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Investigating the potential of coral fragment-based CaO-ZnO biocomposites for the sustainable photodegradation of chloramphenicol

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ABSTRACT

Environmental pollution from pharmaceutical waste, particularly antibiotics like chloramphenicol, poses significant risks to marine ecosystems. Chloramphenicol, widely used in aquaculture, is often found in wastewater, leading to bacterial resistance and disrupting aquatic environments. Adsorption techniques, particularly using natural adsorbents like coral-derived calcium oxide (CaO), are emerging as cost-effective methods for mitigating such pollution. However, the limited photocatalytic activity of CaO requires enhancement through the incorporation of semiconductor materials such as zinc oxide (ZnO). This study investigates the synthesis and performance of a CaO-ZnO composite derived from coral fragments for the photocatalytic degradation of chloramphenicol in water. The composite was characterized by XRF, FTIR, SEM, and XRD, and its photocatalytic activity was evaluated over a range of degradation times under UV light. Results demonstrated that the CaO-ZnO composite significantly improved chloramphenicol degradation compared to individual CaO and ZnO, with the highest efficiency observed at 240 minutes, reaching 66.99%. These findings highlight the potential of the CaO-ZnO composite as an effective and sustainable solution for chloramphenicol removal from wastewater.

Keywords: coral fragment valorization, environmental chemistry, marine coral, marine biotechnology, marine chemistry, marine pollution, wastewater treatment, waste utilization.

INTRODUCTION

Environmental pollution caused by pharmaceutical industrial waste, particularly active compounds such as antibiotics, hormones, and other chemicals, has become a serious issue for ecosystems, especially marine ecosystems (Eapen et al., 2024). Pharmaceutical waste, often incompletely degraded in conventional wastewater treatment systems, can flow into marine waters, leading to the accumulation of hazardous chemicals and harming marine biota (Fakhri et al., 2024). One frequently identified compound is chloramphenicol, a broad-spectrum antibiotic widely used in the aquaculture sector (Masjidin et al., 2024). The use of chloramphenicol to treat infections in farmed fish and shrimp caused by bacteria and parasites often leaves residues in the environment, increases pathogenic bacterial resistance, and disrupts the balance of aquatic ecosystems (Chi et al., 2024; Dinesh et al., 2022).

Adsorption is a cost-effective and easily applicable technique for effectively reducing the concentration of organic compounds, including antibiotic residues, in wastewater (Karina et al., 2024; Prajaputra et al., 2024; Karina et al., 2023). This process involves the uptake of organic compounds, which accumulate on the surface of adsorbent particles, driven by interactions with active sites on the surface (Jeirani et al., 2017; Prajaputra et al., 2021; Prajaputra et al., 2019; Lubis et al., 2018). The success of adsorption largely depends on surface area, the number of active sites, and the accessibility of the adsorbent to the target compounds (Prajaputra et al., 2019; Pui et al., 2019). Various adsorbents have been tested for chloramphenicol removal, including activated carbon, zeolite, chitosan, and other natural materials and biomass (Falyouna et al., 2022; Ajala et al., 2023). Currently, the development of affordable and locally available adsorbents for wastewater treatment has gained significant attention from researchers to enhance the efficiency and sustainability of the treatment process (Lubis et al., 2016; Abidin et al., 2018; Fahmi et al., 2019; Prajaputra et al., 2021; Zulfahmi et al., 2023).

