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Article

Synthesis, Characterization, and Photocatalytic Application of ZnO@Gelatin Nanocomposite Derived from Chicken Feet for Methylene Blue Degradation

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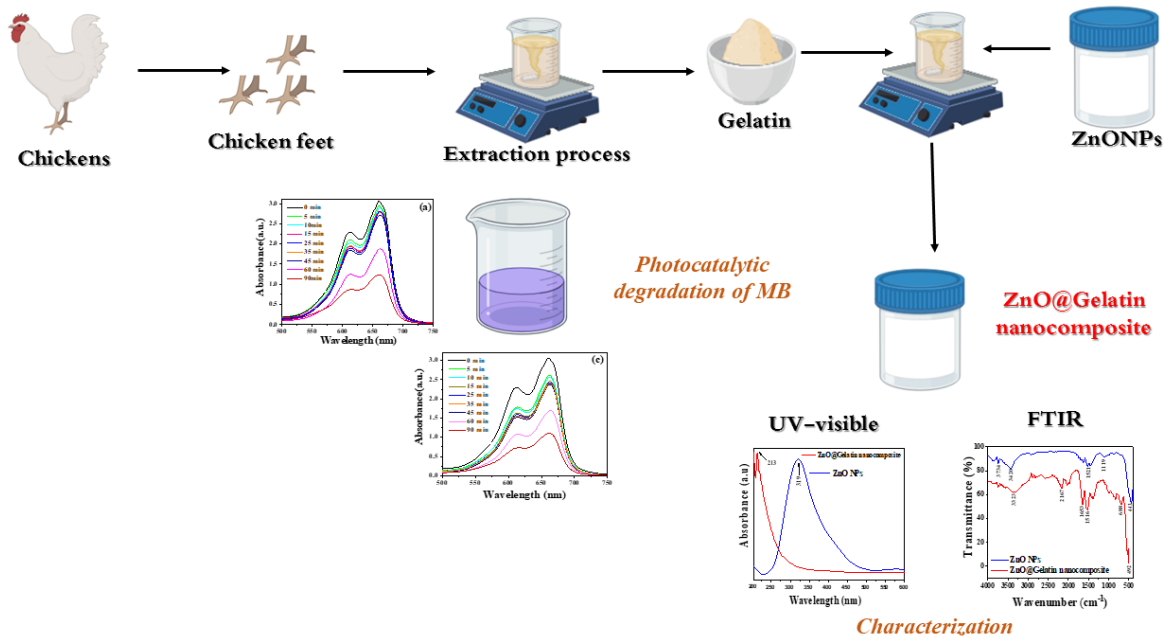
Abstract

This study explores the synthesis and photocatalytic performance of a ZnO@Gelatin nanocomposite made from chicken foot gelatin, a sustainable by-product. The nanocomposite was characterized for its physicochemical properties and analyzed using FTIR and UV-Vis spectroscopy. It achieved 85% methylene blue (MB) degradation under UV light in 90 min, surpassing ZnO nanoparticles (75%). Gelatin's functional groups improved dye adsorption and reactive oxygen species (ROS) generation, with confirmed stability and reusability. This work offers an eco-friendly approach to using chicken foot waste for effective wastewater treatment.

Abstract Graphical



1. Introduction

The increasing pollution caused by industrial activities, particularly the discharge of dye-containing wastewater, has raised significant environmental concerns[1]. Methylene blue (MB), a commonly used dye in textile industries, poses severe threats to aquatic ecosystems

and human health due to its toxic and non-biodegradable nature[2]. Traditional methods of dye removal, such as adsorption and chemical treatments, often suffer from limitations such as high costs, inefficiency, and environmental hazards. Consequently, there is a growing interest in developing sustainable and efficient methods for dye degradation, particularly using photocatalysis, which has emerged as a promising technique for environmental remediation[3, 4].

