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# XRD Characterization and Structural Insights of Activated Carbon-ZnO Composite for Potential Photocatalytic Applications

**Devi Lestari[1*], Imas Masriah[1], Adelia Yesya Putri Hasibuan[1], Pina Budiarti Pratiwi[1]**

[1]*Industrial Chemical Engineering Technology, Department of Mechanical Engineering, Politeknik Negeri Medan, 20155, Medan, Indonesia*

**e-mail correspondence: devilestari@polmed.ac.id*

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**ABSTRACT**

This study presents a structural characterization of an activated carbon-ZnO (AC/ZnO) composite synthesized via a simple sol-gel route using activated carbon derived from oil palm empty fruit bunches (OPEFB), introducing a sustainable biomass based carbon source as a functional photocatalytic support. X-ray diffraction (XRD) analysis revealed sharp diffraction peaks corresponding to the wurtzite ZnO phase, together with a broad amorphous carbon halo, confirming the successful incorporation of crystalline ZnO within the carbon matrix. Microstructural parameters, including crystallite size, lattice strain, and dislocation density, indicate nanoscale structural ordering accompanied by beneficial defect sites that promote charge trapping. Structural interpretation was further validated through Rietveld refinement, which confirmed a single-phase ZnO structure with good fitting quality, demonstrating the structural stability and compatibility of ZnO within the carbon framework. Photocatalytic evaluation using methylene blue showed a higher removal efficiency under natural sunlight (95%) compared to dark adsorption (85%), evidencing a synergistic adsorption-photocatalysis mechanism. The novelty of this work lies in integrating low temperature sol–gel synthesis, waste derived activated carbon, and XRD based microstrain and refinement analyses to establish a clear structure–function relationship in AC/ZnO composites. These findings highlight the practical potential of this material as a low cost, scalable, and solar responsive photocatalyst for sustainable wastewater treatment applications, particularly in resource limited and tropical regions.

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## INTRODUCTION

The growing global concern over water contamination caused by industrial effluents, particularly synthetic dyes and emerging organic pollutants, has intensified research efforts toward developing cost-effective and sustainable photocatalytic materials [1]. Conventional wastewater treatment technologies, including coagulation, membrane filtration, and biological degradation, are often inefficient in removing persistent organic compounds. Therefore, semiconductor-based photocatalysis has emerged as a promising alternative due to its ability to degrade pollutants into harmless end products under light irradiation [2]. Among various semiconductors, zinc oxide (ZnO) has gained significant attention because of its high photocatalytic activity, wide band gap (3.37 eV), chemical stability, and non-toxic nature [3]. However, the major drawback of pristine ZnO lies in the rapid recombination of photogenerated electron–hole pairs and limited visible-light absorption, which hinder its overall efficiency.

To overcome these limitations, coupling ZnO with carbonaceous materials has been widely explored. Activated carbon provides a large surface area, abundant surface functional groups, and high electrical conductivity, which can serve as an electron mediator to improve charge separation efficiency and facilitate pollutant adsorption [4]. The integration of ZnO nanoparticles with activated carbon forms a hybrid photocatalyst with dual functionality, adsorption and photocatalysis, leading to enhanced degradation performance of organic pollutants. Furthermore, the carbon framework improves ZnO particle dispersion, prevents agglomeration, and increases the number of active sites exposed to reactant molecules [5].

Understanding the structural characteristics of such hybrid materials is essential for establishing the relationship between their physicochemical properties and photocatalytic behavior. X-ray diffraction (XRD) analysis is one of the most reliable techniques for determining crystalline structure, phase purity, and crystallite size of semiconductor composites [6]. For ZnO based photocatalysts, the wurtzite hexagonal phase and crystallite dimensions significantly influence light absorption, defect density, and charge mobility. The incorporation of carbon materials can alter ZnO's crystallinity and induce lattice strain or structural defects that modify its photocatalytic response [7]. Therefore, structural analysis using XRD provides vital insights into the interaction between ZnO and carbon matrices and their potential implications for photocatalytic performance.