Coral reef fragments rich in calcium carbonate (CaCO₂) are gaining attention as natural adsorbents for removing organic pollutants from wastewater (Trach et al., 2022; Muzaki et al., 2019). Their abundant availability and porous surface structure offer significant potential for wastewater treatment applications. Through calcination, CaCO₂ from coral fragments can be converted into calcium oxide (CaO), which possesses basic properties, a larger surface area, and more active sites for adsorption. However, while CaO is effective in uptaking various contaminants, its capability is limited to physical adsorption without chemical degradation of pollutants, especially complex organic compounds like chloramphenicol (Mohamed, 2021). The effectiveness of CaO as an adsorbent is also constrained by its low chemical stability and lack of photocatalytic properties, which are essential for pollutant degradation. To address these limitations, CaO needs to be composited with semiconductor materials like ZnO to achieve photocatalytic properties capable of breaking down organic compounds in water. ZnO is known as a semiconductor with a 3.4 eV bandgap, antibacterial properties, chemical stability, and high efficiency for photodegradation (Aldeen et al., 2022). ZnO is widely used in wastewater treatment for its ability to degrade organic compounds through photocatalysis under UV light. However, ZnO faces challenges as a photocatalyst due to its small particles, which tend to remain suspended in water, necessitating its combination with other materials.

Previous studies have shown that ZnO-based composites have been extensively developed for the photocatalytic degradation of chloramphenicol in water. For instance, graphene oxide-ZnO composites have proven effective in enhancing the photocatalytic activity of ZnO due to the high surface area of activated carbon, which accelerates the adsorption and degradation of chloramphenicol (Sodeinde et al., 2022). Similarly, TiO2-ZnO composites have improved photocatalytic efficiency through the synergy between TiO₂ as a strong catalyst and ZnO with stable photocatalytic properties under UV light (Karajz et al., 2024). In addition, bentonite-ZnO composites have been used to address antibiotic contamination in water, with bentonite serving as both an adsorbent and a support for ZnO to enhance its photocatalytic stability (Desnelli et al., 2024). However, despite the promising performance of various ZnO composites, the effectiveness of CaO-ZnO composites for chloramphenicol photocatalysis has not been studied. Therefore, this research aims to synthesize and investigate the performance of CaO-ZnO composites derived from coral reef fragments for the photocatalytic degradation of chloramphenicol in water.

MATERIALS AND METHODS

Materials

This research was conducted in marine chemistry and biotechnology fisheries laboratory, Faculty of Marine and Fisheries, Universitas Syiah Kuala. The primary samples, consisting of marine coral fragments, were collected from the Inong Bale Beach area, Aceh Besar. The acquired samples were then rinsed in distilled water, dried at 100°C, and ground into a powder before used as raw materials for preparing the calcium oxide (CaO). All chemical used in this experiment, including chloramphenicol ($C_{11}H_{12}C_{12}N_2O_5$), hydrogen chloride (HCl), and sodium hydroxide (NaOH) were ordered from Merck with high grade quality.

CaO preparation from coral fragments

The marine coral samples were rinsed and soaked in distilled water to remove impurities and residual salts from seawater. The samples were then dried, ground, and sieved using a 100-mesh sieve. A total of 60 g of coral powder was placed in a porcelain crucible and calcined at 900°C for 3 hours. The resulting CaO was then used for the synthesis of CaO-ZnO composite.

Synthesis of CaO-ZnO biocomposite from marine coral fragments

A total of 2.5 g of CaO was mixed with 125 mL of H_2O and 10 mL of methanol, then stirred

using a hotplate at 40°C for 3 hours until homogeneous. While stirring, 2.5 g of $ZnCl_2$ was gradually added and allowed to react for the designated time. After 3 hours, the mixture was filtered using filter paper and a vacuum pump to obtain the residue. The resulting solid was rinsed with H₂O and dried in an oven at 80°C for approximately 30 minutes until a constant weight was achieved. The dried solid powder was then calcined in a furnace at 900°C for 3 hours to produce the CaO-ZnO biocomposite. The resulting biocomposites were characterized using XRF, FTIR, SEM, and XRD.

Variation in chloramphenicol photodegradation time

In the photocatalytic degradation test, degradation time was used as a parameter to evaluate the effectiveness of the CaO-ZnO composite over time. The degradation times tested were 0.5, 1, 2, 3, and 4 hours, with a chloramphenicol volume of 50 mL and an adsorbent dosage of 0.125 g for each treatment. All experiments were conducted in a closed system under UV light at a wavelength of 273 nm. The chloramphenicol concentration after the photocatalytic process was measured using a UV-Vis spectrophotometer, and the photo removal efficiency of chloramphenicol (%) was calculated using the Equation 1.

