

ENGINEERING ZnO/BIOCHAR COMPOSITES FOR ENHANCED PHOTOCATALYTIC PERFORMANCE: MECHANISTIC ELUCIDATION OF TETRACYCLINE DEGRADATION AND UV-VIS SPECTROSCOPY-BASED KINETIC ANALYSIS

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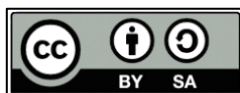
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ABSTRAK

Antibiotik tetrasiklin yang mencemari lingkungan perairan sulit dihilangkan dengan metode konvensional karena sifatnya yang stabil dan persisten. Penelitian ini bertujuan mengembangkan komposit ZnO/Biochar serta mengevaluasi aktivitas fotokatalitiknya dalam degradasi tetrasiklin. Metode penelitian menggunakan eksperimen laboratorium dengan variasi massa katalis dan waktu penyinaran UV-Vis. Konsentrasi tetrasiklin dianalisis menggunakan spektrofotometer UV-Vis pada panjang gelombang 378 nm. Katalis 0,5 g memberikan kinerja optimum dengan efisiensi degradasi sebesar 84% setelah 120 menit penyinaran. Peningkatan jumlah katalis hingga batas optimum meningkatkan efisiensi degradasi, sedangkan penggunaan berlebih menurunkan efektivitas akibat meningkatnya kekeruhan sistem. Analisis kinetika menunjukkan bahwa degradasi mengikuti model orde satu semu dengan konstanta laju 0,0142 menit⁻¹. Hasil penelitian menunjukkan bahwa komposit ZnO/Biochar berpotensi sebagai material fotokatalis untuk pengolahan limbah antibiotik.

Kata kunci: biochar, fotokatalisis, kinetika, tetrasiklin, ZnO

ABSTRACT

Tetracycline contamination in aquatic environments is difficult to remove using conventional treatment methods due to its stable and persistent nature. This study aimed to develop ZnO/Biochar composites and evaluate their photocatalytic activity for tetracycline degradation. Laboratory experiments were conducted by varying catalyst dosage and UV-Vis irradiation time. Tetracycline concentration was monitored using UV-Vis spectrophotometry at 378 nm. Catalyst dosage of 0.5 g provided optimum performance, achieving 84% degradation efficiency after 120 min of irradiation. Increasing catalyst dosage up to the optimum level improved degradation efficiency, whereas excessive amounts reduced effectiveness due to increased turbidity. Kinetic analysis indicated that the degradation followed a pseudo-first-order model with a rate constant of 0.0142 min⁻¹. These findings demonstrate that ZnO/Biochar composites have potential as photocatalytic materials for antibiotic wastewater treatment.

Keywords: biochar, kinetics, photocatalysis, tetracycline, ZnO

INTRODUCTION

The global escalation in antibiotic consumption over the past two decades has not only raised public health concerns but has also imposed significant pressure on environmental sustainability. Reports from the World Health Organization indicate that antibiotic usage has increased by approximately 35% since 2000, with the most substantial growth observed in developing countries where wastewater treatment systems remain limited. In addition, the United Nations Environment Programme highlights that more than 50% of pharmaceutical compounds are still detected in aquatic environments at concentrations ranging from 1 to 500 µg/L, even after undergoing conventional treatment processes (Hosny *et al.*, 2022).

Tetracycline is among the most frequently detected antibiotics in the environment and is characterized by its high stability and persistence. Its complex molecular structure hinders effective degradation through both biological and physicochemical pathways. Previous studies have reported that tetracycline can persist for up to 60 days in natural waters, whereas conventional treatment methods typically achieve only 30–40% removal efficiency and may lead to the formation of toxic intermediate by-products (Zhang *et al.*, 2023)

To address these limitations, Advanced Oxidation Processes (AOPs) have been developed, among which semiconductor-based photocatalysis has gained considerable attention. This approach utilizes light energy to generate reactive oxidative species, such as hydroxyl and superoxide radicals, which are capable of degrading complex organic pollutants into simpler and less harmful compounds (Zhang *et al.*, 2023). Zinc oxide (ZnO) is widely employed as a photocatalyst due to its band gap energy of approximately 3.2 eV, good chemical stability, and cost-effectiveness (Adesina *et al.*, 2024). However, pristine ZnO suffers from a high recombination rate of electron–hole pairs, which limits its photocatalytic efficiency. One strategy to enhance ZnO performance is through its integration with carbon-based materials such as biochar. Biochar possesses a porous structure and large surface area, enabling improved initial adsorption of pollutants and facilitating charge transfer, thereby enhancing photocatalytic degradation efficiency (Hosny *et al.*, 2022). Previous studies have demonstrated that ZnO/Biochar composites significantly improve tetracycline degradation efficiency; however, investigations concerning the influence of operational parameters, including catalyst dosage and irradiation time, remain limited (Tuama *et al.*, 2024). This study aims to evaluate the effectiveness of ZnO/Biochar photocatalysts in degrading tetracycline under varying catalyst dosages (0; 0.1; 0.5; and 1 g) and irradiation times (0–120 minutes) using UV–Vis

light. Furthermore, the reaction kinetics are analyzed using a pseudo-first-order model to obtain a quantitative understanding of the photocatalytic degradation process.