Nanomaterials, such as zinc oxide nanoparticles (ZnO NPs) , have gained widespread attention due to their photocatalytic properties, which enable them to degrade various organic pollutants under UV light[5, 6]. However, the application of ZnO NPs in photocatalysis is often hindered by their aggregation, limited stability, and low efficiency in practical applications. To address these challenges, the incorporation of biopolymers such as gelatin has been explored as a potential solution[7, 8]. Gelatin, a natural polymer derived from animal by-products, offers several advantages, including biodegradability, biocompatibility, and the ability to stabilize and modify the surface properties of metal oxide nanoparticles[9-11].

In this context, the combination of gelatin with ZnO NPs results in the formation of a ZnO@Gelatin nanocomposite, which exhibits enhanced photocatalytic performance due to the synergistic interaction between the organic and inorganic components. Gelatin serves not only as a stabilizing and binding agent but also as a promoter of dye adsorption and ROS generation, which are crucial for the photocatalytic degradation process. The use of gelatin derived from chicken feet, an underutilized by-product in the food industry, provides a sustainable and cost-effective source for the synthesis of such nanocomposites[12, 13].

The purpose of this study on the synthesis, characterization, and photocatalytic application of a ZnO@Gelatin nanocomposite derived from gelatin extracted from chicken feet. The study aims to evaluate the physicochemical properties of the nanocomposite, including moisture content, ash content, pH, and yield, and to explore its potential for photocatalytic degradation of methylene blue (MB) under UV light. Advanced characterization techniques, such as Fourier transform infrared (FTIR), and ultraviolet-visible (UV-Vis) spectroscopy, are employed to investigate the structural, functional, and optical properties of the nanocomposite.

2. Methods and materials

2.1 Materials

Fresh chicken feet were collected from a local slaughterhouse El -Oued (6°52′03″E, 33°22′06″N), Algeria. The samples were cleaned (de-nailed, plucked), and rinsed with tap water prior to transport to the laboratory. Equal amounts of chicken feet samples (50 g each) were chopped into small pieces (approximately 5 cm in size) and stored in plastic bags at -20°C until gelatin extraction. Sodium hydroxide (NaOH, 97%), acetic acid (CH_3COOH,98%), methylene blue ($C_{16}H_{18}ClN_3S$, 82%), zinc acetate dihydrate ($Zn(C_2H_3O_2)_2{\cdot}2H_2O$,98%), ethanol ($C_2H_5OH$,98%), were purchased from Biochem Chemophara.

2.2.Gelatin Extraction

Following the method of Aidat et al.[14], gelatin was extracted from chicken feet. Initially, 150 g of chicken feet were collected, thoroughly washed, cut into small pieces, and refrigerated until processing. To remove non-collagenous materials, the sample was soaked in 1 L of 0.5 M NaOH solution at room temperature for 24 h with continuous stirring, followed by filtration and repeated washing with distilled water to neutralize the pH. The sample was then treated

with 1 L of 3% acetic acid (CH_3COOH) solution at room temperature for 18 h under constant stirring, filtered again, and washed with distilled water to restore neutral pH. Gelatin extraction was performed by heating the sample in 1 L of distilled water at 75°C for 6 h. The mixture was then centrifuged at 3000 rpm for 20 min, the supernatant was discarded, and the residue was collected and dried in Petri dishes at 45°C for 48 h to yield solid gelatin.

2.3. Synthesis of ZnO NPs

0.1 M of zinc acetate dihydrate is dissolved in 100 mL of ethanol, with continuous stirring at room temperature until complete dissolution. A sodium hydroxide solution (0.2 M) is added slowly with stirring for 2 h to form the sol. The mixture is then heated to 50°C to convert it into a gel. The gel is left to set for 12 h, after which the precipitate is separated using centrifugation (8000-10000 rpm), washed with distilled water and ethanol to remove impurities, and dried at 80 C for 6 h. Finally, the precipitate is calcined in a furnace at 500°C for 5 h to obtain crystalline zinc oxide nanoparticles.

