



Sustainable CNT–Biopolymer/TiO₂ Bio-nanocomposite Thin Films for Efficient Visible-light-driven Wastewater Remediation

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ABSTRACT

The photocatalytic degradation process is widely used to effectively remove organic contaminants from wastewater, contributing significantly to environmental protection and ecological sustainability. In this study, a CNT–chitosan/PVA/TiO₂ bio-nanocomposite thin film (CNT–CPT) was prepared using chitosan (CS) and polyvinyl alcohol (PVA) as the matrix for loading TiO₂ nanoparticles via solution casting. Furthermore, blending with carbon nanotubes (CNTs) improved charge-carrier separation and enhanced photocatalytic activity. The novelty of this work lies in the synergistic integration of CNTs within the CS/PVA/TiO₂ matrix to improve charge separation and enable efficient visible-light-driven photocatalysis. The structural and morphological properties of the bio-nanocomposite films were investigated using XRD, FTIR, photoluminescence, and SEM. The photoluminescence properties of CNT–chitosan/PVA/TiO₂ (CNT–CPT) thin-films were compared with those of chitosan/PVA/TiO₂ (CPT) thin films to highlight the role of CNTs in reducing charge recombination. The bio-nanocomposite films were prepared with varying concentrations of CNT–TiO₂ (10 mg, 50 mg, and 100 mg) to optimize performance. The photocatalytic degradation efficiency of the CNT–CPT bio-nanocomposite was evaluated using methylene blue (MB) under visible-light irradiation. It was found that the bio-nanocomposite film prepared with 10 mg of CNT–TiO₂ exhibited the highest photocatalytic degradation efficiency toward MB. It was also observed that CNT–CPT films achieved higher MB degradation efficiency (94.5%) than CPT films (80.3%). The improved photocatalytic performance of CNT–CPT is attributed to reduced charge-carrier recombination resulting from enhanced charge separation facilitated by CNT incorporation.

Keywords: CNT–TiO₂ composite; Bio-nanocomposite thin film; Visible-light photocatalysis; Methylene blue degradation; Charge separation.

1. INTRODUCTION

In recent years, the development of advanced materials for environmental remediation has garnered significant attention due to the escalating concerns over water pollution and the imperative need for sustainable solutions. Among these materials, blended bio-nanocomposites present a promising avenue for addressing such challenges, offering a unique combination of natural and synthetic components tailored to achieve enhanced performance in pollutant degradation (Annu *et al.* 2024). However, despite significant progress, many reported photocatalytic systems suffer from limitations such as poor charge separation efficiency and reduced activity under solar irradiation, highlighting the need for improved material design.

This study focuses on the fabrication and characterization of a novel blended bio-nanocomposite film composed of chitosan (CS), polyvinyl alcohol

(PVA), titanium dioxide (TiO₂) nanoparticles, and carbon nanotubes (CNTs). The integration of these materials aims to exploit their respective properties synergistically, particularly for the photocatalytic degradation of methylene blue (MB), a common and persistent organic dye pollutant found in industrial wastewater. Chitosan exhibits excellent film-forming properties, biocompatibility, and biodegradability, making it an attractive matrix material (Croisier and Jérôme, 2013). PVA complements chitosan by enhancing mechanical strength and chemical stability (Bonilla *et al.* 2014). TiO₂, a widely used photocatalyst, facilitates the generation of reactive oxygen species responsible for pollutant degradation (Mohamad *et al.* 2022). However, pristine TiO₂ is primarily active under ultraviolet (UV) light and suffers from rapid electron–hole recombination. To address these limitations, carbon nanotubes (CNTs)

are incorporated into the composite. CNTs possess high electrical conductivity and large surface area, which enhance charge separation and electron transport, thereby improving photocatalytic efficiency. In addition, CNTs contribute to better light absorption, enabling improved photocatalytic activity under solar irradiation conditions (Yao *et al.* 2008).

The unique combination of CS, PVA, TiO₂, and CNTs in a blended bio-nanocomposite film provides a multifunctional platform for efficient pollutant degradation. The novelty of this work lies in the synergistic integration of CNTs within the CS/PVA/TiO₂ (CPT) matrix to enhance charge transport and enable effective photocatalytic performance under solar irradiation. Therefore, the present study aims to synthesize CNT–CPT bio-nanocomposite films and evaluate their photocatalytic efficiency for methylene blue degradation under direct solar irradiation. Furthermore, the study investigates the structural properties, degradation kinetics, and possible photocatalytic mechanism of the developed composite.