Photo-removal efficiency =
$$\frac{C_0 - C_f}{C_0} \times 100\%$$
 (1)

where: C_0 and C_f are the initial concentration of chloramphenicol (mg/L) and the concentration of chloramphenicol after UV irradiation (mg/L), respectively.

RESULTS AND DISCUSSION

Chemical composition of calcined coral fragments and composite

The chemical composition analysis of coral fragments before and after calcination, as well as the CaO-ZnO composite, is shown in Table 1. Calcination resulted in a notable transformation, particularly in calcium oxide (CaO) content. CaO increased significantly from 55.4% in raw coral fragments to 94.8% after calcination, indicating the successful decomposition of calcium carbonate (CaCO₃) into CaO. These results are consistent with the study conducted by Dampang et al. (2021), which reported that the composition of CaO increases after undergoing the calcination process. In the CaO-ZnO composite, the CaO content slightly decreased to 94.6% due to the addition of zinc oxide (ZnO).

Minor components, such as magnesium oxide (MgO) and silicon oxide (SiO), showed slight increases after calcination, from 2.5% to 2.7% and from 0.8% to 0.9%, respectively, while decreasing to 2.2% and 0.6% in the composite. Strontium oxide (SrO) remained constant at 0.4% across all samples, suggesting its stability during calcination and composite synthesis. Trace elements like sodium oxide (Na₂O), potassium oxide (K₂O), iron oxide (Fe₂O₃), and manganese oxide (MnO) showed minimal changes, remaining consistent before and after calcination and in the composite.

The ZnO content increased from 0.05% in calcined coral fragments to 1.15% in the composite, reflecting the integration of ZnO during synthesis. Meanwhile, phosphorus pentoxide (P₂O₅) decreased slightly from 0.4% in raw

Percentage (%) Chemical composition Coral fragment Calcined coral fragment CaO-ZnO composite CaO 55.4 94.8 94.6 MgO 2.5 2.7 2.2 SiO 0.8 0.9 0.6 SrO 0.4 0.4 0.4 Na₂O 0.3 0.3 0.3 K₂O 0.2 0.2 0.2 0.3 0.3 0.2 Fe₂O₂ 0.05 0.05 0.05 MnO ZnO 0.05 0.05 1.15 0.3 P205 0.4 0.3 LOI 39.6 -

Table 1. Chemical composition of coral fragments before an after calcined process

coral fragments to 0.3% in calcined samples and the composite. The loss on ignition (LOI) decreased significantly from 39.6% in raw fragments to negligible levels after calcination, confirming the removal of organic and volatile compounds. This highlights the effective transformation of raw coral into a material predominantly composed of CaO, further enhanced by ZnO in the composite.

FTIR analysis

Figure 1 presents the FTIR spectra of CaO from coral fragments, ZnO, and CaO-ZnO composite. Based on the FTIR spectra, the characteristic peak for CaO was observed at 3643 cm⁻¹, confirming the presence of calcium oxide in the calcined material. For ZnO, a distinct Zn-O peak appeared at 475 cm⁻¹, consistent with its typical fingerprint region. The FTIR spectrum of the CaO-ZnO composite displayed a combination of these peaks, including the CaO peak at 3643 cm⁻¹ and the Zn-O peak at 475 cm⁻¹, indicating the successful integration of both oxides into the composite material. The absence of carbonate (CO_{3²⁻)} peaks in the composite further supported the decomposition of calcium carbonate during calcination. This result aligned with findings by Suwannasingha et al. (2022), where the decrease in carbonate peak intensity after calcination confirmed the transformation of calcium carbonate into calcium oxide. These findings validated that the calcination process effectively converted coral fragments into CaO and confirmed the incorporation of ZnO into the composite.

