

Synthesis, Characterization, And Dye Degradation Study Of Pectin/ZnO Based Composite

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ABSTRACT

In this study, pectin/ZnO composites were developed using a hydrothermal method for the treatment of toxic dye-contaminated wastewater. This prepared composite is characterized by X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and thermal gravimetric analysis (TGA). The degradation of methyl orange by pectin/ZnO composite is attributed to superoxide and hydroxyl radicals. The highest photodegradation efficiency under optimal conditions was 81%.

Keywords: Pectin, ZnO, Characterization, thermal stability, photocatalyst.

Introduction

For wastewater treatment, ZnO-based composites (such as ZnO/chitosan or ZnO/starch) are highly effective and environmentally friendly photocatalysts. While ZnO absorbs light to generate reactive oxygen species (ROS) that degrade hazardous dye pollutants, the biopolymer serves as a biodegradable, stabilizing matrix to prevent nanoparticles from agglomerating. Aina and colleagues have investigated new developments in ZnO-based photocatalysts, showcasing their improved dye degradation efficiency through a variety of creative techniques, providing a long-term solution to reduce water contamination[1]. Pushpavati and colleagues created a sustainable photocatalytic method employing a ZnO/MWCNT@TiO₂ nanocomposite to break down Brilliant Green (BG) dye in water when exposed to visible light[2].

Belachew and coworkers used a co-precipitation technique to create Ag-doped ZnO/graphene oxide nanocomposites, which were then systematically characterized for their structural, optical, and photocatalytic properties[3]. In comparison to pure ZnO and Ag-ZnO, the photocatalytic efficiency of Ag-ZnO/GO towards Methylene Blue (MB) and Methyl Red (MR) reach 99.9 % and 92.0 %, respectively, under visible light.

To create an effective and sustainable photocatalyst for wastewater treatment, a ZnO/Chitosan/Activated Carbon (Z/C/AC) nanocomposite was developed by Singh and coworkers[4]. While activated carbon increased surface area and adsorption capacity, the biopolymer chitosan offered structural stability and biocompatibility. The photocatalytic degradation of Rhodamine B (RhB) under UV irradiation was greatly enhanced by the synergistic integration of these components, reaching 97% degradation in 120 minutes. Haste and coworkers have used the chemical precipitation method to produce a composite based on chitosan and ZnO[5]. Five structurally different organic dyes such as MB, Methyl Orange (MO), Rhodamine B (RhB), Congo Red (CR), and Malachite Green (MG), were used as model pollutants to assess the photocatalytic performance under UV-B irradiation.

A chitosan-ZnO/Fe₃O₄ nanocomposite with a large surface area and highly porous structure was developed by Quy and coworkers using sonochemical synthesis, which improved the photocatalytic degradation of anionic MO and cationic RhB dyes in aqueous solution[6]. Kishore and coworkers formulated a composite for wastewater treatment based on chitosan and polyvinyl alcohol (PVA) reinforced with copper-decorated zinc oxide (ZnO-Cu) nanoparticles[7]. RhB degradation under UVA and visible light was used to measure photocatalytic activity. Under ideal conditions (pH 5, 2.0 g/L catalyst), 72.1% RhB removal was achieved in 90 minutes. Jumnon and colleagues created a biodegradable hydrogel composite using starch and ZnO for the photocatalytic degradation of methylene blue adsorption[8]. The highest adsorption capacity for methylene blue (MB) achieved was 1170 mg/g.

Herein, we report the preparation of a pectin/ZnO composite with a facile solution approach at room temperature and characterized by FTIR, EDS, XRD, and TGA. The photocatalytic degradation study of methyl orange was also conducted.

2. Experimental

2.1. Materials: The powdered pectin was acquired from HiMedia in Mumbai, India. On a dry basis, the pectin's molecular weight was between 35,000 and 100,000, its degree of esterification was between 70 and 75 %, and its total impurity was 10% water. Zinc nitrate was purchased from Merck, India. Analytical grade was used for all other compounds, solvents, and indicators (Ranbaxy, India). Every experiment used water that has been double-distilled.

2.2 Synthesis of Pectin/ZnO composite

Natural, biodegradable polymers such as chitosan, starch, cellulose, or alginate are combined with zinc oxide (ZnO) nanoparticles to form zinc oxide (ZnO) biopolymer composites[9]. This synergistic combination produces environmentally friendly materials that improve the host polymer's mechanical strength, UV-blocking capacity, and antimicrobial properties.