2. MATERIALS AND METHODS

2.1 Chemicals

The chemicals used were of analytical reagent (AR) grade and high purity. It included titanium(IV) isopropoxide (97%, Merck), polyvinyl alcohol (99% hydrolyzed, Sigma-Aldrich), chitosan (≥85% deacetylated, Sigma-Aldrich), benzyl alcohol (99%, Merck), acetic acid (99.5%), and ethanol (99.9%). Double-distilled water was used in the experiment.

2.2 Characterization Techniques

The PL spectra of the photocatalysts were collected with a UV–Vis–NIR spectrofluorometer (FP-8600 NIR Spectrofluorometer, JASCO, Japan) to analyze the charge-carrier recombination behavior. The UV–visible absorption spectra were recorded using a UV–visible spectrophotometer (JASCO V-670, Japan) to monitor the photocatalytic degradation of methylene blue. The structural characterization of the photocatalysts was carried out using an X'Pert X-ray diffractometer

(PANalytical) with Co K α radiation over a 2 θ range of 0–150°. FT-IR spectra were recorded using KBr discs on a PerkinElmer 1650 spectrometer in the range of 4000–400 cm^{−1} to identify functional groups.

2.3 Synthesis of CNT–TiO₂ Nanocomposite

CNT (0.25 g) was dispersed in 164 mL of ethanol using an ultrasonic bath at a power of 80 W for 10 min, followed by a 5-minute rest period (Fig. 1). This cycle was repeated two more times to obtain a homogeneous dispersion (Solution 1). Separately, 10.8 g of benzyl alcohol was mixed with 50 mL of deionized (DI) water and stirred for 10 min to prepare Solution 2. Solution 1 was then transferred into a volumetric flask placed in an ice bath. Once the temperature of Solution 1 reached 0 °C, Solution 2 was gradually added under continuous stirring. The resulting mixture was stirred for 2 h to obtain Solution 3. In another container, 17 mL of titanium isopropoxide (TTIP) was added dropwise to 80 mL of ethanol and stirred for 30 min to form Solution 4. Subsequently, Solution 4 was slowly introduced into Solution 3 under continuous stirring at constant speed (500 rpm) to ensure uniform mixing for an additional 2 h. The final mixture was then allowed to age at room temperature, followed by filtration and drying in an oven at 80 °C.

2.4 Synthesis of CNT–CPT Bio-nanocomposite Film

500 mg of chitosan was dissolved in 20 mL of aqueous acetic acid under continuous stirring to obtain a homogeneous solution (Fig. 2). Separately, 500 mg of PVA was dissolved in 20 mL of hot distilled water (~80 °C) with constant stirring for 30 min. The two solutions were then mixed under stirring to form a uniform polymer blend. Subsequently, the synthesized CNT–TiO₂ nanocomposite was added in varying concentrations (10 mg, 50 mg, and 100 mg) into the polymer matrix and stirred thoroughly to obtain a viscous gel. The resulting mixture was cast into petri dishes and dried at room temperature for 48 h to form bio-nanocomposite thin films. The prepared films were carefully peeled off and used for further characterization and photocatalytic studies.