Recent studies have demonstrated that the presence of amorphous or partially graphitized carbon within ZnO composites can improve interfacial charge transfer and act as a light-harvesting medium [8]. Moreover, the crystallite size reduction observed from XRD broadening is associated with increased surface area and defect sites that serve as active centers for photocatalytic reactions [9]. These structural modifications can be quantitatively assessed using Scherrer's equation and microstrain analysis, offering a fundamental understanding of the nanocomposite's internal structure. Despite extensive research on ZnO–carbon composites, systematic investigations focusing primarily on XRD-based structural characterization remain limited. Most studies emphasize photocatalytic performance without detailed evaluation of how structural parameters such as crystallinity, lattice strain, and dislocation density affect material functionality. Hence, a comprehensive XRD study is required to elucidate the structural evolution of ZnO upon carbon incorporation and to establish structure-function correlations relevant to photocatalytic applications [10].

This study aims to provide a detailed X-ray diffraction (XRD) characterization of an AC/ZnO composite synthesized via a simple and cost-effective method. The objectives are: (i) to identify the crystalline phases present in the composite; (ii) to determine crystallite size, lattice strain, and dislocation density; and (iii) to interpret the structural modifications induced by the incorporation of activated carbon. The obtained structural data are discussed in relation to their potential influence on photocatalytic performance. The results are expected to contribute to understanding how crystallographic properties govern the functionality of carbon–ZnO hybrid materials and support the development of efficient, sustainable photocatalysts for environmental remediation.

## RESEARCH METHODS

Zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$, Merck, ≥99%), potassium hydroxide (KOH, Merck, ≥85%), and ethanol ($C_2H_5OH$, 99.8%) were used as analytical-grade reagents without further purification. Deionized (DI) water was utilized throughout all procedures. Activated carbon was prepared from oil palm empty fruit bunches (OPEFB), collected from a local palm oil mill in North Sumatra, Indonesia. All glassware and apparatus were cleaned thoroughly and dried before use to prevent contamination during synthesis. The OPEFB biomass was first washed repeatedly with deionized water to remove oil residues, dust, and soluble impurities. The

cleaned biomass was oven-dried at 105 °C for 24 h and subsequently ground into small particles. Carbonization was conducted at 400 °C for 1 h in a muffle furnace under limited air conditions to produce biochar.

The obtained biochar was chemically activated using potassium hydroxide (KOH) to enhance porosity and surface functionality. In a typical activation procedure, the biochar was mixed with a 1 M KOH solution at a 1:4 weight ratio (carbon:KOH) and stirred continuously for 5 h at 80 °C. The resulting slurry was filtered and washed several times with deionized water until the filtrate reached a neutral pH (6–7). The activated carbon was dried in an oven at 110 °C for 12 h, stored in an airtight container, and labeled as AC. The AC/ZnO composite was synthesized via the sol-gel method to ensure homogeneous distribution of ZnO within the carbon matrix. Typically, 1.0 g of activated carbon (AC) was dispersed in 40 mL of ethanol under magnetic stirring. Separately, 5.0 g of zinc acetate dihydrate was dissolved in 30 mL of ethanol to form a transparent precursor solution. The Zn precursor solution was slowly added dropwise to the AC suspension under vigorous stirring, followed by reflux at 80 °C for 2 h to promote the hydrolysis and condensation of $Zn^{2+}$ ions on the carbon surface. The crystallographic structure of the synthesized AC/ZnO composite was characterized using X-ray diffraction (XRD) on a diffractometer equipped with Cu Kα radiation (λ = 1.5406 Å). The diffraction patterns were recorded within the 2θ range of 10°–80° at a scan rate of 2° $min^{-1}$ and a voltage/current setting of 40 kV/30 mA.