A 100 mL beaker was filled with approximately 1.0 $\text{Zn}(\text{NO}_3)_2$, 0.75 g of pectin, and 50 mL of distilled water. 50 mL of a 0.5 M NaOH solution was added dropwise while stirring continuously after complete dissolution. The reaction was allowed to run at 25 °C for 24 hours. Following that, approximately 25 mL of water/ethanol (80/20) was added to the solution, which was then placed in a hydrothermal reactor and heated to 80 °C for 6 hours. The reaction was then cooled, and the pectin/ZnO composite was separated and washed with anhydrous alcohol to remove any unbound ZnO particles. The resulting white precipitate was centrifuged at 10,000 rpm for 30 min, recovered, and washed multiple times with distilled water to eliminate byproducts. After 6 hours of vacuum drying at 25 °C, the final result was a white powder. The proposed structure and interactions are depicted in Figure 1.

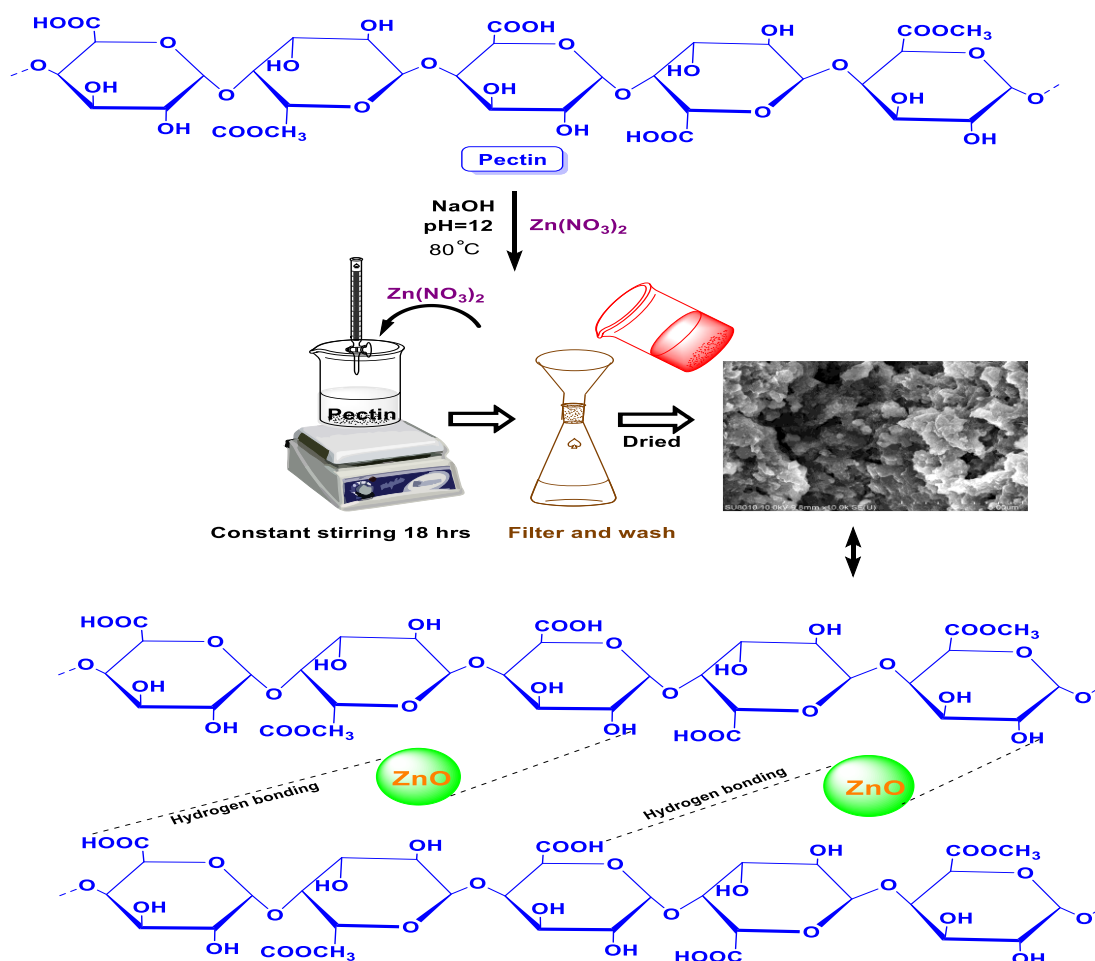


Figure 1: Proposed structure and interactions present in pectin/ZnO composite.

