

ZnO/CNT Nanocomposite: Synthesis, Characterization and Application in Methylene Blue Dye Removal

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Abstract

ZnO/CNT nanocomposites were synthesized via a ball-milling approach to explore their potential for the photocatalytic removal of Methylene Blue dye, a common organic pollutant found in wastewater from textile industries. The synthesized nanocomposites were characterized for their structural, chemical, optical, and thermal properties. XRD diffraction analysis showed a lattice strain of 0.00534 and a crystalline size of about 21 nm. XPS results confirm the elemental composition and chemical states of the components, which were further supported by FTIR results. UV-Visible spectroscopy showed a band gap of 2.25 eV and an absorption peak at 369 nm. TGA results indicate distinct thermal transitions and provide insights into the material's thermal stability. The ZnO/CNT composite demonstrated effective dye removal and achieved a significant reduction of MB concentration within 80.5 hours under UV irradiation, while only 1.5 hours under sunlight, which highlights the potential of this nanocomposite as a robust photocatalyst for wastewater treatment applications.

Keywords

Ball-Milling, Dye Removal, Photocatalytic, CNT, Nanocomposites, ZnO

1. Introduction

The widespread use of synthetic dyes, especially in textile, printing, and dyeing industries, has led to severe environmental concerns due to the discharge of toxic and non-biodegradable pollutants into water bodies [1]. Methylene Blue (MB), a common organic dye, is frequently detected in industrial wastewater and poses

serious ecological and health risks even at low concentrations [2] [3]. Its persistence in aquatic systems not only threatens aquatic life but also reduces the availability of potable water, especially in regions already facing water scarcity [4]-[6].

Traditional water treatment techniques frequently fail to adequately eliminate such persistent contaminants. In this context, semiconductor-based photocatalyst is presents a sustainable and efficient method for wastewater treatment [7]. A particularly effective approach within this domain is the use of nanocomposites, which leverage the synergistic interactions between their nanoscale components [8]. These materials exhibit improved physicochemical properties, including an enhanced surface-to-volume ratio, leading to significantly boosted photocatalytic performance [9]. Zinc oxide (ZnO), one of the many photocatalysts, has attracted interest because of its potent oxidative power, affordability, and durability. ZnO exhibits high surface reactivity [10] and has been demonstrated to be a more efficient photocatalyst than TiO₂. However, its photocatalytic efficiency is often limited by the rapid recombination of photo-generated charge carriers. To overcome these limitations, ZnO can be coupled with carbon nanotubes (CNTs), particularly multi-walled carbon nanotubes (MWCNTs), which offer exceptional electrical conductivity, high surface area, and strong adsorption capacity. The resulting ZnO/CNT nanocomposite exhibits enhanced photocatalytic activity due to improved charge separation and extended light absorption range, making it highly suitable for environmental applications such as dye removal from wastewater [11].

A ball-milling technique was used to create ZnO/CNT nanocomposites in this study to ensure uniform dispersion and efficient interfacial contact between the two phases. In order to illustrate the potential of the synthesized nanocomposites as effective and environmentally friendly materials for wastewater remediation, their photocatalytic performance was then assessed for the removal of MB dye under both UV and natural sunlight irradiation.

2. Experimental Section

2.1. Synthesis of ZnO/CNT Nanocomposites

ZnO/CNT nanocomposites were synthesized by incorporating zinc oxide (ZnO) nanoparticles with multi-walled carbon nanotubes (MWCNT), which have 3 - 15 walls and lengths ranging from 1 - 10 μm , procured from M/s. Alfa Aser. Green synthesis was used to synthesize ZnO nanoparticles [12]. Initially, 0.032 g of MWCNTs was dispersed in 50 mL of ethanol using ultrasonic vibrations in a sonicator for approximately 1 hour to ensure uniform dispersion. ZnO nanoparticles weighing 0.968 g were introduced to the mixture following sonication. The solution was heated to 80 °C and then stirred with a magnetic stirrer until the solvent evaporated completely, leaving behind a dry powder. The collected powder was subsequently transferred to a Planetary Ball Milling Machine (Retsch PM 100) and subjected to ball milling for 1 hour to synthesize the ZnO/CNT nanocomposites. A ball-to-powder ratio (BPR) of 20:1 was maintained, and the milling was carried

out at 300 rpm. To improve milling efficiency, 1 - 2 drops of acetone were added during the loading process. Before material loading, the ball mill was thoroughly cleaned to avoid contamination. Following an hour of milling, the ZnO/CNT nanocomposite material was extracted for additional analysis and future use.