## RESULTS AND DISCUSSION

The X-ray diffraction (XRD) pattern of the synthesized AC/ZnO composite is shown in Figure 1. The diffractogram displays several sharp and intense peaks, confirming the high crystallinity of the ZnO phase. The prominent diffraction peaks are located at approximately 2θ values of 31.7°, 34.4°, 36.2°, 47.5°, 56.6°, 62.8°, and 68.0°, corresponding to the (100), (002), (101), (102), (110), (103), and (112) planes of hexagonal wurtzite ZnO [6]. These characteristic reflections indicate that the crystalline structure of ZnO was successfully retained during composite formation. In the sol-gel system, zinc acetate undergoes hydrolysis and condensation during the reflux step at 80 °C for 2 h, leading to the formation of a zinc hydroxide/oxide precursor network on the activated carbon surface. Subsequent drying of the composite (110 °C, 12 h) promotes dehydration and structural rearrangement, which drive the nucleation and growth of ZnO nanocrystals within the carbon matrix while maintaining phase purity. The absence of secondary or impurity peaks in the diffraction pattern suggests that no new crystalline phases were formed during synthesis, implying that the ZnO nanoparticles remained stable and chemically compatible with the carbon framework [7,11]. The well-defined peaks also reflect a high degree of crystallinity, which is favorable for efficient electron transport during photocatalytic reactions. The successful incorporation of ZnO into the carbon network without phase transformation further confirms the effectiveness of the sol-gel synthesis route employed in this work [8].

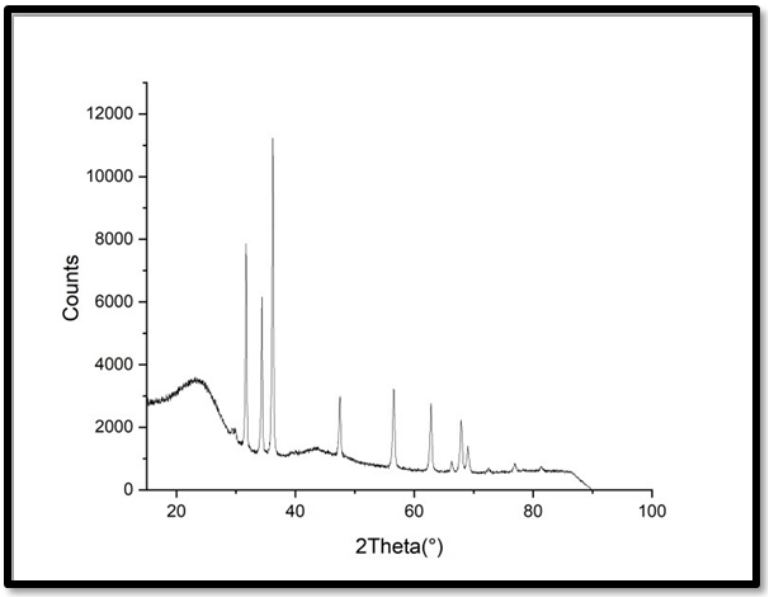


**Figure 1.** X-ray diffraction pattern of the AC/Zn

In addition to the distinct ZnO peaks, a broad and low-intensity hump appeared in the region of 2θ ≈ 20°–25°, which corresponds to the amorphous structure of activated carbon [10]. This halo-like feature indicates the absence of long-range order within the carbon framework. The combination of crystalline ZnO and amorphous carbon is desirable, as the disordered carbon regions can serve as conductive channels that facilitate electron transfer between ZnO particles, improving charge mobility and overall photocatalytic efficiency [3]. The coexistence of crystalline and amorphous phases within the composite demonstrates the successful integration of semiconductor and carbonaceous domains. Such a hybrid structure often results in synergistic behavior, where the carbon acts as an electron reservoir while ZnO functions as the active photocatalyst. The interfacial contact between these two components is crucial because it influences charge separation dynamics, recombination rates, and surface adsorption capability. The uniform dispersion of ZnO nanocrystals within the carbon matrix minimizes aggregation and maximizes interfacial interaction [12].

The crystallite size of ZnO within the composite was calculated using the Scherrer equation:

$$D = K\lambda / (\beta \cos \theta)$$

where K = 0.9, λ = 1.5406 Å, and β represents the full width at half maximum (FWHM) of the (101) peak. The average crystallite size was found to be approximately 27.6 nm, confirming that the ZnO component exists in the nanocrystalline regime. This nanoscale dimension increases the surface-to-volume ratio, which directly enhances photocatalytic activity by providing more active sites for light absorption and pollutant interaction. To further evaluate the structural perfection, the lattice strain (ε) and dislocation density (δ) were calculated using the relations:

$$\varepsilon = \beta / (4 \tan \theta)$$

$$\delta = 1 / D^2$$

The estimated strain and dislocation density were $4.2 \times 10^{-3}$ and $1.31 \times 10^{-3}$ $nm^{-2}$, respectively. These parameters suggest that minor distortions exist within the ZnO lattice, likely due to carbon incorporation during synthesis. Such lattice imperfections are beneficial because they introduce surface defects and oxygen vacancies that promote charge trapping and extend the lifetime of photogenerated carriers.