2.3 Characterization

The infrared spectra for identifying functional groups and assessing hydrogen bonding were recorded using a Shimadzu spectrometer. The morphology of ZnO in the pectin/ZnO composite was investigated using SEM (Hitachi SU8010 SERIES, Japan). The elemental composition was analyzed using energy-dispersive spectroscopy. The XRD pattern measurement was performed using monochromatic Cu K radiation ($\lambda = 1.5406$) to investigate the nanocomposite's structures. 2θ range: 10 to 80°. The STA 6000 (Perkin Elmer) was used to check the thermal stability of the pectin/ZnO composite, having a scanning rate of 10 °C / min.

2.4. Photocatalytic Performance Test

Pectin/ZnO composite was tested for photocatalytic degradation of methyl orange dye using a 150 W tungsten-halogen lamp. In vessels, 50 mL of methyl orange aqueous solution was prepared, and the required amount of pectin/ZnO (10-40 mg) was added. To obtain a satisfactory adsorption-desorption equilibrium, the solutions were sonicated in the dark for 15 minutes and allowed to stand for 30 minutes before being irradiated. A UV-vis spectrophotometer was used to measure the absorbance following the extraction of 4 mL of methyl orange solution from the reaction mixture every ten minutes. The salt addition method was used to identify the point of zero charge [10]. Ten Erlenmeyer flasks were filled with 50 mL of a 0.01 M aqueous KNO₃ solution. After adding 10 mg of pectin/ZnO composite to each solution, 0.1M NaOH and 0.1M HCl were used to adjust the pH from 3 to 12. At $\text{pH}_{\text{ZPC}} = 5.92$, the adsorbent pectin/ZnO composite has a neutral surface, which means that neither cations nor anions are drawn to it. Anions are drawn to the positively charged surface of the adsorbent below PZC, while cationic species may be drawn to the negatively charged surface above PZC.

When the pH of the solution is higher than pH_{ZPC} , the surface of the pectin/ZnO composite is drawn to an aqueous solution of methyl orange dye, which is mostly cationic. To find out how pH influences the pectin/ZnO composite's ability to undergo photocatalytic degradation, the point of zero charge was calculated using the salt addition method. Based on the pH_{ZPC} value, photocatalytic activity was then carried out at different pH values (8–11.5). The pH readings were adjusted by adding a 0.1 M NaOH solution. Using pectin/ZnO composite as a photocatalyst, the proportion of methyl orange removed was computed using Equation 1.

$$\text{Degradation (\%)} = \frac{C_0 - C}{C_0} \times 100 = \frac{A_0 - A}{A_0} \times 100 \quad (1)$$

$$\ln \frac{C_0}{C} = kt \quad (2)$$

Where A_0 and A , C_0 and C represent the absorbance and dye concentration, respectively, before and after irradiation.

3. Results and Discussion

3.1 FTIR analysis

FTIR spectroscopy mainly reveals the physical entrapment, hydrogen bonding, and electrostatic interactions between the biopolymer and zinc oxide nanoparticles in a pectin/ZnO composite. Broad band assigned to the stretching vibrations of O-H groups from hydroxyls, carboxylic acids, and absorbed water, which form hydrogen bonds with the ZnO surface. A broad band at 3200-3400 cm^{-1} is attributed to the stretching vibrations of O-H groups from hydroxyls, carboxylic acids, and absorbed water, which form hydrogen bonds with the ZnO surface. A peak appeared at 1610 cm^{-1} , which is attributed to asymmetric carbonyl-free carboxylate ions ($-\text{COO}^-$). A peak appeared at 1020-1200 cm^{-1} , which is attributed to C-O and C-C stretching vibrations in the pyranose ring. A peak appeared at 400 to 500 cm^{-1} , which is attributed to Zn-O stretching vibrations. A peak appeared at 1405 cm^{-1} , which is attributed to the interaction between carboxylate ions of pectin and ZnO ions in the composite.