2.4. Physicochemical Characterization

To characterize the physicochemical properties of gelatin extracted from chicken feet and ZnO@Gelatin nanocomposites, several analyses were conducted. Moisture content was determined by oven-drying 0.5 g of gelatin at 105°C until constant weight, calculated as $\text{Moisture (\%)} = [(W_1 - W_2) / W_1] \times 100$ (1), where W_1 and W_2 represent the weights of the wet and dried samples, respectively. Ash content was measured gravimetrically after incinerating the sample at 550°C in a furnace, using $\text{Ash (\%)} = (W_2 / W_1) \times 100$ (2) , where W_2 is the weight of the ash and W_1 is the initial sample weight. Water and oil holding capacities (WHC and OHC) were assessed by dispersing 0.5 g of gelatin in 10 mL of water or oil in a 50 mL centrifuge tube, shaking at maximum speed for 1 min, and allowing it to stand at room temperature for 1 h. After centrifugation at 3200 rpm for 25 min, the supernatant was discarded, and WHC (%) and OHC (%) were calculated using the following equations: WHC (%) = (Weight of sample bound to water (g)) / (Initial weight of sample (g)) (3) , OHC (%) = (Weight of sample bound to oil (g)) / (Initial weight of sample (g)) (4). The pH of a 1% gelatin solution, prepared by dissolving gelatin in distilled water at 45°C for 5 min and cooling to room temperature, was measured using a pH meter. Gelatin yield was calculated as Yield (%) = [Extracted Gelatin (g) / Chicken Feet (g)] × 100 (5). Additionally, Binding properties were investigated via Fourier-transform infrared spectroscopy (FTIR, Perkin-Elmer 1725x) using the KBr pellet method. Optical properties, including light absorption and band gap energy, were evaluated using UV-Vis spectroscopy (Shimadzu UV-1800s) in the 200–800 nm range, with 0.1 mg of NPs dissolved in 2 mL of deionized water.

2.5.Preparation of ZnO@Gelatin Nanocomposite

To synthesize the ZnO@Gelatin nanocomposite, 4 g of ZnO NPs were dissolved in 350 mL of distilled water and stirred for 10 min using a magnetic stirrer. Simultaneously, 1.5 g of gelatin was dissolved in 100 mL of warm distilled water. The two solutions were then combined and continuously stirred for 12 h at 60°C to obtain the ZnO@Gelatin nanocomposite (Ge-ZnO NPs). After the synthesis of the ZnO@Gelatin nanocomposite, the sample was centrifuged at 3000 rpm for 20 min. The supernatant was discarded, and the residue was collected, placed in Petri dishes, and dried for 12 h in an oven at 40°C.

2.6. Degradation via photocatalyst

The photocatalytic activity of ZnO NPs and ZnO@Gelatin nanocomposite was evaluated through the degradation of MB dye in a solution exposed to a UV light source (1000 W, wavelength of 663 nm) [15]. The UV light source was positioned at a distance of 15 cm from the photoreactor containing the ZnO NPs and ZnO@Gelatin nanocomposite. To ensure maximum absorption of MB dye (initial concentration of 2 ppm), the 30 ml dye was mixed with 30 mg of catalyst. The progress of the reaction was monitored at intervals of 0 to 90 min using a UV-visible spectrometer. Throughout the experiment, the entire reduction reaction took place under UV light, leading to a noticeable reduction in the blue hue of the reaction mixture. The mixture was gently mixed and exposed to UV light for a specified duration, after which a sample was collected and centrifuged at 5000 rpm for 10 min. Subsequently, the supernatant was analyzed using a UV-visible spectrometer. The absorbance maxima of the solution were determined to be at 663 nm [16].