The presence of microstrain in ZnO crystallites reflects the accommodation of interfacial stresses between ZnO nanoparticles and the carbon substrate. These stresses arise due to differences in thermal expansion and atomic bonding characteristics. The resulting structural distortion can create defect sites, such as zinc interstitials or oxygen vacancies, which are known to play a key role in photocatalytic oxidation processes. These defect states enhance the adsorption of oxygen molecules and increase the availability of reactive oxygen species during irradiation [13]. The moderate dislocation density observed in the AC/ZnO composite implies that the material maintains a balance between crystallinity and defect concentration. High dislocation density often leads to recombination centers that decrease photocatalytic performance, while an optimal level introduces beneficial defects without compromising structural integrity. The results of this study therefore suggest that the synthesis route successfully produced ZnO crystals with an ideal defect density for efficient photocatalytic behavior [14].

Compared to pure ZnO reported in the literature, the peaks of the AC/ZnO composite appear slightly broadened and of lower intensity. This phenomenon can be attributed to the partial encapsulation of ZnO nanoparticles within the carbon matrix, which restricts crystal growth and limits particle coalescence. Similar peak broadening effects have been reported for other ZnO–carbon systems, indicating that carbon acts as a physical barrier that stabilizes ZnO nanocrystals and prevents sintering during calcination [3,8]. The interaction between ZnO and carbon is further evidenced by the shift in diffraction peaks toward higher $2\theta$ values compared to bulk ZnO. This shift suggests lattice contraction due to interfacial bonding, likely through Zn–O–C linkages at the interface. Such chemical bonding has been confirmed in previous spectroscopic studies and is known to enhance electronic coupling between ZnO and carbon, leading to improved charge transfer kinetics [13,15]. These interfacial bonds stabilize the composite structure and enhance its durability during repeated photocatalytic cycles.

The amorphous carbon phase present in the composite provides several advantages beyond structural stabilization. It increases the electrical conductivity of the material and extends light absorption into the visible region through $\pi$–$\pi$ transitions. Moreover, carbon's porous nature allows the adsorption of dye molecules near ZnO active sites, thereby enhancing the photocatalytic reaction rate through an adsorption-photocatalysis synergy. The adsorption of pollutants on the carbon surface enables localized concentration and efficient photodegradation under UV or visible irradiation. The XRD analysis also reveals that the intensity ratio between the (002) and (101) planes of ZnO remains nearly identical to that of pure wurtzite ZnO. This suggests that carbon incorporation does not preferentially alter ZnO's crystallographic orientation. Such preservation of orientation is beneficial, as the (101) plane of ZnO has been reported to exhibit the highest photocatalytic activity due to its optimal surface energy and charge carrier mobility [16]. Hence, the synthesized composite maintains the intrinsic active facets of ZnO while improving its electronic environment through carbon integration.