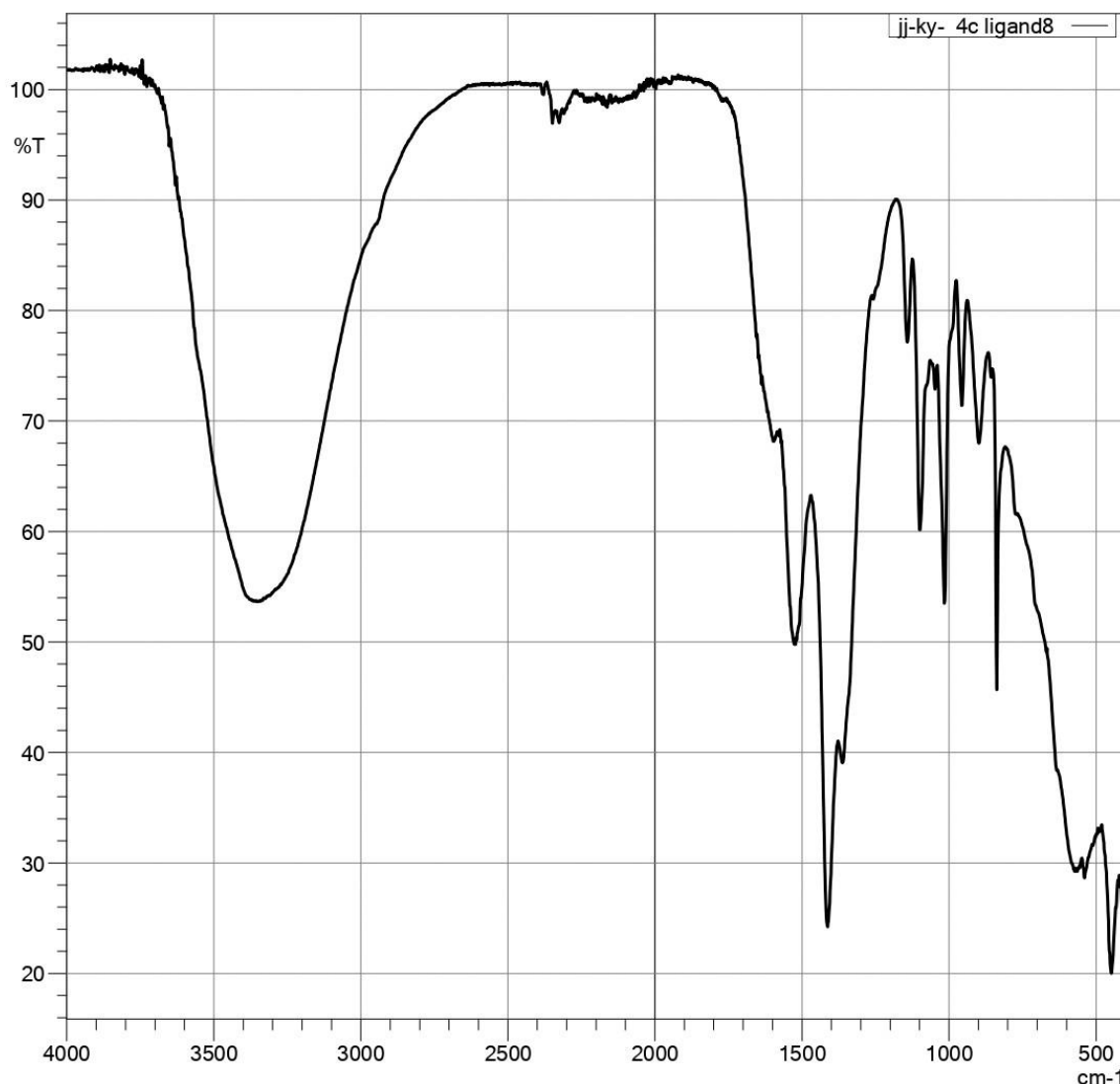


Figure 2: FT-IR spectrum of pectin/ ZnO composite.