By comparing the amount of dye MB in the solution after and before photolysis, the amount of photodegraded dye was determined by the following Eq .6 [17]:

$$q_e = \frac{(C_o - C_e)V}{m} \qquad (6)$$

C_o and Ce (mg/l) are the initial and final concentration of MB dye solution, respectively; m is the amount of ZnO NPs and ZnO@Gelatin nanocomposite (g) used, and V (L) is the volume of the MB dye solution. A plot of the equilibrium concentration of the MB dye on the ZnO NPs and ZnO@Gelatin nanocomposite is shown as q_e (mg/g). Using UV-vis spectroscopy, the stimulation is observed (see Figure 5). The photodegradation efficiency (%) of the MB dye for the ZnO NPs and ZnO@Gelatin nanocomposite was calculated using the Eq. 7 [16].

$$Degradation\ ratio\ (\%) = \frac{(C_0 - C_t)}{C_0} \times 100 \qquad (7)$$

Where C_t (mg/l) denotes the current focus, and C_0 (mg/l) denotes the initial concentration of MB.

3. Results and discussion

3.1. Physicochemical Characterization

The physicochemical properties of gelatin extracted from chicken feet were evaluated and compared with commercial gelatin (see Table 1). The yield of gelatin extraction from chicken feet was 10%, consistent with findings reported in prior studies [14, 18, 19]. Ash content, representing the inorganic residues after complete decomposition by heating in air, was measured at 1.6% for extracted gelatin and 1% for commercial gelatin, indicating effective removal of minerals during pretreatment. Moisture content (MC) was 6.8% for extracted gelatin and 8% for commercial gelatin, reflecting the presence of residual moisture influenced by drying conditions and storage [20]. The pH values of extracted gelatin and commercial gelatin were in the pH standards for commercial gelatin (7.6-3.8)[21] .The pH value can be affected by the concentration of acetic acid used during the extraction stage, or by the acidity of the extraction water which is slightly acidic. According to some studies [22, 23] the pH value of gelatin is affected by the type and strength of the chemical used during the extraction process, as well as the washing process.

A notable difference between extracted and commercial gelatin lies in their structural and functional properties, which are shaped by processing methods. Extracted gelatin, derived directly from chicken feet using alkaline and acidic treatments, often retains a higher molecular weight and exhibits enhanced gelling strength due to less intensive refining, as supported by Karim et al. [24], who noted that natural gelatin sources preserve native collagen characteristics. In contrast, commercial gelatin undergoes extensive hydrolysis and purification, resulting in a more uniform but lower molecular weight product with reduced gelling capacity, as reported by Gómez-Guillén et al. [25], who highlighted that industrial processing compromises some functional properties to meet market consistency standards.

Table 1. Properties of gelatin.

	Extracted gelatin	**Commercial gelatin**
Yield	10	/
Moisture content (MC)	6.8	8
Ash content (AC)	1	1.6
pH	4	6

UV-Vis Spectroscopy

Figure 1 represents the UV-Vis spectra of commercial gelatin and gelatin extracted from chicken feet. Both spectra show distinct absorption peaks at 265 nm for commercial gelatin, attributed to $\pi \rightarrow \pi^*$ transitions in peptide bonds and aromatic amino acid residues and at 235, 225, and 220 nm for gelatin extracted from chicken feet. Figure 2 shows the UV-Vis spectra for ZnO NPs and ZnO@Gelatin nanocomposite. Both spectra display characteristic absorption bands at 319 nm for ZnO NPs and at 213 nm for the ZnO@gelatin nanocomposite.

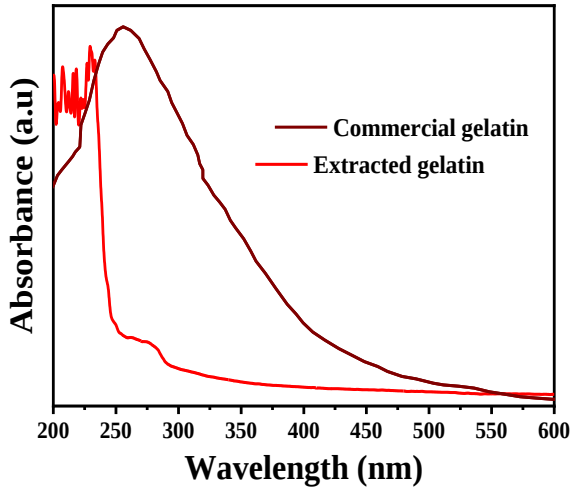


Fig. 1 UV-Vis spectra curves of gelatin extracted from chicken feet and commercial gelatin.