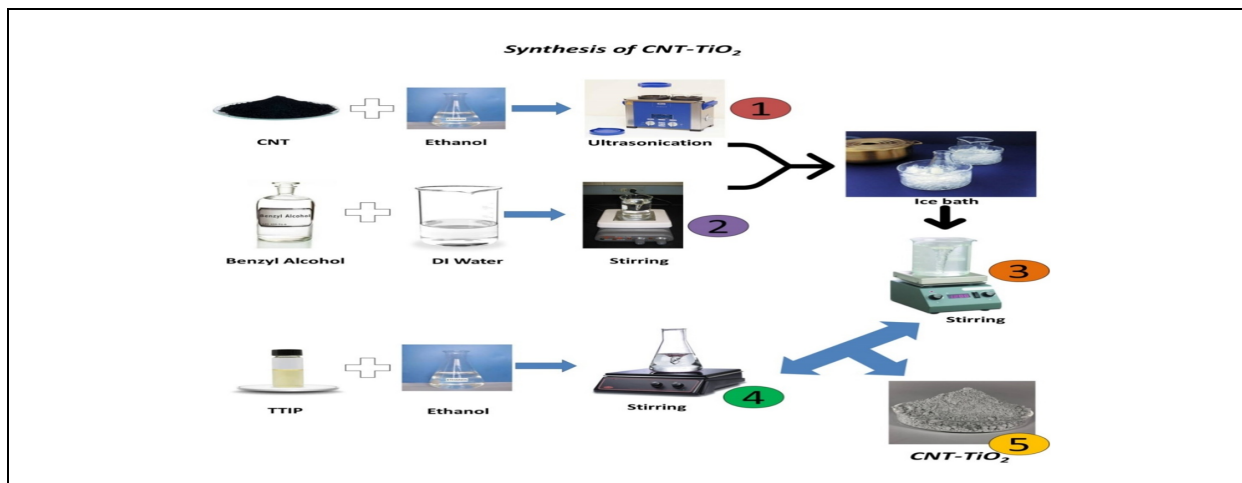
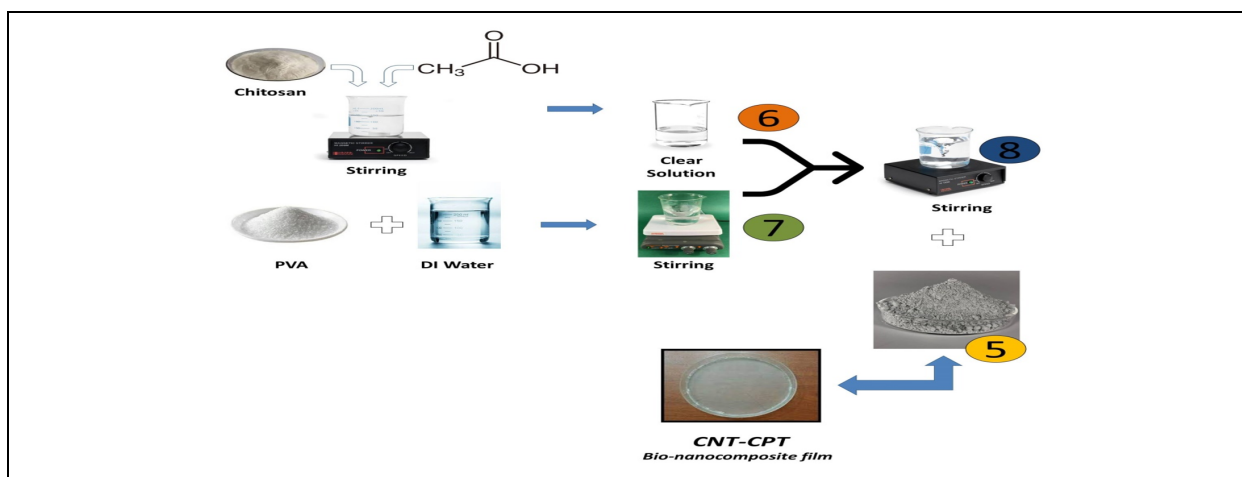
Fig. 1: Synthesis of CNT-TiO₂ nanocomposite

Fig. 2: Synthesis of CNT-CPT bio-nanocomposite film

3. RESULTS AND DISCUSSION

3.1 PL Studies

Fig. 3 shows the room temperature PL spectra of thin-film photocatalysts of CNT-CPT and CPT bio-nanocomposite thin films recorded at an excitation wavelength of 260 nm. The results showed a decrease in the PL intensity of the CNT-CPT-doped thin film compared to that of the CPT thin film. In general, lower photoluminescence intensity indicates a reduced rate of electron-hole recombination and, consequently, enhanced photocatalytic activity of the photocatalyst (Yu *et al.* 2003; Li *et al.* 2001). This suggests that the incorporation of CNTs improves charge separation efficiency.