METHOD

Tools and Materials

The materials used in this study included tetracycline hydrochloride (Suprabiatic® Tetracycline HCl 500 mg, PT Phapros Tbk., Indonesia) as a model pharmaceutical pollutant, zinc oxide (ZnO, Smart-Lab, Indonesia) as the photocatalytic semiconductor material, 96% ethanol (Smart-Lab, Indonesia), methanol analytical grade (Merck, Germany), and distilled water (Bratachem, Indonesia). Biochar was produced from corn cob biomass obtained from local farmers in Bandar Lampung, Lampung, Indonesia. All chemicals were of analytical grade and used without further purification.

The instruments used in this study included an analytical balance (Ohaus Pioneer PX224, USA), drying oven (Mettler UN55, Germany), muffle furnace (Nabertherm L3/11, Germany), ultrasonic bath (Branson 2800, USA), and hotplate magnetic stirrer (IKA C-MAG HS 7, Germany). Material characterization was carried out using Fourier Transform Infrared Spectroscopy (FTIR) (Shimadzu IRTracer-100, Japan), X-Ray Diffraction (XRD) (PANalytical X'Pert PRO, Netherlands), and Scanning Electron Microscopy (SEM) (JEOL JSM-6510LV, Japan). Tetracycline concentration was analyzed using a UV–Visible spectrophotometer (Shimadzu UV-1800, Japan) equipped with deuterium and tungsten-halogen lamps, a monochromator, a quartz cuvette, and a photodiode detector. Photodegradation experiments were conducted using a 15 W UV-C lamp (Philips, Netherlands) and a 250 W UV–Vis lamp as irradiation sources. Standard laboratory glassware (Pyrex, USA) was also used throughout the experimental procedures.

Procedure

Biochar Preparation : Corn cobs were washed thoroughly with distilled water to remove adhering impurities and subsequently dried under sunlight, followed by oven drying at 105°C to reduce moisture content. The dried biomass was carbonized in a muffle furnace to produce biochar. The resulting biochar was ground and sieved to obtain a uniform particle size before being used for composite synthesis.

Synthesis of ZnO/Biochar Composite : The ZnO/Biochar composite was synthesized using an ultrasonication-assisted method. Biochar was dispersed in a suitable solvent and homogenized using an ultrasonic bath. ZnO was then added to the suspension, followed by continuous stirring using a hotplate magnetic stirrer until a homogeneous mixture was obtained. The resulting suspension was subsequently dried and thermally treated to enhance structural stability and interfacial interaction between ZnO and biochar.

Characterization : The synthesized ZnO/Biochar composite was characterized using FTIR to identify surface functional groups, XRD to determine crystalline phases and crystal structure, and SEM to investigate surface morphology and particle distribution. These analyses were conducted to evaluate the physicochemical properties of the synthesized composite.

Photocatalytic Degradation of Tetracycline : The photocatalytic activity of the ZnO/Biochar composite was evaluated through the degradation of tetracycline solution. Photodegradation experiments were conducted under UV–Vis irradiation (250 W), UV-C irradiation (15 W), and dark conditions as a control. Prior to the photocatalytic experiments, the maximum absorption wavelength (λ_{max}) of tetracycline was determined, and a calibration curve was established using a series of standard tetracycline solutions. The ZnO/Biochar composite was then added to the tetracycline solution and the mixture was homogenized before irradiation. Samples were collected at predetermined time intervals and separated from the photocatalyst prior to analysis. The concentration of tetracycline was determined using a UV–Vis spectrophotometer based on absorbance measurements at λ_{max} . The degradation efficiency was calculated from the percentage reduction in tetracycline concentration after the photocatalytic treatment.