2.2. Characterization of ZnO/CNT Nanocomposites

Various characterization techniques were employed to confirm the structural, chemical, and optical properties of the synthesized ZnO/CNT nanocomposite. The crystalline structure of the material was ascertained by XRD analysis using a *CuK α* source, and the XRD spectra were used to calculate parameters like strain and crystallite size.

XPS was conducted using a Thermo Scientific Nexsa G2 system to analyze the elemental composition and chemical states present in the material. UV-Vis Spectroscopy was carried out using an Agilent Cary 60 Spectrophotometer to investigate the optical absorption characteristics of the nanocomposite. FTIR measurements were obtained using the FTIR Spectrum 2 (Perkin Elmer) machine, covering the spectral range of 4100-400 cm^{-1} , to determine the functional groups present in the sample. Additionally, TGA was performed using an STA 7300 HITACHI instrument to examine the thermal stability and decomposition behavior of the material. These characterization techniques collectively provide comprehensive insights into the structural and functional properties of the ZnO/CNT nanocomposite.

2.3. Photocatalytic Dye Removal Experiment

The photocatalytic dye removal of Methylene Blue (MB) was investigated using ZnO/CNT nanocomposites as the photocatalyst. In this experiment, 2 mg of MB dye was mixed with 50 mg of ZnO/CNT photocatalyst and diluted to a total volume of 100 mL using deionized (DI) water. The prepared solution was kept in the dark for 60 minutes to establish adsorption-desorption equilibrium. The initial absorbance was then recorded, with the maximum absorption peak of MB observed at 662 nm. The dye removal experiment was conducted under UV light irradiation for a total duration of 80.5 hours and under sunlight for 1.5 hours. Changes in the absorption spectra were monitored at regular intervals to assess the photocatalytic efficiency of the ZnO/CNT nanocomposite in the removal of MB dye from the solution.

3. Results and Discussion

3.1. Microstructural Analysis of ZnO/CNT Nanocomposites

To determine the structural properties of the ZnO/CNT nanocomposite, Rietveld refinement was performed. The structural evaluation was carried out using the VESTA software, providing detailed insights into the crystal structure. The refinement results for the ZnO/CNT nanocomposite sample include: Chi-Square (χ^2): 1.49, Bragg R-Factor: 25.79, Rf-Factor: 13.93. **Figure 1** displays the X-ray diffraction

(XRD) spectrum of ZnO/CNT nanocomposites. The presence of distinct low intensity peaks in the XRD spectrum suggests that the nanocomposite is nano-crystalline.

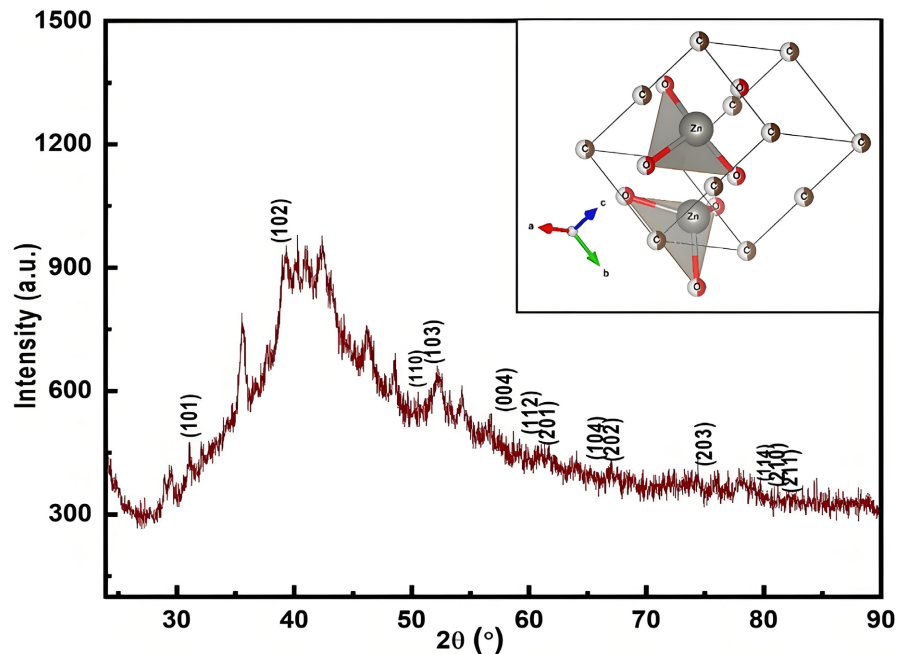


Figure 1. X-Ray diffraction pattern of ZnO/CNT nanocomposites and Vesta structure of ZnO/CNT Nanocomposite is shown in the inset.