3.2 CNT-CPT thin films prepared with different concentrations of CNT-TiO₂

To further understand the effect of CNT-TiO₂ concentration on charge carrier dynamics, PL analysis

was carried out for samples with different dopant concentrations. Fig. 4 shows the PL spectra of the CNT-CPT bio-nanocomposite thin-films prepared with different concentrations of CNT-TiO₂ (10 mg, 50 mg, and 100 mg) recorded with an excitation wavelength of 240 nm. With the increase in dopant concentration, the increase in PL intensity is observed (Agrawal, 2022). In general, lower PL intensity indicates reduced charge-carrier recombination and enhanced photocatalytic activity (Yu *et al.* 2003; Li *et al.* 2001). This may be attributed to the agglomeration of nanoparticles and the formation of recombination centers, which adversely affect charge separation. In contrast, the sample with 10 mg CNT-TiO₂ exhibits relatively lower PL intensity, indicating optimal charge separation and reduced recombination. Therefore, the thin film prepared with 10 mg CNT-TiO₂ was selected for further photocatalytic studies due to its superior charge carrier dynamics. The variation in excitation wavelength (260 nm and 240 nm) was chosen based on the absorption characteristics of the samples and does not affect the comparative interpretation of PL results.

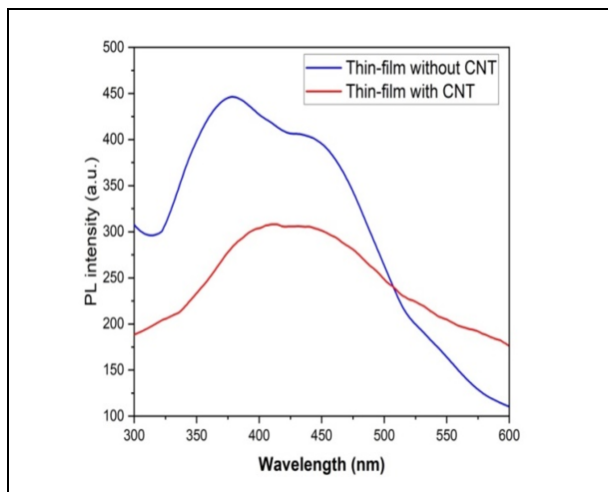


Fig. 3: PL spectra of CPT and CNT-CPT bio-nanocomposite thin films

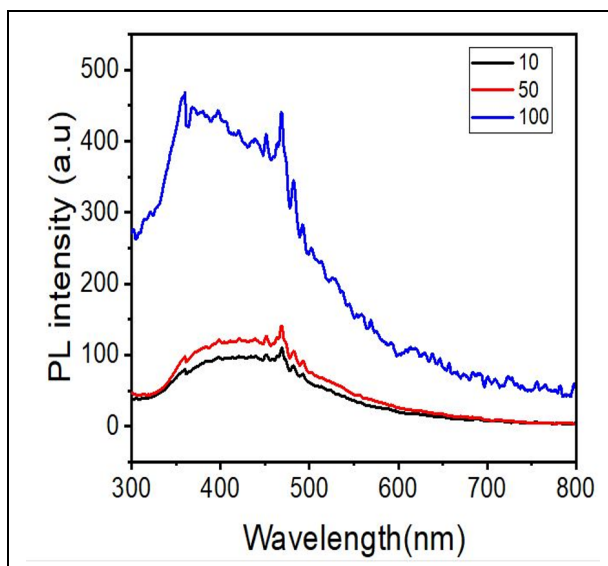


Fig. 4: PL spectra of CNT-CPT bio-nanocomposite thin films prepared with different concentrations of CNT-TiO₂ (10 mg, 50 mg, and 100 mg)

3.3 X-ray Diffraction

X-ray diffraction (XRD) measurements were performed to determine the structural properties of the prepared CNT-CPT bio-nanocomposite thin-film photocatalyst (Fig. 5). The peak at $2\theta = 21.98^\circ$ represents the chitosan@PVA complex (Meydan *et al.* 2024). The peaks at around 25° and a broad peak at 43° in the XRD pattern correspond to the (002) and (100) reflection planes of CNTs, confirming their successful incorporation into the composite (JCPDS Card No.75-1621) (Abbasi *et al.* 2013). The XRD image shows the presence of characteristic peaks at 34° and 63° , which are well indexed to the standard rutile TiO₂ phase (JCPDS Card No. 89-4920) (Pugazhendhi *et al.* 2017). It is noted that slight overlapping of diffraction peaks may occur

due to the proximity of CNT and TiO₂ reflection planes, indicating good dispersion and interaction between the components within the composite matrix. The presence of both sharp peaks corresponding to TiO₂ and CNTs and a relatively broad peak associated with the polymer matrix suggests the coexistence of crystalline and amorphous phases. This mixed-phase structure is beneficial for photocatalytic activity, as the crystalline regions contribute to charge generation while the amorphous matrix enhances the adsorption of dye molecules.