Characterization of ZnO, Biochar, and ZnO/Biochar Composite Materials

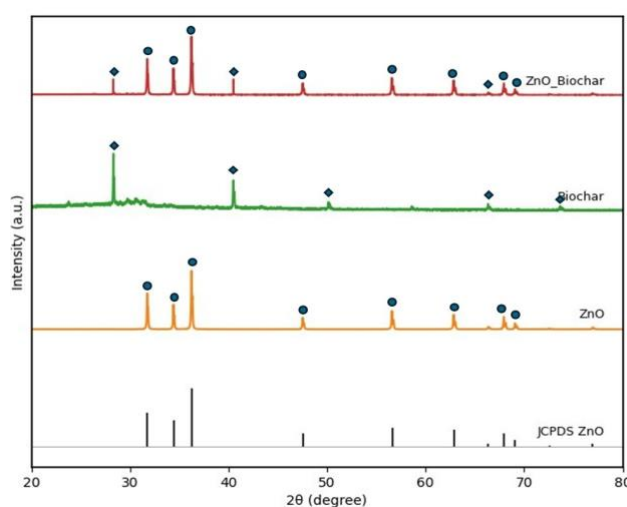


Figure 1. XRD Patterns of ZnO, Biochar, and ZnO/Biochar Composite

As shown in Figure 1 the XRD patterns of ZnO, biochar, and the ZnO/Biochar composite compared to the JCPDS standard. ZnO exhibits sharp diffraction peaks, confirming its crystalline hexagonal (wurtzite) structure and high phase purity, while biochar shows broad, low-intensity peaks indicating its amorphous nature. In the composite, the characteristic ZnO peaks remain visible with slightly reduced intensity, suggesting that the crystalline structure is preserved and ZnO is well dispersed within the biochar matrix without the formation of new crystalline phases.

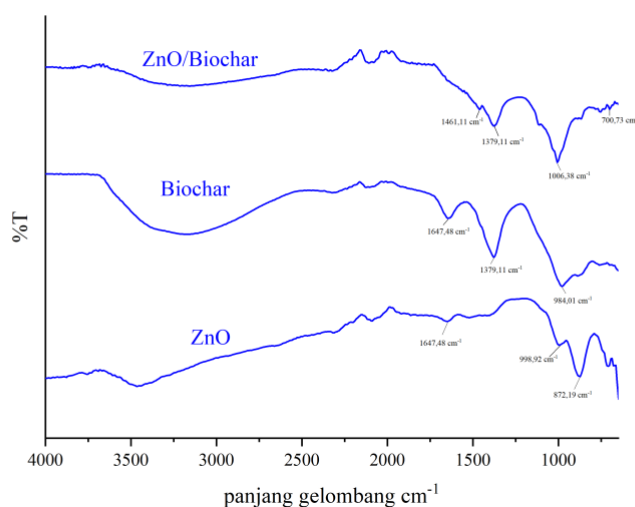


Figure 2. FTIR Spectra of ZnO, Biochar, and ZnO/Biochar Composite

As shown in Figure 2 the FTIR spectra of ZnO, biochar, and the ZnO/Biochar composite, confirming the presence of characteristic functional groups. Biochar shows absorption bands of O–H, C=O, and C–O, indicating oxygen-containing groups, while ZnO exhibits bands below 1000 cm^{-1} corresponding to Zn–O vibrations. In the composite, the characteristic peaks of both materials are retained with slight shifts in intensity and position, suggesting interactions between ZnO and biochar. These results confirm successful composite formation and enhanced interfacial interactions that may improve photocatalytic performance.

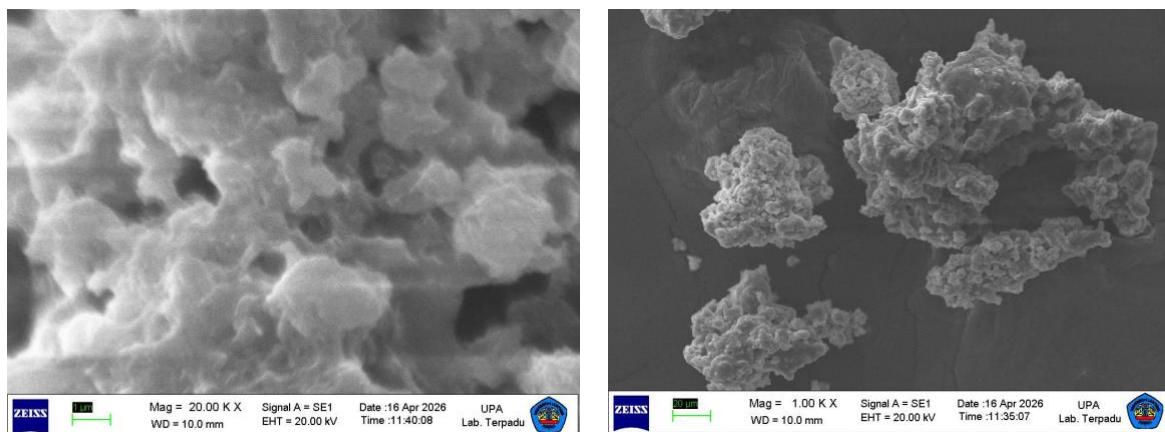


Figure 3. SEM Images of Biochar

As shown in Figure 3 SEM images of biochar at 1,000 $\times$ (a) and 20,000 $\times$ (b). The images reveal irregular agglomerated particles with a rough and highly porous structure, including interconnected pores and uneven textures. This well-developed porous network provides a large surface area and abundant active sites, supporting its effectiveness as a support material for enhancing the photocatalytic performance of the ZnO/Biochar composite.