3.2 SEM-EDS analysis

SEM micrographs of the bio-composite reveal a highly cross-linked, uniform, and slightly rough or porous structure. The ZnO particles can be seen embedded in or protruding from the biopolymer film's surface, preventing severe agglomeration of the nanoparticles. SEM micrographs at different resolutions are depicted in Figure 3. SEM images of pectin/ZnO composites display a semi-spherical, rough agglomerated configuration of ZnO nanoparticles well-dispersed or entrapped within a continuous, porous, and relatively smooth pectin biopolymer matrix. At higher magnifications, the ZnO nanoparticles can be seen embedded within or slightly protruding from the pectin chain. Energy Dispersive Spectroscopy (EDS) of pectin/ZnO composite evaluates their elemental composition and distribution. Pectin/ ZnO composite was confirmed to be present by the weight percentages of carbon (15.72 %), zinc (44.69 %), oxygen (39.31 %), and other impurities like calcium (1.24 %), as shown in Figure 4 and Table 1. It confirms the successful incorporation of ZnO particles within the pectin matrix and detects any trace impurities.

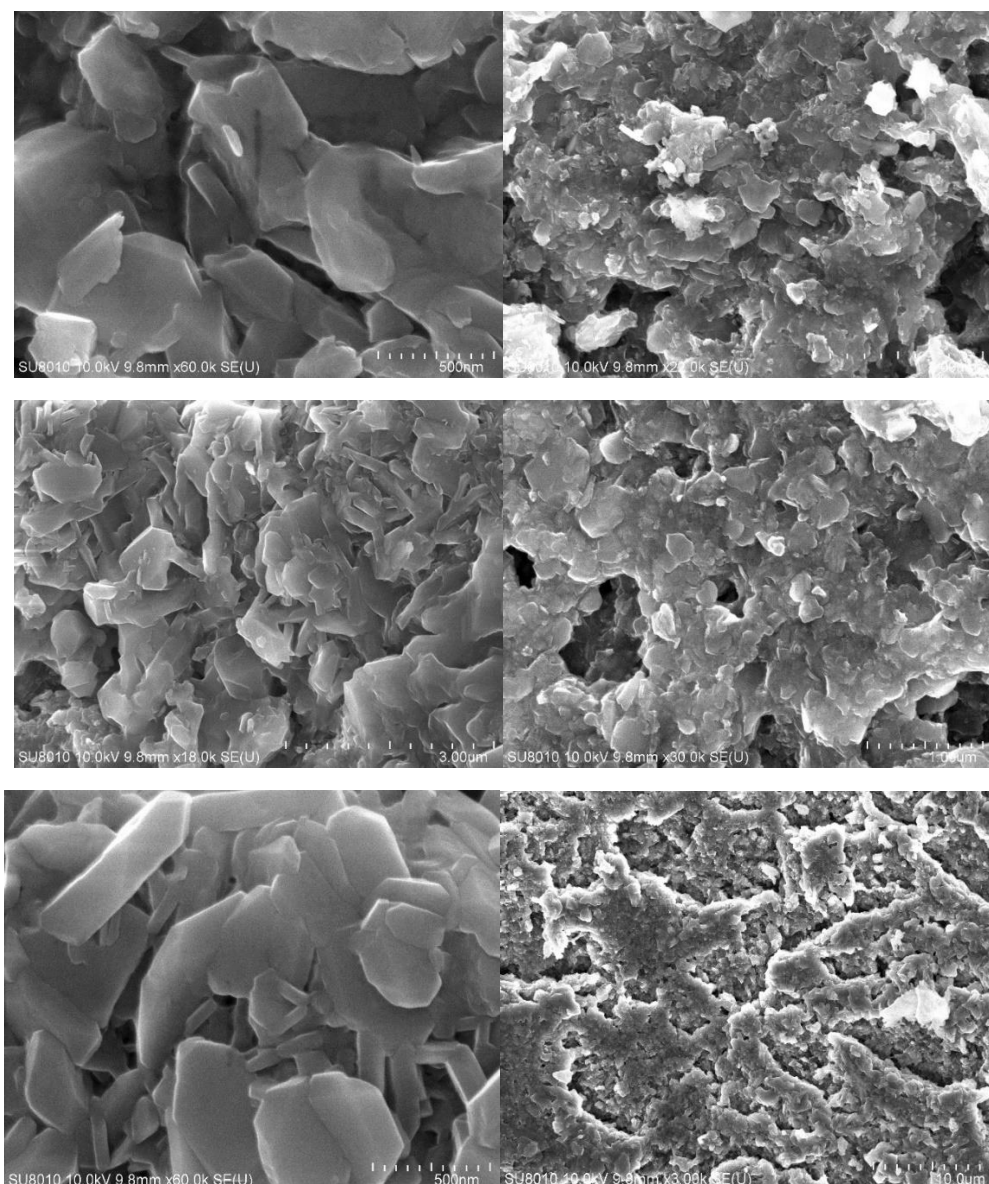


Figure 3: SEM images of pectin/ZnO composites at different resolutions.