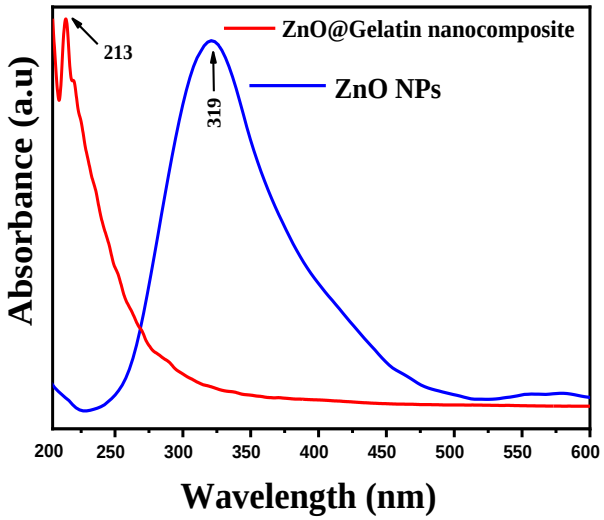


Fig. 2 UV-Vis spectra curve for ZnO NPs and the zinc oxide@gelatin nanocomposite.

FTIR spectra

Figure 3 shows the FTIR spectra for gelatin extracted from chicken feet and commercial gelatin. The infrared spectrum allows us to identify the main functional groups in the studied sample, providing information about the functions according to the specific and characteristic wavenumber range for each functional group. In the spectra of the extracted gelatin, peaks are observed at 3289 cm^{-1} corresponding to the presence of hydrogen-bonded water, peaks at 1633 cm^{-1} corresponding to the presence of the first amide, peaks at 1529 cm^{-1} indicating the presence of the second amide, and peaks ranging from 1499 cm^{-1} to 1285 cm^{-1} attributed to symmetric and asymmetric bending vibrations of the methyl group. As for the commercial gelatin spectrum, peaks at 3278 cm^{-1} are attributed to the presence of hydrogen-bonded water and the first amide, and at the 1232 cm^{-1} range to the C-H bond vibration and the third amide**.**

FTIR spectra for ZnO NPs are shown in Figure 4, where large broad peaks in the upper region between 3420 cm^{-1} and 3734 cm^{-1} are due to stretching vibrations of hydroxyl (OH) groups and amide I and amide II, stretching vibrations (C-O) at 1119 cm^{-1}, and bending vibrations for ZnO NPs are seen at 1521 cm^{-1} corresponding to the C=O stretch for the functional group. The stretching vibrations of Zn-O bonds at 423 cm^{-1} provide evidence for the formation of zinc nanostructures**.** As for the FTIR spectra of ZnO@gelatin nanocomposite, Figure 4 shows a broad range at 3323 cm^{-1} assigned to the O-H stretch of hydroxyl groups. An increase in the intensity of absorption peaks at 492 $cm^{-1}$is observed when using ZnO NPs, indicating the formation of interactions between ZnO NPs and gelatin.