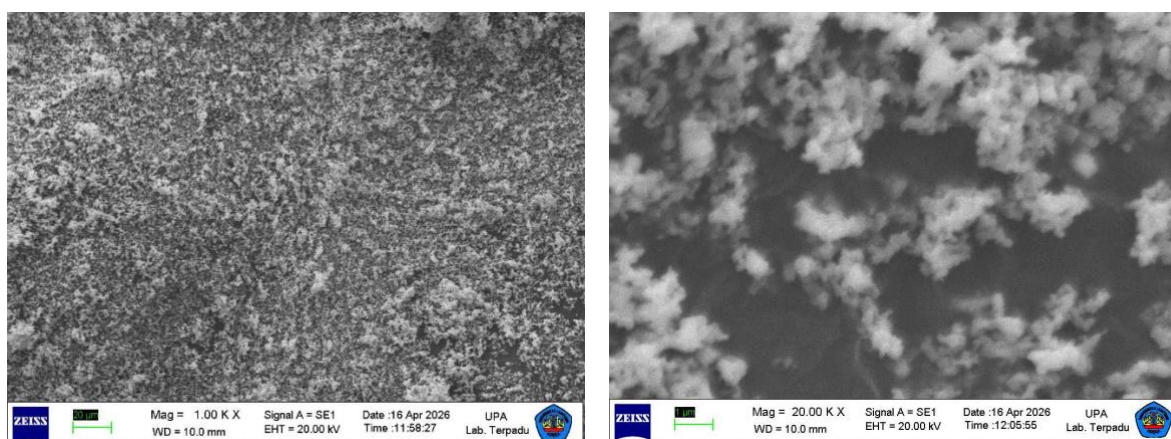


Figure 4. SEM Images of ZnO

The results depicted in Figure 4 indicate SEM images of ZnO at magnifications of 1,000 $\times$ (a) and 20,000 $\times$ (b). ZnO appears as relatively uniform agglomerated particles, with higher magnification revealing clustered granular structures indicative of nanoparticle formation. The surface is denser and less porous than biochar, reflecting its crystalline nature, with some agglomeration due to high surface energy. Overall, morphology confirms the successful formation of ZnO particles contributing to photocatalytic activity.

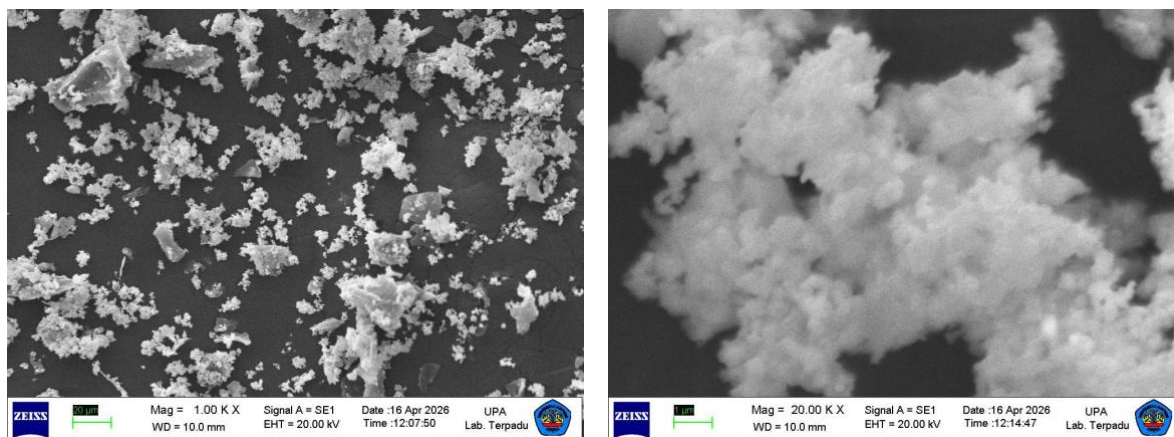


Figure 5. SEM Images of ZnO/Biochar Composite

The results depicted in Figure 5 indicate SEM images of the ZnO/Biochar composite at magnifications of 1,000 $\times$ (a) and 20,000 $\times$ (b). The composite exhibits a heterogeneous structure with ZnO particles dispersed on the biochar matrix, indicating successful integration. At higher magnification, ZnO appears anchored on the porous biochar surface, forming interconnected clusters. Compared to pure ZnO, the composite shows better dispersion and higher porosity, providing more active sites and improved charge separation, which enhances photocatalytic performance.

Procedure

A tetracycline model solution was prepared, and its maximum absorbance wavelength was identified using UV–Vis spectrophotometry. Photocatalytic degradation tests were then performed by dispersing ZnO/Biochar composites into the solution with catalyst loadings of 0, 0.1, 0.5, and 1 g under batch conditions, followed by exposure to UV–Vis irradiation.

Samples were periodically withdrawn at 0, 30, 60, 90, and 120 minutes during the reaction process. Each sample was analyzed spectrophotometrically to record its absorption spectrum. The degradation behavior was evaluated based on the variation in absorbance intensity of tetracycline at its characteristic maximum wavelength.

