the surface of activated carbon, carbonyl groups serve as functional active sites that enhance the material's adsorption capacity and surface reactivity. They also contribute to improving the structural stability of the synthesized carbon [6,14].

The absorption peak at a wavenumber of 1130.9 cm^{-1} indicates the stretching vibration of

C–O groups, associated with ether linkages (C–O–C) or alcohol groups on the carbon surface. The presence of these relatively strong C–O groups points to a high content of oxygen-containing functional groups on the activated carbon surface, resulting from the ZnCl_2 activation process. This supports the findings of Gueye et al. [6], who observed that C–O–C peaks were more pronounced in activated carbon samples processed at lower temperatures, suggesting better retention of oxygen-containing functional groups. C–O groups on the carbon surface also facilitate the adsorption of various pollutants and target molecules by the activated carbon material [6,10].

The absorption peak observed at a wave-number of 621 cm^{-1} indicates the presence of Fe–O bonds, resulting from the FeCl_3 doping process, which successfully incorporated iron atoms into the activated carbon structure. The bonding of iron atoms to the carbon surface via oxygen groups forms Fe–O–C complexes, which help enhance the material's electrical conductivity and provide additional active sites on the activated carbon surface. Overall, the FTIR spectrum of the CW-01 sample reveals that the palm-shell-derived activated carbon possesses a surface rich in oxygen-containing functional groups (–OH, C=O, C–O), as well as Fe–O bonds originating from the iron chloride doping. The presence of these functional groups works synergistically to improve the adsorption capacity, surface reactivity, and overall quality of the resulting activated carbon material [6,10,14].

XRD Analysis of 0.1 Molar Palm Kernel Shell Sample

The X-ray diffraction (XRD) pattern obtained from the palm kernel shell-derived activated carbon sample, CW-01, is presented in Figure 3. The XRD pattern exhibits characteristics of an amorphous or turbostratic carbon material, with several identifiable diffraction features. The pattern generally shows a relatively high background intensity at low angles, indicating that an amorphous carbon phase dominates the sample.

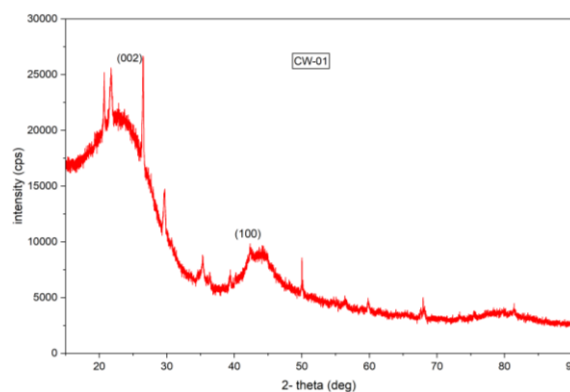


Figure 3. XRD spectrum.

A broad diffraction hump observed at approximately $2\theta = 22^\circ - 23^\circ$ is characteristic of the (002) plane in a turbostratic amorphous carbon structure. The breadth of this peak suggests a low degree of crystallinity, with the graphenic layers arranged irregularly and lacking a clear orientation. These results align with findings by Gueye et al. [6], where a broad peak in the $2\theta = 15^\circ - 25^\circ$ range indicates the amorphous nature of carbon material activated with FeCl_3/KOH . Furthermore, activated carbon derived from sugar palm (*Arenga pinnata*) petioles, activated using KOH, also exhibits a semi-crystalline peak at the (002) plane around 24° [8]. The d-spacing value for the (002) plane peak is approximately $3.59\text{ \AA}$, which is larger than that of commercial graphite ($3.35\text{ \AA}$) [6]. A larger d-spacing value indicates a more expanded and porous carbon structure. Both of these characteristics contribute to an increased surface area and enhanced adsorption capacity in the activated carbon material [6,16,17].

The 2θ range of $43^\circ - 44^\circ$ exhibits a second broad diffraction peak, corresponding to the (100) and (101) planes of the in-plane structure of the activated carbon. Despite its low intensity and broad shape, the presence of this peak indicates that the carbon material possesses local in-plane order, even in the absence of high overall crystallinity. In a study by Gueye et al. [6], similar results were observed for activated carbon derived from jackfruit biomass. Diffraction peaks at $2\theta = 24.4^\circ$ and 43.6° suggest that the amorphous biomass exhibits good electrical conductivity. Furthermore, a narrower peak appears atop the amorphous hump at approxi-

mately $2\theta = 26^\circ - 27^\circ$; this can be attributed to the formation of small crystalline graphitic phases during the carbonization process, indicating the existence of localized regions with a higher degree of graphenic order within the amorphous carbon matrix.

Overall, the XRD analysis of the CW-01 M sample reveals that the palm shell-derived activated carbon—synthesized via ZnCl_2 activation and FeCl_3 doping—possesses a predominantly amorphous/turbostratic structure with a d-spacing of 3.59 Å. Characterized by a high degree of amorphousness, an expanded d-spacing, and localized graphenic order, this material exhibits excellent structural properties, making it a highly promising functional activated carbon for applications in adsorption and energy storage [6,8,16].

CONCLUSION

Based on the research results and analysis conducted, it can be concluded that the synthesis of activated carbon from palm kernel shell biomass—as observed in sample CW-01 via FTIR analysis—yields four primary absorption peaks: an –OH group at 3413.5 cm^{-1} , a C=O group at 1627.8 cm^{-1} , a C–O group at 1130.9 cm^{-1} , and an Fe–O bond at 621 cm^{-1} . This confirms that the activation with ZnCl_2 and doping with FeCl_3 successfully functionalized the carbon surface.

XRD analysis of sample CW-01 reveals a predominantly amorphous/turbostratic carbon structure, indicated by a broad (002) peak at approximately $2\theta = 22^\circ - 23^\circ$ and a (100) peak at $2\theta = 43^\circ - 44^\circ$. Overall, the activated carbon derived from palm kernel shells and doped with 0.1 M FeCl_3 possesses a combination of an amorphous structure with large d-spacing, a surface rich in oxygen-containing functional groups, and active Fe–O sites—all of which contribute to enhanced adsorption capacity and surface reactivity of the material.

AUTHOR CONTRIBUTIONS

Dhea Nur Haliza: Investigation, analysis, visualization, original draft. Iwan Nugraha: Con-

ceptualization, methodology, supervision, review and editing. Nidya Chitraningrum: Methodology, resources. Achmad Subhan: Validation, analysis. Hana Arisesa: Validation, review and editing. Irma Apriyani: Analysis, review and editing. All authors approved the manuscript.

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DECLARATION OF AI USE

The authors declare that no AI tools were used to prepare this manuscript.

CONFLICT OF INTEREST

The authors declare no conflicts of interest.

DATA AVAILABILITY STATEMENT

All supporting data are included in this article. No additional datasets are available.

ETHICAL APPROVAL

Ethical approval was unnecessary, as no humans or animals were involved.

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