However, some minor peaks corresponding to specific crystal planes are observed at different diffraction angles, which are indexed as (101), (102), (110), (103), (004), (112), (200), (201), (104), (202), (203), (114), (210), (104), and (211). These peaks appear at 31.73° , 40.15° , 50.29° , 51.71° , 57.53° , 58.34° , 58.61° , 60.53° , 65.53° , 66.29° , 74.91° , 79.78° , 80.71° , 80.99° , and 82.64° , respectively. The VESTA-generated structural model is shown inset of **Figure 1**, which confirms the hexagonal structure of ZnO, with a unit cell volume of 73.399 \AA^3 . The Full Width at Half Maximum (FWHM) method was used to calculate additional structural parameters, and the results were as follows: Crystallite size: 21 nm, Microstrain: 0.00534, Dislocation density: $0.0027460 \text{ nm}^{-2}$. These structural findings provide critical insights into the composition and crystalline characteristics of the ZnO/CNT nanocomposite.

3.2. Chemical Compositions of ZnO/CNT Nanocomposites

XPS was used to find surface contaminants, elemental composition and chemical states of the ZnO/CNT nanocomposite. The results are illustrated in **Figure 2(a)-(d)**. **Figure 2(a)** shows the wide-range survey spectrum, verifying that the main components are carbon (C), oxygen (O), and zinc (Zn). This overall scan validates the synthesis of the ZnO/CNT nanocomposite with no significant contamination. The quantitative analysis reveals the element's atomic percentages in the sample, as shown in **Table 1**.

The high-resolution C1s spectrum is presented in **Figure 2(b)**. Deconvolution of

this spectrum reveals peaks centered at 284.6 eV (C–C), 285.8 eV (C–OH), 287 eV (C=O) and 288.8 eV (O–C=O), indicating the presence of graphitic carbon and various carbon-oxygen functional groups on the CNT surface, confirming the active fictionalization of CNT with ZnO. **Figure 2(c)** displays the O 1s core-level spectrum, where a strong peak is observed at ~530.1 eV, corresponding to lattice oxygen (O^{2-}) in ZnO. Additional minor features suggest the presence of oxygen vacancies, such as surface-adsorbed species etc., which may enhance photocatalytic activity.

Table 1. Atomic percentages of elements present in ZnO/CNT nanocomposite.