RESULTS AND DISCUSSION

1. Calibration Curve of Standard Tetracycline Solution

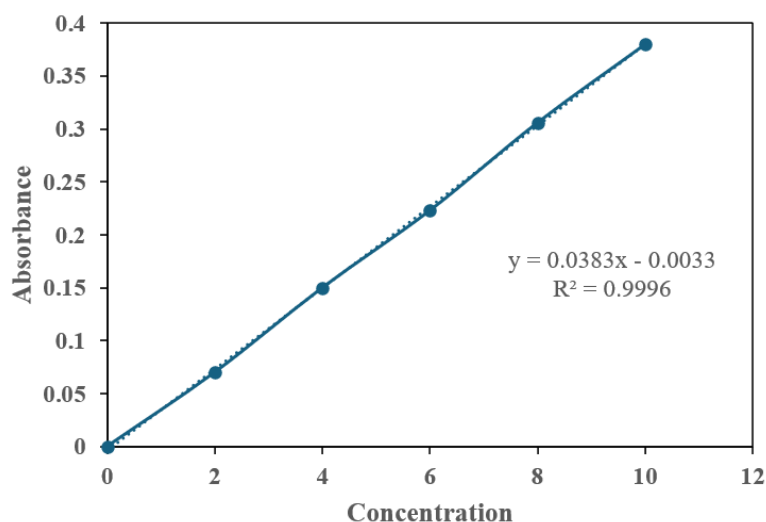


Figure 6. Calibration Curve of Standard Tetracycline

The results depicted in Figure 6 indicate the measurement results, yielding a linear regression equation of $y = 0.0383x - 0.0033$ with a coefficient of determination (R^2) of 0.9996. The exceptionally high R^2 value indicates a strong linear relationship between concentration and absorbance, demonstrating excellent correlation. These findings suggest that the analytical method employed provides high accuracy in determining tetracycline concentrations in the samples studied.

2. Effect of ZnO/Biochar Photocatalyst Loading on the Degradation of Model Antibiotic Wastewater

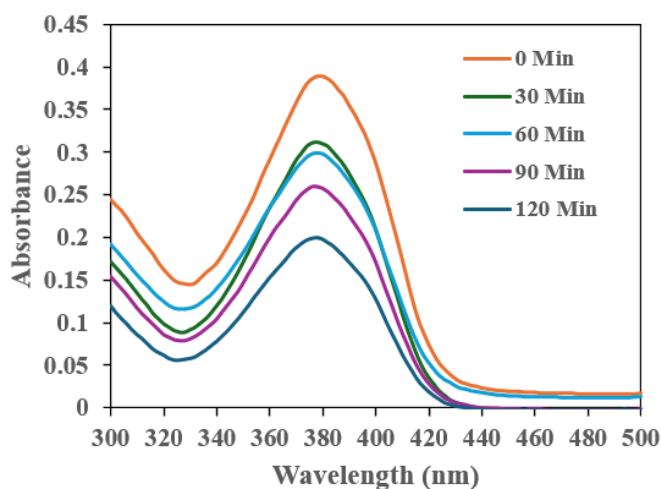


Figure 7. Photodegradation Results of Model Tetracycline Wastewater (0 g)

Figure 7 illustrates the UV–Vis absorption spectra of tetracycline within the wavelength range of 300–500 nm in the absence of a photocatalyst (0 g). The maximum absorbance at 378 nm corresponds to the characteristic λ_{max} of tetracycline, where the compound exhibits its highest absorbance and greatest sensitivity to concentration changes. Monitoring absorbance at λ_{max} allows more accurate determination of tetracycline concentration during the degradation process. A gradual decrease in absorbance at 378 nm was observed throughout the irradiation period (0–120 min), indicating a reduction in tetracycline concentration, although the change remained relatively limited under catalyst-free conditions. These results suggest that tetracycline degradation proceeds slowly in the absence of a photocatalyst

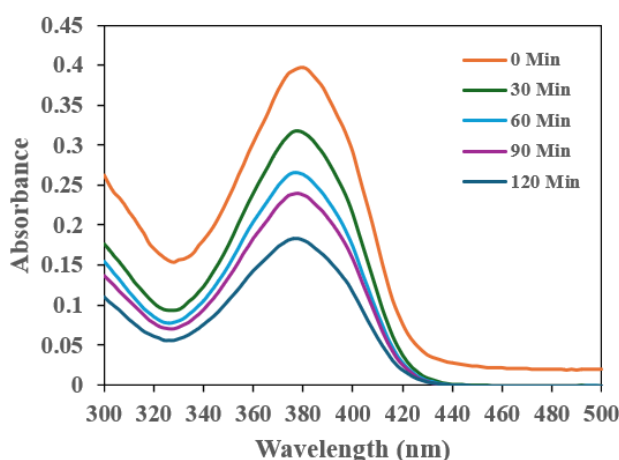


Figure 8. Photodegradation Results of Model Tetracycline Wastewater (0.1 g)

Figure 8 illustrates the UV–Vis absorption spectra of tetracycline over the wavelength range of 300–500 nm under catalyst-free conditions (0.1g). The highest absorbance peak is observed at 378 nm, which represents the characteristic maximum wavelength (λ_{max}) of tetracycline. A slight and gradual decline in absorbance at this wavelength is observed throughout the irradiation period (0–120 minutes), indicating only minimal changes over time. The absence of a catalyst suggests that the degradation of tetracycline occurs at a relatively slow rate.