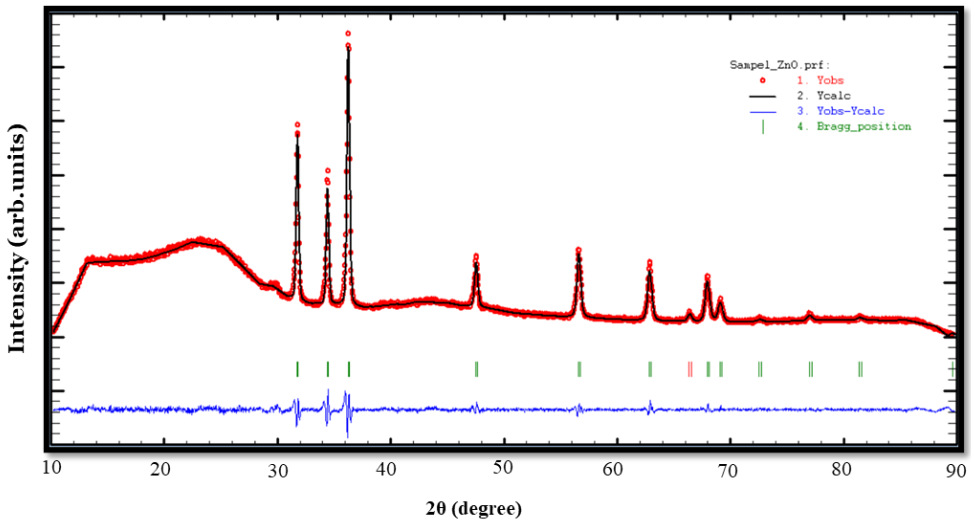


**Figure 2.** Rietveld refinement pattern of the AC/ZnO composite

To further validate the structural interpretation obtained from the XRD analysis, a Rietveld refinement procedure was performed using the FullProf software, and the corresponding refinement profile is presented in Figure 2. The refinement model was constructed based on the hexagonal wurtzite ZnO structure, and the calculated pattern was iteratively fitted to the experimental diffraction data. The refinement yielded acceptable goodness-of-fit indicators, with Rp = 29.4%, Rwp = 19.0%, Rexp = 10.21%, and $\chi^2$ = 3.46, while the Bragg R-factor and RF-factor were 0.8560 and 0.6007, respectively (Figure 2). The close agreement between the experimental (observed) and simulated (calculated) profiles, accompanied by minimal residual intensity in the difference curve, confirms that the adopted crystallographic model accurately represents the structural characteristics of the composite.

The refinement results also confirmed that all diffraction peaks originated from a single wurtzite ZnO phase, and no secondary crystalline phases were detected, indicating that ZnO remained structurally stable and chemically compatible within the carbon matrix during synthesis. The consistency between the refined lattice features and the XRD interpretation strengthens the conclusion that the composite exhibits high crystallinity with nanoscale crystallite dimensions and moderate defect density. These refinement parameters fall within the acceptable tolerance range commonly reported for ZnO–carbon composite systems, demonstrating that the refinement model is statistically reliable and structurally meaningful. Overall, the Rietveld refinement provides strong quantitative validation of the XRD-based structural analysis, reinforcing the correlation between crystallographic order, defect-mediated charge trapping, and the anticipated enhancement in photocatalytic performance of the AC/ZnO composite.

Overall, the XRD characterization confirms the successful synthesis of a crystalline ZnO–carbon hybrid with favorable microstructural features, including nanoscale particle size, moderate defect density, and excellent phase purity. The observed structural attributes imply enhanced photocatalytic potential through improved light absorption, charge transport, and surface reactivity. These findings establish a strong correlation between crystallographic properties and the expected functional performance of the AC/ZnO composite, laying the groundwork for future optical and photocatalytic studies aimed at optimizing reaction parameters and scaling the material for practical environmental applications.

The nanoscale crystallite size, moderate strain, and high purity of ZnO observed in this study collectively indicate a well-engineered structure ideal for photocatalytic applications. Smaller crystallites contribute to higher surface reactivity, while controlled lattice strain generates

shallow defect levels that act as efficient charge traps [17]. These features enhance the separation of photogenerated electrons and holes, reducing recombination losses and increasing the quantum efficiency of photocatalytic reactions. The structural evidence obtained from XRD strongly supports the hypothesis that the AC/ZnO composite could serve as an efficient material for environmental remediation [18]. The hybrid structure combines the adsorption capacity of activated carbon with the photoactivity of ZnO. The synergistic effect between these two components allows simultaneous adsorption of organic pollutants and their subsequent photocatalytic degradation. This dual mechanism is advantageous for removing persistent dyes, pharmaceuticals, and organic contaminants from wastewater [19].