Component Name	Atomic %
Zn 2p _{1/2}	30
Zn 2p _{3/2}	54.09
O 1s	14.41
C 1s	1.50

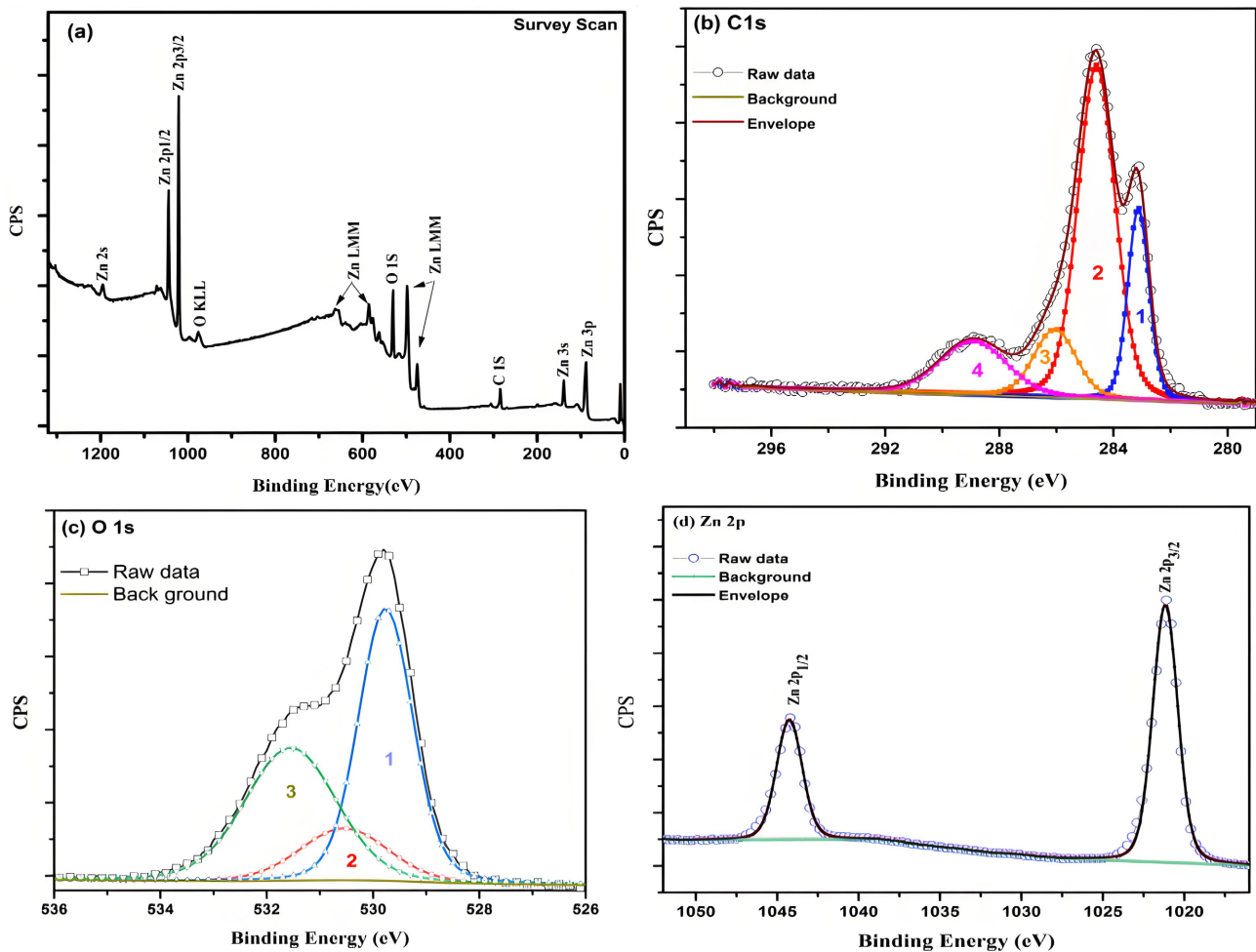


Figure 2. X-ray Photoelectron Spectroscopy (XPS) analysis of the ZnO/CNT nanocomposite: (a) survey spectrum (b) C 1s core level spectrum [peaks as 1: C–C, 2: C–OH, 3: C=O, 4: O–C=O], (c) O 1s [peaks as 1: O^{2-} , 2: oxygen vacancies, 3: oxygen from functionalized CNT] core level spectrum, and (d) Zn 2p core level spectrum.

Figure 2(d) illustrates the Zn 2p spectrum, with well-defined peaks for Zn 2p_{3/2} at 1021 eV and Zn 2p_{1/2} at 1044 eV, confirming the Zn²⁺ oxidation state. These values are consistent with those reported for ZnO in the wurtzite phase. A minor binding energy shift (~0.95 eV) compared to pure ZnO suggests interaction between ZnO and CNT components. These results confirm the effective integration of ZnO and CNTs in the composite material and support the presence of chemical environments beneficial for photocatalytic dye removal. Similar observations were reported by Li *et al.* [13] and Hanif *et al.* [14], reinforcing the findings.

3.3. Optical Studies of ZnO/CNT Nanocomposites

The absorption behavior and band gap of the ZnO/CNT nanocomposites were investigated using UV–Visible spectroscopy. As illustrated in **Figure 3**, the absorption spectrum exhibits a notable peak at 370 nm, indicating the material's enhanced ability to absorb light in the near-UV region. This red shift compared to pure ZnO suggests strong electronic interactions between ZnO and CNTs, confirming the successful formation of the nanocomposite structure [15]. The Tauc plot, shown in the inset of **Figure 3**, estimates the band gap of the ZnO/CNT nanocomposite to be 2.25 eV, which is significantly lower than that of pristine ZnO. This band gap narrowing can be attributed to quantum confinement effects and interfacial charge transfer within the composite. Other researchers have reported similar red shifts and reductions in band gap values. For example,