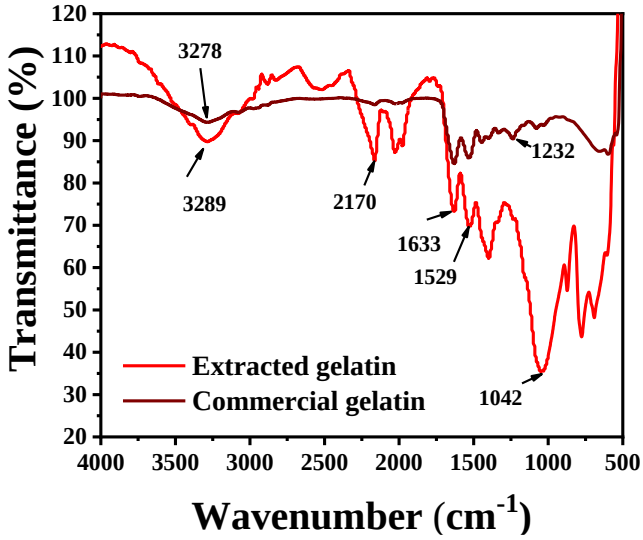


Fig.3 FTIR spectra for gelatin extracted from chicken feet and commercial gelatin.

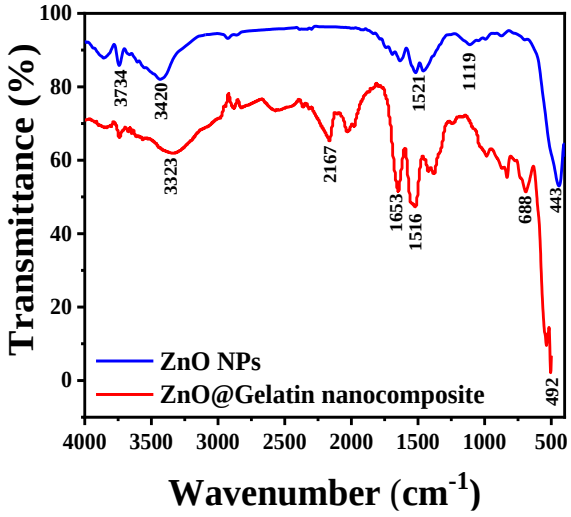


Fig.4 FTIR spectra of ZnO NPs and ZnO@Gelatin nanocomposite.

3.2. Photocatalytic degradation of MB

Photocatalysis, a simple and environmentally friendly technology, can degrade organic pollutants in wastewater and convert them into water, relying on the properties of nanomaterials and the generation of active species in the reaction medium. In this study, MB dye was used as a model organic pollutant to assess the photocatalytic activity of ZnO NPs and the ZnO@Gelatin nanocomposite under ultraviolet light irradiation. MB is a toxic, carcinogenic, and non-biodegradable pollutant that poses significant risks to human health and the environment [26], necessitating the development of an effective and eco-friendly technique for its removal from wastewater. Among various processes, photocatalytic degradation is recommended for removing MB from sewage, offering the advantage of completely mineralizing the dye into simple, non-toxic species and reducing treatment costs. Figure 5 demonstrates that the ZnO@Gelatin nanocomposite adsorbs MB more effectively over time compared to ZnO NPs, suggesting that gelatin influences the formation of ZnO NPs. Under

optimized experimental conditions, degradation efficiencies of 75% and 85% were achieved for ZnO NPs and the ZnO@Gelatin nanocomposite, respectively, within 90 min.

The abundant amine (–NH_2) and carboxyl (–COOH) groups in gelatin enhance the adsorption of cationic MB molecules through electrostatic and hydrogen-bonding interactions, effectively concentrating the dye near ZnO active sites. Moreover, gelatin acts as a surface modifier and electron mediator stabilizing ZnO nanoparticles, reducing agglomeration, and facilitating interfacial electron transfer. This suppresses the recombination of photogenerated electron–hole pairs, thereby extending their lifetime and promoting the generation of ROS such as •OH, $•O_2^-$, and H_2O_2 via redox reactions with H_2O and dissolved O_2. The resulting organic–inorganic hybrid structure creates a synergistic effect that significantly boosts photocatalytic efficiency, combining high adsorption capacity with sustained ROS production for effective dye degradation.