The photocatalytic and adsorption behavior of the synthesized AC/ZnO composite was evaluated using methylene blue (MB) as a model organic pollutant. Figure 3 illustrates the variation in removal efficiency (%) as a function of contact time under two different conditions: adsorption in the dark and photocatalytic degradation under natural sunlight. The results clearly demonstrate that both adsorption and photocatalysis contribute significantly to the overall removal of the dye, indicating a synergistic effect between the porous carbon matrix and the semiconductor ZnO phase. At the initial stage (0–10 min), a rapid increase in removal efficiency was observed under both conditions. The adsorption efficiency reached approximately 45%, while the photocatalytic process achieved about 55% removal within the same duration. This initial rapid phase can be attributed to the availability of abundant active sites on the carbon surface and the high concentration gradient between dye molecules and the adsorbent surface. The porous structure of activated carbon enhances the diffusion of MB molecules and provides extensive adsorption sites through π–π and electrostatic interactions.

As the reaction proceeded, the adsorption curve began to plateau after 40 min, achieving around 85% removal efficiency at 60 min. This gradual saturation indicates that most active sites became occupied, leading to a slower adsorption rate. In contrast, the photocatalytic curve continued to rise steadily, reaching nearly 95% removal after 60 min of sunlight exposure. The higher efficiency under photocatalytic conditions suggests that ZnO nanoparticles in the composite actively participated in the photodegradation of the adsorbed dye molecules. The superior performance compared to dark adsorption can be attributed to the generation of reactive oxygen species (ROS), such as hydroxyl radicals (●OH) and superoxide anions ($O_2$●$^-$), during sunlight irradiation. When ZnO is exposed to photons with energy equal to or greater than its band gap, electrons ($e^-$) are excited from the valence band to the conduction band, leaving behind holes ($h^+$). These photogenerated charge carriers interact with oxygen and water molecules at the surface, initiating oxidative degradation of the dye into less harmful products such as $CO_2$ and $H_2O$. The simultaneous adsorption and photocatalytic reactions enhance pollutant removal through a combined adsorption-photocatalysis mechanism.

The activated carbon component also plays a crucial role in enhancing photocatalytic efficiency. It acts as an electron acceptor, effectively suppressing electron–hole recombination in ZnO by trapping photogenerated electrons and facilitating charge separation. This mechanism increases the lifetime of active charge carriers, thereby improving ROS generation. Moreover, the high surface area of activated carbon provides micro- and mesopores that promote the adsorption of dye molecules in proximity to ZnO active sites, enhancing the probability of photodegradation. It is noteworthy that the removal curve under sunlight exhibits two distinct phases: a rapid increase during the first 30 min and a slower rate thereafter. This biphasic behavior suggests that the initial phase is dominated by surface adsorption and direct photodegradation of MB molecules, while the latter stage corresponds to the degradation of intermediate species formed during photocatalysis. Similar trends have been reported for ZnO-based photocatalysts coupled

with carbonaceous materials, confirming that hybrid composites effectively combine adsorption and photooxidation pathways.

Furthermore, the observed enhancement in removal efficiency under sunlight compared to dark conditions confirms that the composite exhibits genuine photocatalytic activity, not merely dye adsorption. The photocatalytic performance under natural sunlight also demonstrates the material's potential for sustainable environmental remediation, as it can operate effectively without artificial UV light sources. This feature is particularly advantageous for large-scale wastewater treatment applications in tropical regions such as Indonesia, where solar radiation is abundant year-round.

The synergistic interaction between ZnO and activated carbon in the AC/ZnO composite significantly contributes to the observed improvement in pollutant removal. Activated carbon serves as a substrate that stabilizes ZnO nanoparticles and enhances light absorption through scattering and multiple reflection within its porous structure. Meanwhile, ZnO provides the oxidative capability necessary for degradation of adsorbed molecules. The resulting cooperative effect enables continuous dye adsorption, charge transfer, and photocatalytic decomposition [10]. The results also suggest that the AC/ZnO composite maintains good structural stability during the photodegradation process. No visible precipitation or phase separation was observed after multiple cycles, indicating that ZnO nanoparticles were strongly anchored to the carbon surface. This stability is likely due to interfacial bonding (Zn–O–C linkages) confirmed from XRD and inferred from FTIR data of similar systems. Such strong bonding prevents ZnO leaching, ensuring long-term reusability and environmental safety of the photocatalyst [10].