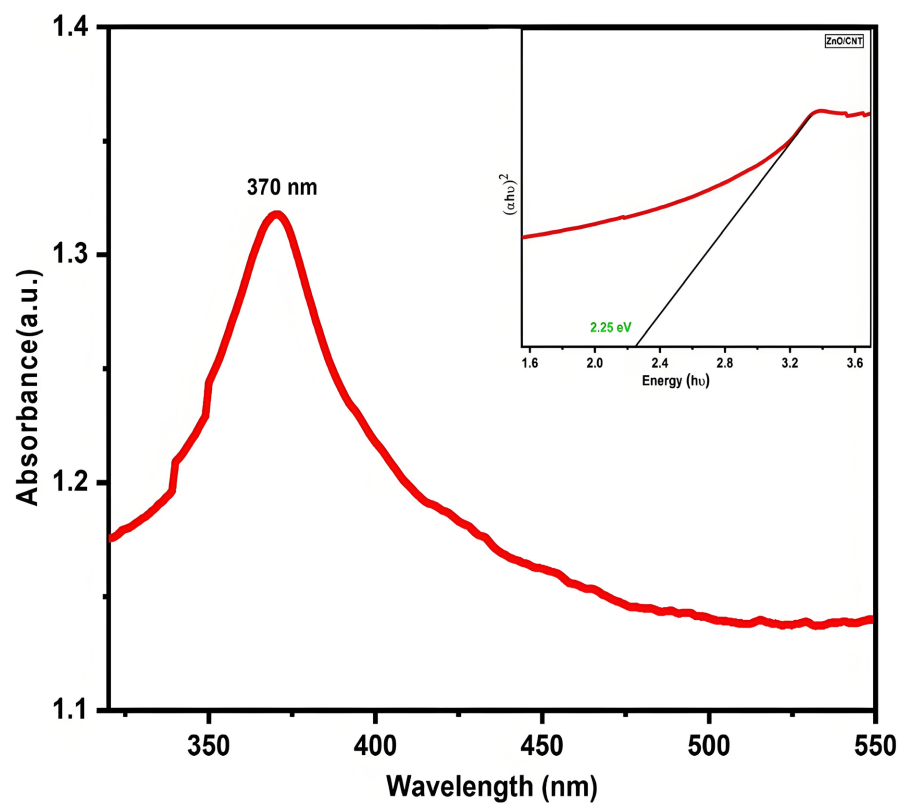


Figure 3. Absorbance and Band gap (inset figure) using Tauc plot of ZnO/CNT Nanocomposites.

Zhi *et al.* [15] observed that pure ZnO had an absorption maximum at 362 nm and a band gap of 3.18 eV, while N-doped ZnO/CNT composites showed a red-shifted peak at 368 nm with a reduced band gap of 2.65 eV [16]. These findings emphasize the impact of composite formation on the absorption characteristics and electronic structure of ZnO-based nanomaterials, which are critical parameters for photocatalytic applications.

3.4. Functional Characterization of ZnO/CNT Nanocomposites

FTIR spectroscopy was employed to analyze the functional groups present in the ZnO/CNT nanocomposite, with spectra recorded in the range of 4000 - 400 cm^{-1} , and are illustrated in **Figure 4**. A broad absorption peak at approximately 3247 cm^{-1} corresponds to the symmetric stretching vibrations of hydroxyl (O-H) groups, indicative of adsorbed water molecules on the ZnO/CNT surface. The peak observed at 1405 cm^{-1} is attributed to the stretching vibrations of the C-O-H functional group, suggesting the presence of hydroxyl functionalities.