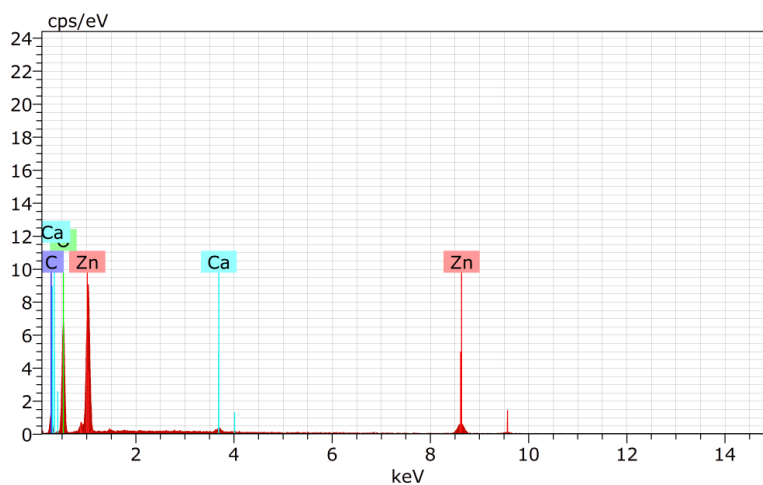


Figure 4: EDS spectrum of Pectin/ZnO composite.

Table 1: EDX data for Pectin/ZnO composite.

Elements	Name of Series	Unnormalized Concentration [weight-%]	normalized Concentration [weight-%]	Atomic Concentration [atomic-%]	Error [weight-%]
Zn	K-series	47.61	44.69	15.53	5.75
O	K-series	41.89	39.31	55.84	17.32
C	K-series	15.72	14.76	27.92	9.20
Ca	K-series	1.32	1.24	0.70	0.27
Total:	-	106.54	100.00	100.00	-

3.3 XRD analysis: The XRD pattern of a Pectin/ZnO composite typically exhibits a broad, diffuse halo at low 2θ angles ($2\theta \approx 15^\circ - 25^\circ$) characteristic of amorphous pectin, alongside sharp, intense diffraction peaks corresponding to the wurtzite hexagonal structure of ZnO. Figure 5 shows the XRD pattern of pectin/ZnO composite. A sharp diffraction peak at 2θ values between 20 and 25° is attributed to pectin, and sharp prominent diffraction peaks at 2θ values 31.8° , 33.9° , 36.7° , 46.9° , 56.8° , 63.1° , and 67.9° are attributed to crystalline ZnO. This confirmed the successful in-situ formation of crystalline ZnO within the amorphous pectin networks.