SEM analysis

The XRD results of CaO, ZnO, and the CaO-ZnO composite, as shown in Figure 2, revealed distinct particle morphologies and distributions for each material. SEM analysis of CaO displayed irregularly shaped particles with rough surfaces, a common characteristic of inorganic materials with varying particle sizes. For ZnO, SEM images exhibited a more uniform morphology with rod-shaped or hexagonal particles, consistent with its typical wurtzite crystal structure, and smoother surfaces compared to CaO. In the CaO-ZnO composite, SEM revealed a mixture of the morphologies of both oxides, with finer and more regular ZnO particles distributed among the larger, irregular CaO particles. Additionally, SEM analysis of the composite may indicate physical bonding or agglomeration between CaO and ZnO particles, suggesting interactions between the two oxides within the composite.

XRD analysis

The XRD results of CaO, ZnO, and the CaO-ZnO composite in Figure 3 revealed the distinct crystallographic characteristics of each material. For CaO, the XRD diffraction pattern exhibited sharp peaks at 20 angles of approximately 8.95° , 17.90° , 27.02° , and 45.67° . In the case of ZnO, the diffraction pattern displayed prominent peaks at 20 angles of around 31.78° , 34.45° , 36.39° , 47.46° , 56.55° , 62.89° , 67.87° , and 69.02° , corresponding to the typical hexagonal wurtzite structure. In the composite, the XRD pattern showed peaks from



Figure 1. FTIR spectra of CaO from coral fragments, ZnO, and CaO-ZnO composite



Figure 2. Morphological of (a) CaO from coral fragments, (b) ZnO, and (c) CaO-ZnO composite

both materials (CaO and ZnO), indicating the successful formation of the CaO-ZnO biocomposite.

Chloramphenicol photodegradation

Figure 4 illustrates the photocatalytic degradation of chloramphenicol (20 mg/L) using CaO, ZnO, and the CaO-ZnO biocomposite over a period of 30 to 240 minutes. The degradation results show distinct patterns in chloramphenicol removal efficiencies for each material, highlighting the differences in photocatalytic performance. CaO demonstrated the lowest removal efficiency, starting at 8.92% after 30 minutes and gradually increasing to only 13.29% at 240 minutes. This suggests that CaO has limited photocatalytic activity, likely due to its poor light absorption capacity and small surface area, which reduce its effectiveness in breaking down chloramphenicol molecules.

ZnO exhibited significantly better performance, with removal efficiency reaching 25.54% at 30 minutes and steadily increasing to 47.75% at 240 minutes. This improvement can be attributed to the wurtzite hexagonal structure of ZnO, which facilitates effective photocatalysis under UV light. When ZnO is exposed to UV light with energy equal to or greater than its bandgap (approximately 3.37 eV), electrons in the valence band are excited to the conduction band, leaving behind holes in the valence band. These electron-hole pairs trigger a series of reactions that produce reactive oxygen species (ROS), such as superoxide radicals (•O₂⁻) and hydroxyl radicals (•OH). The excited electrons reduce oxygen molecules to form superoxide radicals, while the holes oxidize water or hydroxide ions to generate hydroxyl radicals. These highly reactive species then attack and degrade chloramphenicol, breaking it down into smaller, less harmful compounds. The degradation process may result in the formation of aromatic intermediates, such as nitrobenzene derivatives, as well as carboxylic acids like acetic acid, and simple amines from the reduction of the nitro group. Ultimately, under complete degradation, chloramphenicol is mineralized into carbon dioxide and water. The strong oxidizing potential of ZnO holes, combined with its efficient generation of ROS, makes ZnO highly effective in degrading chloramphenicol under UV light, outperforming CaO.