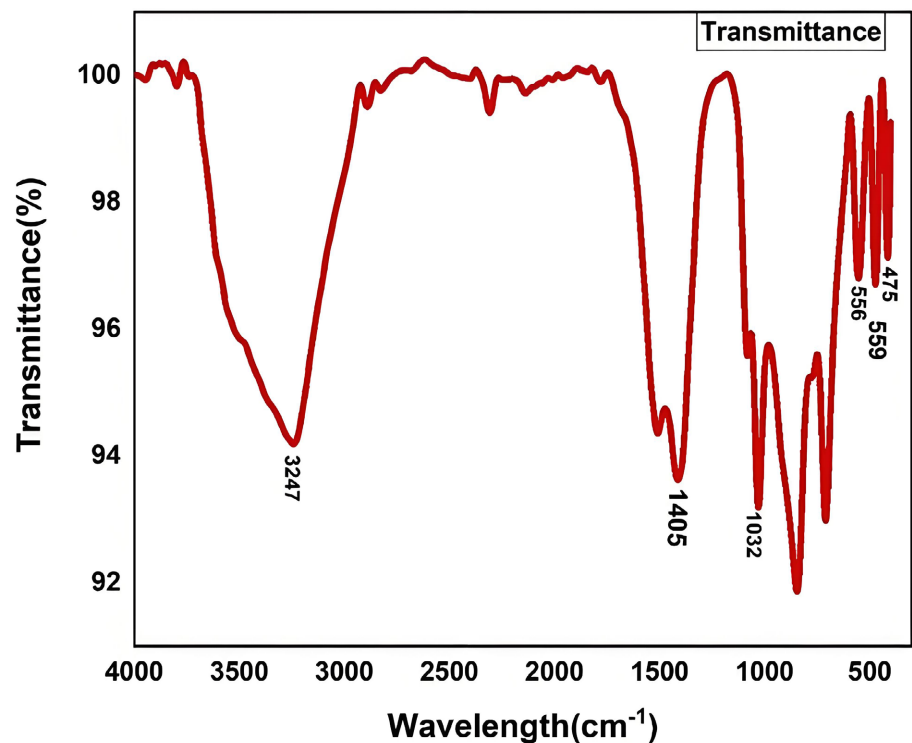


Figure 4. FTIR spectra of ZnO/CNT Nanocomposite.

Additionally, a vibration peak at 1032 cm^{-1} corresponds to the stretching of C-O bonds in carbon nanotubes, for ZnO/CNT nanocomposites. In the lower wave number region, peaks at 475 cm^{-1} , 556 cm^{-1} , and 559 cm^{-1} are indicative of Zn-O bond stretching, confirming the formation of the ZnO phase within the composite [17]-[19]. Overall, the FTIR analysis provides information for the successful integration of ZnO with CNTs, as evidenced by the characteristic absorption bands corresponding to hydroxyl groups, C-O-H functionalities, and Zn-O bonds.

3.5. Thermal Stability Analysis of ZnO/CNT Nanocomposites

Thermogravimetric measurements were conducted on the ZnO/CNT nanocomposites to measure its thermal stability and composition. The TGA curve, depicted in **Figure 5**, illustrates weight loss (wt%) over the temperature range of 43 °C to 500 °C, revealing distinct stages: (i) temperature range: 76.83 °C - 113 °C: A minor weight loss of approximately 0.25%, likely due to the evaporation of adsorbed water molecules; (ii) temperature range: 113 °C to 145 °C: about 2.04% weight reduction, potentially attributable to the desorption of residual solvents or the decomposition of organic impurities; (iii) temperature range: 145 °C to 290 °C: A further weight loss of 2.799%, which may correspond to the decomposition of functional groups associated with the CNTs; (iv) temperature range: 290 °C to 490 °C: A weight decrease of 1.66%, possibly resulting from the degradation of more stable organic components or the oxidation of CNTs.

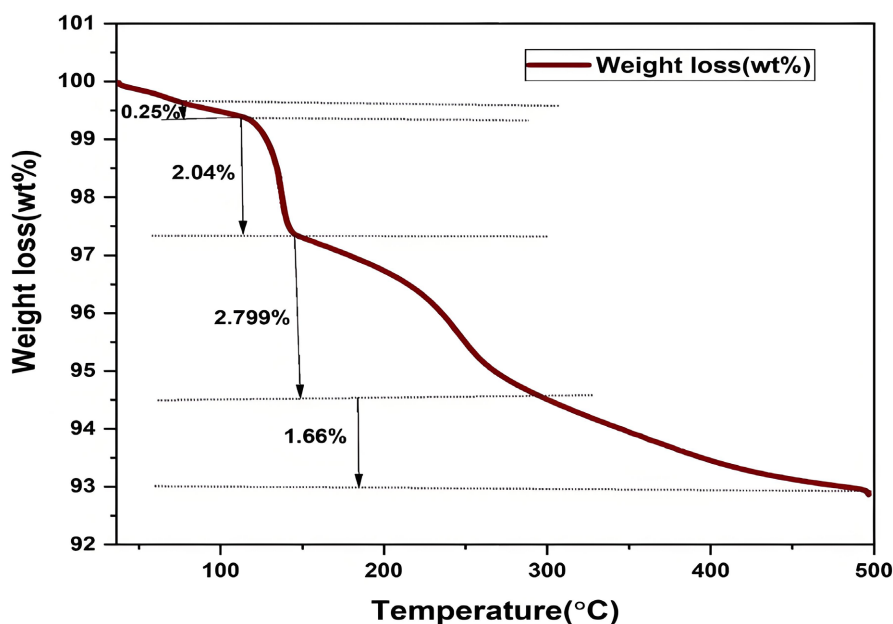


Figure 5. Thermogravimetric Analysis of ZnO/CNT Nanocomposites.