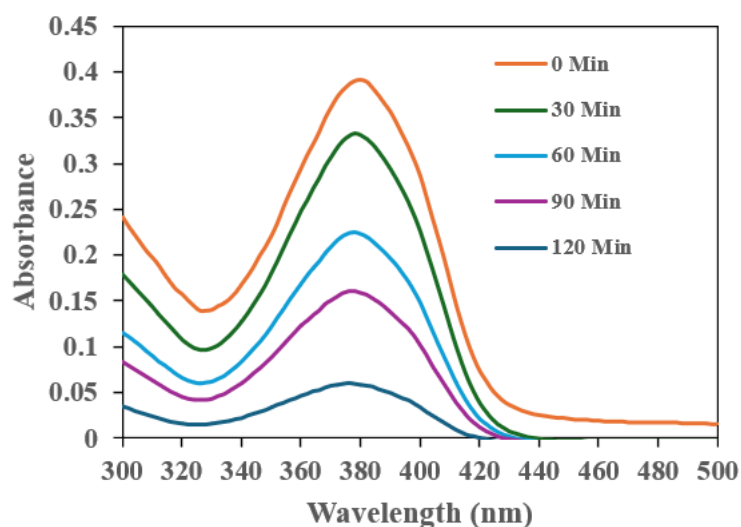


Figure 9. Photodegradation Results of Model Tetracycline Wastewater (0.5 g)

Figure 9 illustrates the UV–Vis absorption spectra of tetracycline within the wavelength range of 300–500 nm in the presence of 0.5 g of photocatalyst. The maximum absorbance peak is located at 378 nm, corresponding to the characteristic λ_{max} of tetracycline. A pronounced decrease in absorbance at this wavelength is observed over the irradiation period (0–120 minutes). The greater reduction in absorbance compared to previous conditions indicates a significant enhancement in tetracycline degradation efficiency upon the addition of 0.5 g of catalyst.

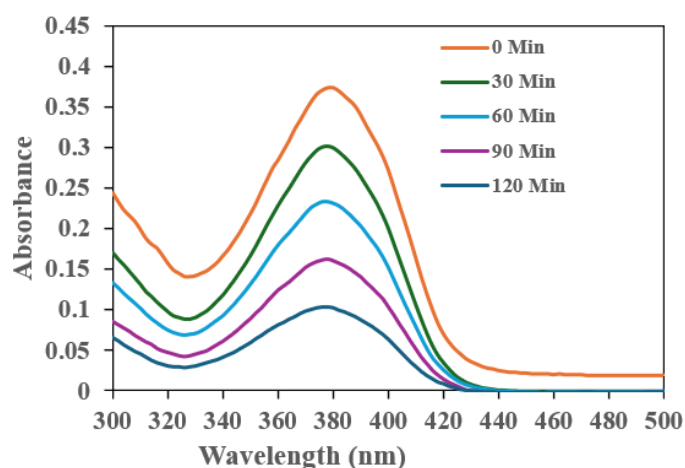


Figure 10. Photodegradation Results of Model Tetracycline Wastewater (1 g)

Figure 10 illustrates the UV–Vis absorption spectra of tetracycline over the wavelength range of 300–500 nm in the presence of 1 g of photocatalyst. The maximum absorbance is observed at 378 nm, representing the characteristic λ_{max} of tetracycline. A progressive decline

in absorbance at this wavelength is observed throughout the irradiation period (0–120 minutes). However, the magnitude of this decrease is lower than that obtained with a catalyst dosage of 0.5 g, indicating that increasing the catalyst amount to 1 g does not improve the degradation performance and is comparatively less effective than the 0.5 g condition.