Figure 3. XRD spectra of CaO, ZnO and CaO-ZnO biocomposite



Figure 4. Photocatalytic degradation of chloramphenicol

In contrast, the CaO-ZnO biocomposite exhibited the highest removal efficiency, achieving 43.38% at 30 minutes and reaching 66.99% at 240 minutes. This improvement can be attributed to the synergistic effect between CaO and ZnO, which enhances their photocatalytic performance under UV light. As irradiation time increases, the formation of photogenerated charge carriers (electron-hole pairs) becomes more efficient, leading to a higher photo-removal efficiency of chloramphenicol. ZnO, as a semiconductor, generates electron-hole pairs under UV light, which in turn produce ROS such as hydroxyl radicals and superoxide ions that effectively degrade chloramphenicol. CaO, on the other hand, acts as a promoter for ZnO's photocatalytic activity. The alkaline nature of CaO helps stabilize the surface charge of ZnO, increasing the lifetime of the electron-hole pairs and reducing recombination, a common issue in photocatalytic reactions. Additionally, CaO serves as an electron sink, capturing the excited electrons from ZnO and preventing their recombination with holes. This prolongs the electron's activity in the degradation process, enhancing the overall efficiency of the CaO-ZnO

composite in removing pollutants. Consequently, the composite CaO-ZnO outperforms both individual CaO and ZnO in photocatalytic degradation efficiency.

The performance of various adsorbents in the photo-removal of chloramphenicol is presented in Table 2. Sodeinde et al. (2022) reported that the rGO-ZnO composite achieved a removal efficiency of 90% within 100 minutes. This high performance is attributed to the synergistic effect between reduced graphene oxide (rGO) and ZnO, which facilitates enhanced electron transfer and minimizes electron-hole recombination, leading to improved photocatalytic efficiency. Similarly, the study by Palma et al. (2020) demonstrated the effectiveness of TiO2 and PbS-TiO2 composites in chloramphenicol photo-removal. The PbS-TiO₂ composite achieved a 76% removal efficiency for chloramphenicol at a concentration of 10 mg/L, while TiO₂ exhibited an outstanding 98% removal efficiency for the same concentration after 240 minutes. The exceptional performance of TiO₂ can be attributed to its strong photocatalytic activity, high stability, and efficient generation of reactive oxygen species under UV light.

Chloramphenicol Time (min) Percentage removal (%) Adsorbents References concentration (mg/L) rGO-ZnO composite 100 90% Sodeinde et al., 2022 PbS-TiO₂ composite 240 10 76% Palma et al., 2020 TiO, 10 98% Palma et al., 2020 240 13.29% CaO 240 20 This study This study ZnO 240 20 47.75% CaO-ZnO composite 240 20 67% This study

Table 2. Comparative study of various adsorbents for chloramphenicol photo-removal

In comparison, the CaO-ZnO composite showed a removal efficiency of 67% for chloramphenicol at a concentration of 20 mg/L within 240 minutes. While this is lower than the removal efficiencies of the rGO-ZnO and PbS-TiO2 composites, the CaO-ZnO composite outperformed its individual components, CaO (13.29%) and ZnO (47.75%), under identical conditions. This improvement highlights the synergistic effect of combining CaO and ZnO, where CaO contributes to adsorption and ZnO enhances photocatalytic degradation under UV light. The relatively lower removal efficiency of the CaO-ZnO composite compared to PbS-TiO2 and rGO-ZnO composites can be attributed to differences in experimental conditions, particularly the higher initial concentration of chloramphenicol used in this study. Despite these promising results, further research is necessary to optimize the performance of the CaO-ZnO composite.

CONCLUSIONS

The CaO-ZnO composite derived from coral fragments exhibited enhanced photocatalytic activity for chloramphenicol degradation, outperforming both individual CaO and ZnO in efficiency. The synergistic effect between CaO and ZnO significantly improved the degradation process, with the composite achieving up to 66.99% removal efficiency after 240 minutes of UV irradiation. The characterization of the composite confirmed successful integration of both oxides, with improved surface properties and photocatalytic activity under UV light. These results suggest that CaO-ZnO composites could serve as a promising, environmentally friendly alternative for the removal of pharmaceutical contaminants from wastewater, contributing to the sustainability of aquatic ecosystems. Future studies should explore optimization strategies and long-term stability of the composite for practical applications in large-scale wastewater treatment.

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