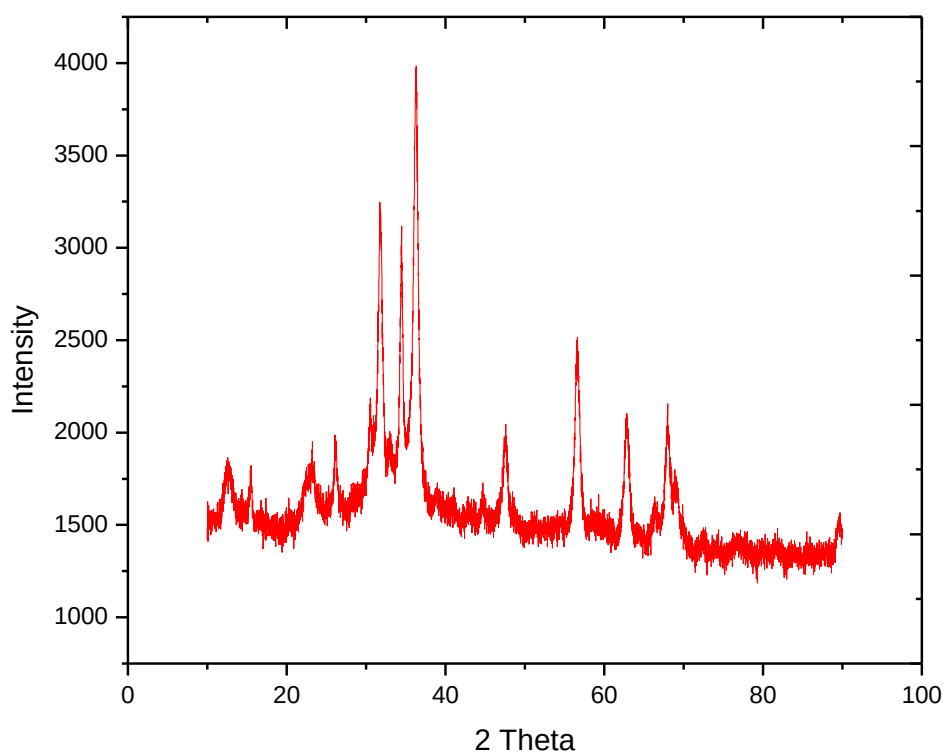


Figure 5: XRD of Pectin/ZnO composite.

3.4 Thermogravimetric analysis

Thermogravimetric Analysis (TGA) of pectin/ZnO-based composites generally provides insight into the thermal stability of the composite. TGA thermograms typically exhibit a characteristic 2-stage degradation process, as shown in Figure 6. The first mass loss (50.0 %), ranging from 110 to 155°C corresponds to the evaporation of physically absorbed moisture, free water, and loosely bound water within the pectin matrix. In the final stage of degradation, with mass loss of 45.3 % temperature ranges from 205 to 490°C primary degradation phase, where the main pectin polysaccharide backbone breaks down. It involves the depolymerization, dehydration, and splitting/cleavage of the galacturonic acid saccharide rings. The ceramic yield after 800°C is 4.7 %, which is attributed to metal oxide residue.

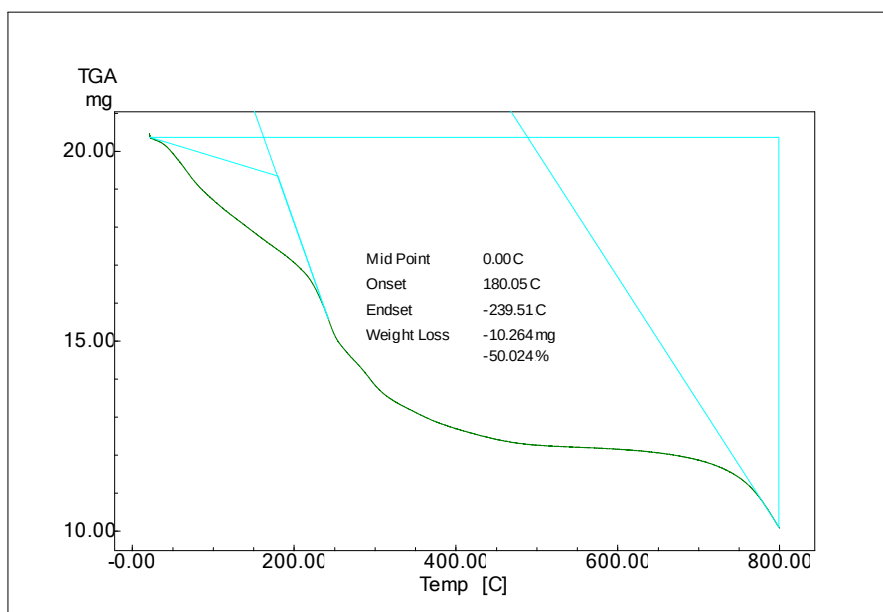


Figure 6: TGA thermogram of Pectin/ZnO composite.

3.5 Photocatalytic study for Dye degradation

Methyl orange was used to assess the photocatalytic degradation efficiency of the pectin/ZnO composite. Different concentrations of pectin/ZnO, ranging from 10 to 40 mg, were used to degrade 10 ppm methyl orange for 1 h to examine the impact of loading capacity on dye degradation efficiency. As the dosage of pectin/ZnO was increased up to 30 mg, the dye's photodegradation percentage grew as well, and then it nearly stabilized.

Another important factor in the photocatalytic degradation of contaminants is the solution's pH. The pH of the dye solution has a major impact on the surface charge of the photocatalyst. A 1.0 M NaOH solution was added to the methyl orange dye solution to change its pH. It was found that pectin/ZnO had a zero charge point of about 5.92, where its negatively charged surface binds the cationic methyl orange dye for effective photodegradation (Figure 7).