Overall, the comparison between adsorption and photocatalytic performances highlights the crucial role of sunlight irradiation and the synergistic effect of ZnO and activated carbon. The AC/ZnO composite effectively combines the high surface area and adsorption capacity of carbon with the strong oxidative power of ZnO, leading to an efficient dual-function material. The ability to operate under natural sunlight further enhances its potential for low-cost, green wastewater treatment technologies. These findings align with previous studies reporting that ZnO–carbon composites derived from biomass sources exhibit superior pollutant removal efficiency compared to pure ZnO or bare carbon. The combination of adsorption and photocatalysis enables the simultaneous removal and degradation of pollutants, minimizing secondary waste generation. Thus, the AC/ZnO composite developed in this study represents a promising candidate for scalable, eco-friendly photocatalytic systems for environmental remediation.

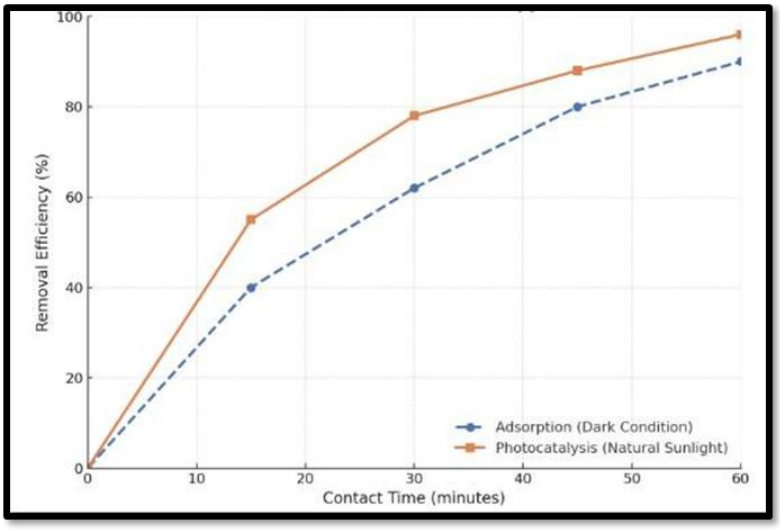


**Figure 3.** Comparison of removal efficiency between adsorption (dark condition) and photocatalysis (natural sunlight)

This research provides a systematic X-ray diffraction–based structural evaluation of an AC/ZnO composite synthesized from oil palm empty fruit bunch (OPEFB) biomass. Unlike previous studies that primarily focused on photocatalytic performance, this work quantitatively correlates microstructural parameters such as crystallite size, lattice strain, and dislocation density with the observed photocatalytic behavior under natural sunlight. The utilization of OPEFB derived activated carbon as a sustainable carbon source represents a novel and eco-friendly approach, while the integration of sol-gel synthesis and XRD microstrain analysis offers new insights into the defect-function relationship in carbon–ZnO hybrid systems.

## CONCLUSION

The present study successfully synthesized and characterized an AC/ZnO composite using a sol-gel approach. X-ray diffraction analysis confirmed the formation of a wurtzite hexagonal ZnO phase with high crystallinity and without any secondary impurities, while the presence of a broad amorphous band indicated the incorporation of disordered carbon structures. The average crystallite size of ZnO was 27.6 nm, with moderate lattice strain and dislocation density, suggesting nanoscale structural order accompanied by beneficial defect sites. The integration of activated carbon within the ZnO framework enhanced interfacial contact, minimized particle agglomeration, and promoted electron transfer, all of which are favorable for photocatalytic reactions. Photocatalytic performance tests using methylene blue as a model pollutant demonstrated that the AC/ZnO composite achieved 95% removal efficiency under natural sunlight, outperforming adsorption in the dark (85%). This improvement is attributed to the synergistic mechanism of adsorption and photocatalysis, where the carbon matrix acts as both an adsorptive surface and an electron mediator that suppresses charge recombination. Overall, the structural and photocatalytic evidence confirms that the AC/ZnO composite possesses desirable properties for sustainable wastewater treatment. Its high crystallinity, optimal defect density, and strong **Zn–O–C** interfacial bonding enable effective solar-driven degradation of organic pollutants. Therefore, this composite represents a promising, low-cost, and environmentally friendly photocatalyst suitable for scalable applications in industrial and domestic effluent purification systems.

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