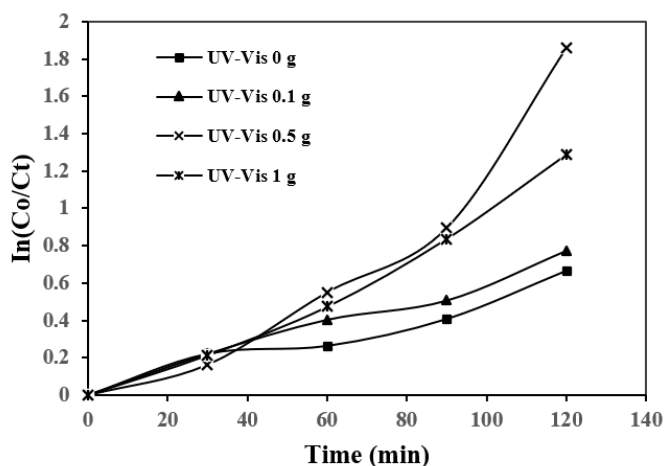


Figure 11. Photocatalytic Reaction Kinetics

Figure 11 illustrates the relationship between $\ln(C_0/C_t)$ and reaction time (minutes) at different catalyst loadings of 0, 0.1, 0.5, and 1 g. The $\ln(C_0/C_t)$ values increase progressively with time for all variations, indicating that tetracycline degradation proceeds throughout the reaction. The most significant increase is observed at a catalyst dosage of 0.5 g, suggesting the highest degradation rate under this condition. In contrast, the use of 1 g results in lower values compared to 0.5 g, implying a decline in efficiency at higher catalyst loading. Meanwhile, the 0 g and 0.1 g conditions exhibit more gradual increases, reflecting slower degradation rates.

Discussion

Material characterization was conducted using XRD, FTIR, and SEM analyses to evaluate the crystal structure, functional groups, and surface morphology of ZnO, biochar, and the ZnO/Biochar composite. The XRD results reveal that ZnO exhibits sharp diffraction peaks consistent with the JCPDS standard, confirming its hexagonal (wurtzite) crystalline structure with high phase purity (Adesina *et al.*, 2024). In contrast, biochar shows broad peaks with low intensity, indicating its predominantly amorphous nature. In the ZnO/Biochar composite, the characteristic peaks of ZnO remain present with slightly reduced intensity, suggesting that the crystalline structure of ZnO is preserved and well dispersed within the biochar matrix without the formation of new crystalline phases (Tuama *et al.*, 2024).

FTIR analysis further supports these findings by identifying characteristic functional groups in each material. Biochar displays absorption bands corresponding to O–H, C=O, and C–O groups, indicating the presence of oxygen-containing functional groups (Hosny *et al.*, 2022). ZnO exhibits absorption bands in the low wavenumber region associated with Zn–O vibrations (Adesina *et al.*, 2024). In the composite, the characteristic peaks of both ZnO and biochar are retained with slight shifts in position and intensity, indicating interactions between ZnO and the functional groups of biochar and confirming successful composite formation (Tuama *et al.*, 2024).

SEM analysis shows that biochar possesses a rough, irregular, and highly porous surface with interconnected pore structures, providing a large surface area and abundant active sites (Hosny *et al.*, 2022). In contrast, ZnO exhibits a denser morphology with agglomerated granular particles, reflecting its crystalline nature (Adesina *et al.*, 2024). In the ZnO/Biochar composite, ZnO particles are uniformly dispersed and anchored onto the porous biochar matrix, resulting in improved particle distribution and enhanced interfacial contact between both components. The porous structure of biochar promotes the adsorption of tetracycline molecules, increasing their proximity to the photocatalytically active ZnO sites. Furthermore, biochar can act as an electron acceptor and transfer medium, facilitating the migration of photogenerated electrons from ZnO and suppressing electron–hole recombination. This synergistic interaction enhances charge separation efficiency and promotes the generation of reactive oxidative species, thereby improving the overall photocatalytic degradation performance of the ZnO/Biochar composite (Tuama *et al.*, 2024).

The calibration curve of the standard tetracycline solution demonstrates a strong linear relationship between concentration and absorbance (Figure 5), described by the regression equation $y = 0.0383x - 0.0033$ with a coefficient of determination (R^2) of 0.9996. The R^2 value, which is close to unity, indicates excellent linearity of the UV–Vis spectrophotometric method, confirming its high accuracy and precision in determining tetracycline concentrations. These results validate the reliability of the analytical method for further experimental analysis (Zahid *et al.*, 2025).

Tetracycline exhibits a maximum absorption peak at a wavelength of 378 nm, which is selected as the analytical wavelength (λ_{max}) due to its optimal sensitivity toward concentration changes. At this wavelength, even small variations in tetracycline concentration result in measurable changes in absorbance, allowing more accurate and reliable quantitative analysis during the photocatalytic degradation process (Taher *et al.*, 2025). The obtained λ_{max} is

consistent with the characteristic ultraviolet absorption behavior of tetracycline, which arises from electronic transitions within its conjugated aromatic ring system. Therefore, 378 nm was used as the reference wavelength for monitoring tetracycline concentration under all experimental conditions (Zhang *et al.*, 2023).