The cumulative weight loss observed up to 500 °C was 6.749%. ZnO-CNT nanocomposites exhibited thermal stability up to approximately 230 °C, with weight loss stages corresponding to the decomposition of functional groups and oxidation of CNTs. The study noted endothermic peaks around 233.7 °C and 326.3 °C, attributed to the breaking of physical bonds between functionalized CNTs and ZnO, and the decomposition of CNT functional groups into CO₂, respectively [20].

3.6. Photocatalytic Removal of MB Dye using ZnO/CNT Nanocomposites

The photocatalytic degradation of methylene blue (MB) dye using ZnO/CNT nanocomposites was investigated under ultraviolet (UV) and natural sunlight ir-

radiation. A 6-watt UV lamp served as the irradiation source throughout the experiment. **Figure 6(a)** illustrates the degradation profile of MB dye under UV exposure, indicating a gradual decrease in absorbance over time, with complete degradation observed after 80.5 hours. The corresponding signifying reduction in dye concentration under natural sunlight is depicted in **Figure 6(c)**, which reveals a steady decline in the characteristic MB absorption peak at 662 nm. **Figure 6(b)** **Figure 6(d)** further provide this trend, demonstrating a consistent decrease in dye concentration from its initial value under UV and sunlight. Tian *et al.* also show that ZnO-reduced graphene oxide-CNT composites achieved a degradation efficiency of 96% under UV light irradiation for 260 minutes, underscoring the role of CNTs in the photocatalytic activity [21].