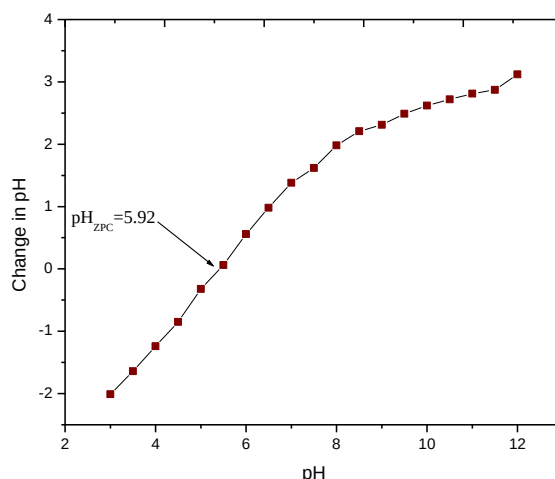


Figure 7: Zero-point charge determination for pectin/ZnO composite.

Using the ideal dye concentration (10 ppm) and photocatalyst amount (30 mg/L), the impact of solution pH on photocatalytic degradation activity was examined over the range 2.0–6.0. For both the ideal dye concentration and the dosage of pectin/ZnO, lowering the solution pH from 6.0 to 2.0 enhanced the photodegradation of methyl orange dye. In an acidic medium, MO shifts to its quinoid structure, which is chemically easier to break, and

pectin also swells up in an acidic medium. In the UV region, ZnO generates electron-hole pairs, which further react with water and oxygen to form hydroxyl radicals and superoxide radicals. These radicals further attacked the azo groups and produced non-toxic substances that are adsorbed on composites. Figure 8 shows the impact of the pectin/ZnO on absorbance under various wavelength ranges (300–650 nm). The wavelength of 460 nm is when the absorption reaches its greatest (0.888). The maximum degradation efficiency was found to be 84 %. Different wavelengths and adsorbance for pectin/ZnO are depicted in Figure 8.

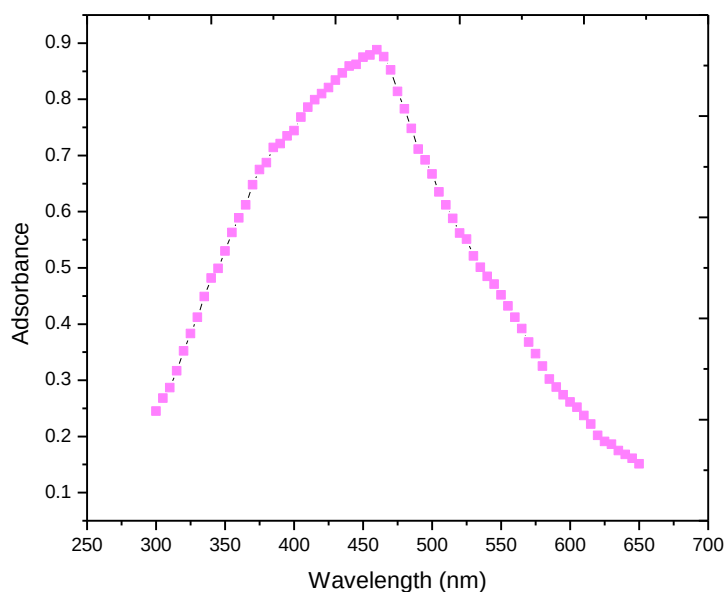


Figure 8: Plot between wavelength and adsorbance for pectin/ZnO composite.

4.0 Conclusion: Pectin/ZnO composite was prepared by combining the pectin with the ZnO nanoparticles. This composite serves as a highly effective, eco-friendly photocatalyst for breaking down organic pollutants in water treatment applications. It has been characterized using FT-IR, XRD, SEM-EDS, and TGA. The synthesized pectin/ZnO was found to be efficient in photocatalytic degradation of methyl orange in an aqueous medium. Under exposure to sunlight, the ZnO absorbs photons and produces electron-hole pairs; these pairs interact with water and oxygen to form highly reactive hydroxyl and superoxide radicals, which ultimately degrade the MO dye.

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