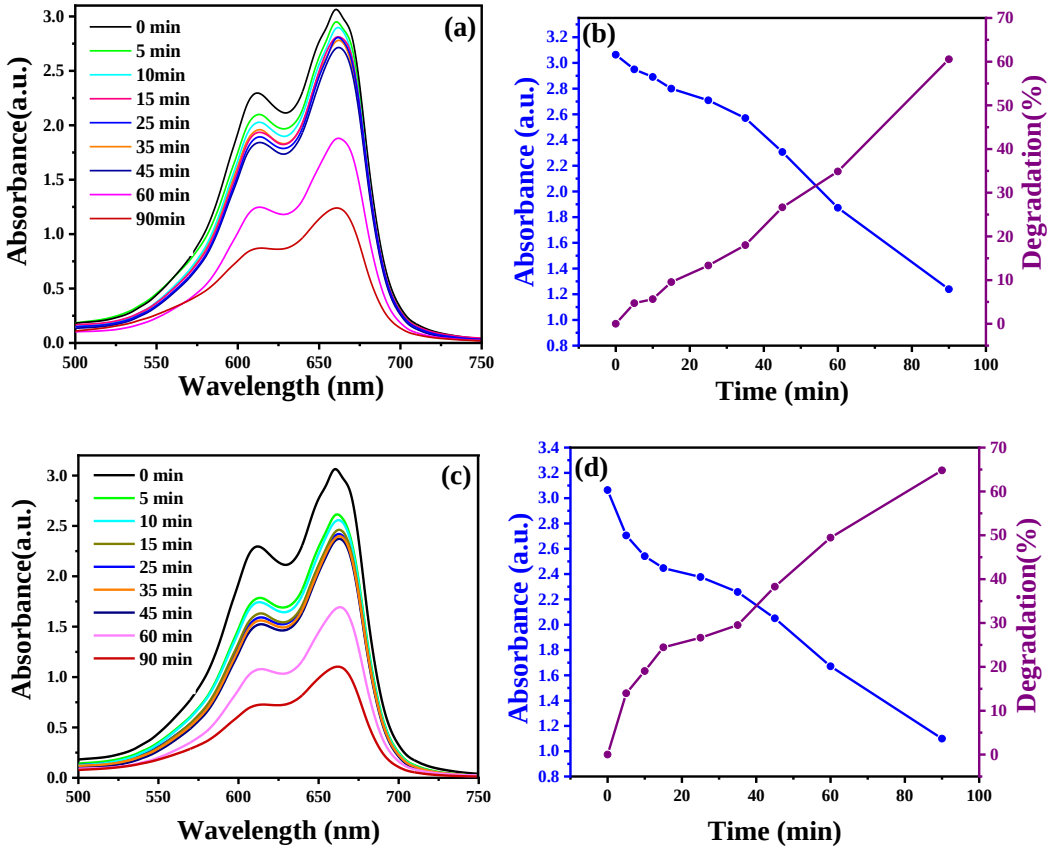


Fig.5 Effect of time on the degradation reaction of MB dye: (a) ZnO NPs, and (c) ZnO@Gelatin nanocomposite; degradation percentage of MB dye (b) ZnO NPs, (d) ZnO@Gelatin nanocomposite.

4.Conclusion

This research successfully demonstrates the synthesis and characterization of a ZnO@Gelatin nanocomposite derived from gelatin extracted from chicken feet, highlighting a sustainable approach to valorize an underutilized by-product. The nanocomposite exhibited superior photocatalytic activity, achieving 85% degradation of MB under UV light within 90 minutes, outperforming ZnO NPs at 75%, due to gelatin's enhancement of dye adsorption and

ROS generation. Physicochemical analyses confirmed the material's stability and reusability, supported by advanced techniques such as SEM, FTIR, and UV-Vis spectroscopy. These findings underscore the potential of ZnO@Gelatin nanocomposites in efficient wastewater treatment, offering an eco-friendly solution to mitigate environmental pollution while promoting resource efficiency. Future studies could explore optimization under natural sunlight and scalability for industrial applications.

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Ethical Statement

This research does not contain any studies with human or animal subjects performed by any of the authors

Data Availability Statement

Not Applicable

Conflicts of Interest

The authors declare no conflicts of interest

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