The variation in tetracycline absorption spectra under different ZnO/Biochar catalyst loadings is presented in Figures 6–10, representing conditions without catalyst (0 g) and with catalyst additions of 0.1 g, 0.5 g, and 1 g. The results indicate a gradual decrease in absorbance with increasing irradiation time, reflecting the progressive degradation of tetracycline in the solution (Mahmoudi *et al.*, 2024). This decline confirms the reduction in tetracycline concentration as a result of the photocatalytic degradation process (Li *et al.*, 2022).

The degradation process is governed by the photocatalytic activity of the ZnO/Biochar composite, which generates electron–hole pairs upon UV–Vis irradiation. Excited electrons in the conduction band and holes in the valence band interact with water and dissolved oxygen to produce reactive oxidative species, such as hydroxyl radicals ($\bullet\text{OH}$) and superoxide radicals ($\text{O}_2\bullet^-$) (Luo *et al.*, 2026). Compared to pure ZnO, the incorporation of biochar provides additional advantages due to its porous structure, large surface area, and strong adsorption capacity. These properties promote the adsorption of tetracycline molecules onto the catalyst surface, increasing their contact with photocatalytically active sites. Furthermore, biochar can facilitate electron transfer and suppress the recombination of photogenerated electron–hole pairs, thereby improving charge separation efficiency and enhancing the generation of reactive oxidative species. As a result, the ZnO/Biochar composite exhibits improved photocatalytic performance for tetracycline degradation compared to pure ZnO (Iyyappan *et al.*, 2024).

The comparison of catalyst loadings indicates that increasing the catalyst amount from 0.1 g to 0.5 g results in a more pronounced decrease in absorbance compared to the catalyst-free condition (0 g). This improvement suggests that a higher catalyst dosage provides more active sites, thereby enhancing the interaction between the catalyst surface and tetracycline molecules during the photocatalytic process (Bekhit *et al.*, 2024). However, further increasing the dosage to 1 g does not lead to a corresponding improvement in degradation efficiency. This phenomenon is likely attributed to increased solution turbidity, which limits light penetration, as well as possible particle agglomeration that reduces the effective surface area of the catalyst (Munien *et al.*, 2025). Which can scatter and absorb a significant portion of the incident UV–Vis light before it reaches the entire catalyst surface. As a result, light penetration into the reaction medium is reduced, leading to non-uniform irradiation and a lower generation of

photoinduced electron–hole pairs and reactive oxidative species. In addition, excessive catalyst particles may agglomerate, decreasing the effective surface area and reducing the number of accessible active sites available for photocatalytic reactions. These combined effects explain the lower degradation performance observed at a catalyst dosage of 1 g compared with the optimum dosage of 0.5 g (Munien *et al.*, 2025).

The gradual decline in absorbance observed over the irradiation period of 0–120 minutes indicates that the photocatalytic degradation proceeds continuously under UV–Vis exposure (Huang *et al.*, 2022). The maximum irradiation time of 120 minutes was selected to provide a sufficient observation period for monitoring changes in tetracycline concentration and evaluating the degradation behavior of the ZnO/Biochar photocatalyst over time. Within this duration, a clear decrease in absorbance was observed, allowing the assessment of photocatalytic activity and degradation kinetics under different catalyst loadings. Longer irradiation times promoted greater light utilization by the catalyst, resulting in the generation of more reactive oxidative species and enhanced degradation performance (Mohtaram *et al.*, 2024). The presence of biochar in the ZnO/Biochar composite also plays a crucial role in enhancing photocatalytic performance. Due to its high surface area and strong adsorption capacity, biochar facilitates greater contact between tetracycline molecules and the catalyst surface. In addition, biochar can act as an electron mediator, suppressing the recombination of electron–hole pairs and thereby improving the overall efficiency of the photocatalytic process (Hosny *et al.*, 2022).

Kinetic analysis, as presented in Figure 11, shows a linear relationship between $\ln(C_0/C_t)$ and time, indicating that the degradation process follows pseudo-first-order kinetics based on the Langmuir–Hinshelwood model. The higher $\ln(C_0/C_t)$ values observed at a catalyst dosage. Therefore, the photocatalytic degradation process was interpreted using the pseudo-first-order kinetic model derived from the Langmuir–Hinshelwood approach. This model was selected based on the observed linear trend of the experimental data throughout the irradiation period. Furthermore, the higher $\ln(C_0/C_t)$ values observed at a catalyst dosage of 0.5 g indicate a faster degradation rate compared to the other catalyst loadings (0 g, 0.1 g, and 1 g). Consequently, the 0.5 g dosage can be considered the optimum condition for enhancing the photocatalytic degradation rate of tetracycline in this system (Kumari *et al.*, 2023).

CONCLUSION

The ZnO/Biochar composite effectively degrades tetracycline under UV–Vis irradiation, with optimal performance at a catalyst loading of 0.5 g. Degradation efficiency increases with irradiation time due to enhanced generation of reactive species. Characterization results (XRD, FTIR, and SEM) confirm successful composite formation, good dispersion of ZnO on porous biochar, and improved active sites, which collectively contribute to enhanced photocatalytic performance.

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