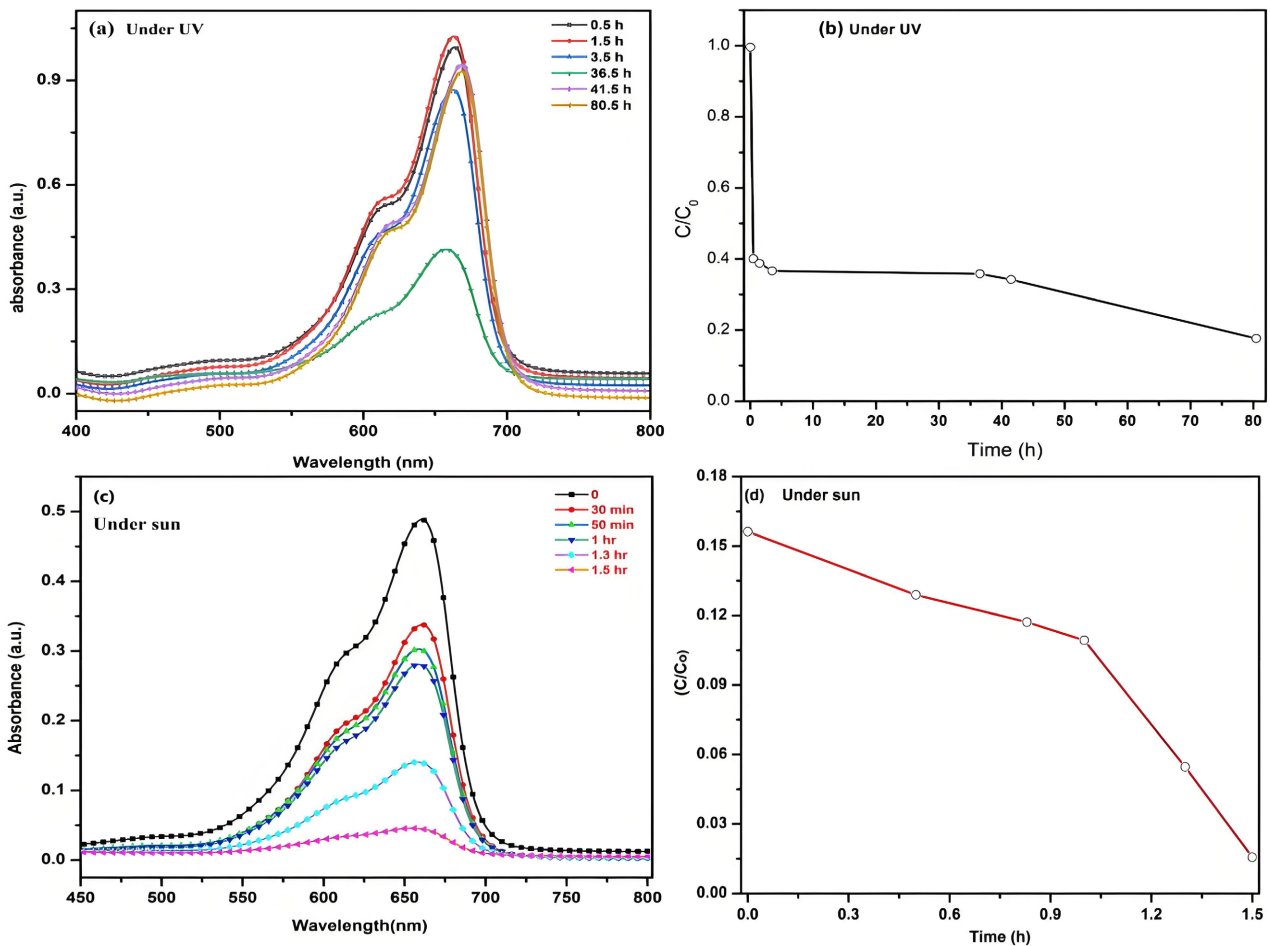


Figure 6. Photocatalytic performance of ZnO/CNT nanocomposite: UV-Vis absorption spectra during photocatalytic under (a) UV light and (c) Natural sunlight for dye removal; absorption of MB dye over time, with the corresponding concentration reduction of MB dye with respect to (b) UV light and (d) Sunlight.

Similarly, Chaudhary *et al.* [22] also reported that ZnO nanoparticles loaded on multi-walled CNTs exhibited enhanced photocatalytic activity and achieved over 90% MB degradation within a few hours through photoelectrochemical water splitting. The prolonged degradation time observed in the current study, com-

pared to these reports, may be attributed to differences in experimental conditions such as photocatalyst concentration, light intensity, and initial dye concentration (2.56948 mg) [23]. However, the results confirm the potential of ZnO/CNT nanocomposites as an effective photocatalyst for the degradation of organic pollutants like methylene blue under UV light irradiation [24]. This highlights how effective ZnO/CNT nanocomposites can be used for the removal of dye, even when used in small concentrations. However, their performance is remarkable under sunlight. This could be because densely packed or unevenly distributed CNTs may block or absorb some of the UV light, reducing how much light actually reaches the catalyst. Also, when there's too much catalyst, the active sites might get saturated, limiting further dye breakdown [25]. Beyond a certain amount, the particles may start to cluster, causing light scattering and shielding effects, which makes the photocatalyst less efficient overall [26].

4. Conclusion

ZnO/CNT nanocomposites were successfully synthesized using a ball-milling approach and systematically characterized to evaluate their structural, optical, thermal, and photocatalytic properties. XRD analysis revealed a transition from the crystalline structure of pure ZnO to a more amorphous state upon integration with CNTs, while XPS confirmed the chemical integrity and purity of the synthesized nanocomposite. The red-shifted absorption peak and narrowed band gap (~2.25 eV) observed in UV-Vis analysis suggested enhanced light absorption, beneficial for photocatalytic activity. FTIR results confirmed the presence of Zn–O bonds along with surface functional groups. Thermogravimetric analysis demonstrated that the composite was thermally stable up to 76.83°C, with subsequent weight losses indicating the decomposition of organic moieties and structural changes at higher temperatures. Crucially, photocatalytic experiments under UV irradiation showed that the ZnO/CNT nanocomposite exhibited effective dye degradation capability, successfully removing approximately 60% of MB dye within 4 hours. Furthermore, the composite demonstrated significant reduction of MB concentration within 80.5 hours under UV light, while it shown a remarkable towards almost zero dye concentration in just 1.5 hours under natural sunlight. Although the degradation rate under UV was relatively slower compared to pure ZnO, the composite maintained stable and sustained photocatalytic activity. This extended yet consistent performance can be attributed to morphological factors, surface area, and enhanced light interaction due to CNT incorporation. Overall, the ZnO/CNT nanocomposite holds great potential as a robust photocatalyst for wastewater treatment applications, particularly in the remediation of dye-contaminated industrial effluents. This immense difference suggests that sunlight (wider range of wavelengths and higher intensity) is much more effective in activating the ZnO/CNT catalyst. It also pointed out that the catalyst might be responsive to visible light, not just UV. This is a valuable insight, especially for practical use, as it shows the potential of using sunlight—a free and renewable

energy source—for faster and more energy-efficient environmental cleanup.

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Conflicts of Interest

The authors declare no conflicts of interest regarding this publication.

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