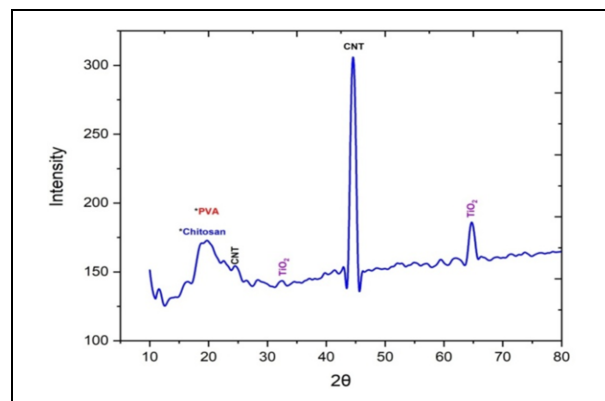


Fig. 5: XRD pattern of CNT-CPT bio-nanocomposite film

3.4 FTIR

The FTIR spectrum of the CNT-CPT bio-nanocomposite film is presented in Fig. 6. A broad absorption band observed in the region $3200\text{--}3600\text{ cm}^{-1}$ (including peaks at 3628 cm^{-1} and 3283 cm^{-1}) corresponds to O-H stretching vibrations, indicating the presence of hydroxyl groups and hydrogen bonding within the composite matrix. The peak around $2978\text{--}2924\text{ cm}^{-1}$ is attributed to C-H stretching vibrations of alkyl groups, while the band at 1462 cm^{-1} corresponds to CH bending vibrations (Esmacili and Beni, 2014; El-Hefian *et al.* 2010). The absorption band at 1599 cm^{-1} is assigned to N-H bending (amide II) of chitosan, and the peak at 1686 cm^{-1} corresponds to C=O stretching (amide I). The band at 1381 cm^{-1} is attributed to CH₃ wagging vibrations. The peak at 1080 cm^{-1} is associated with C-O stretching vibrations, confirming the presence of the polymer matrix (El-Hefian *et al.* 2010). The absorption peak at 851 cm^{-1} is assigned to C-C vibrations of PVA (Alhosseini *et al.* 2012), while the broad band in the range of $550\text{--}800\text{ cm}^{-1}$ corresponds to Ti-O-Ti vibrations, indicating the incorporation of TiO₂ nanoparticles (Abbasizadeh *et al.* 2013). The slight shifting and broadening of characteristic peaks suggest strong intermolecular interactions between chitosan and PVA through hydrogen bonding. Furthermore, the presence of CNTs and TiO₂ within the polymer matrix leads to interactions that enhance compatibility and dispersion, confirming the successful formation of the bio-nanocomposite structure.

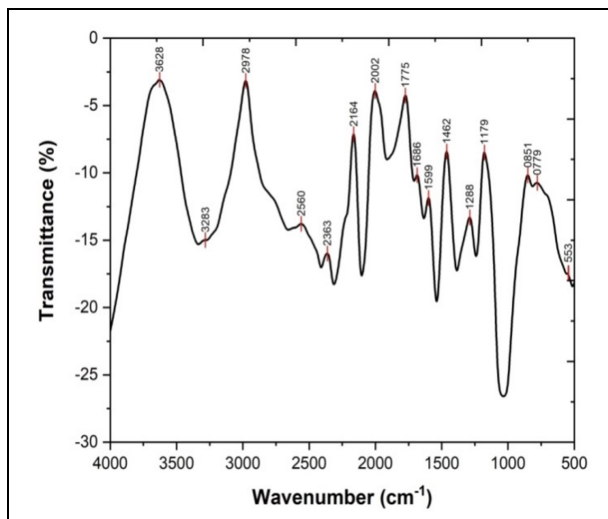


Fig. 6: FTIR spectrum of CNT-CPT bio-nanocomposite film

3.5 Scanning Electron Microscope

The surface morphology of the CNT-CPT bio-nanocomposite film was analyzed using SEM (Fig. 7). The micrograph of the bio-nanocomposite thin-film shows a smooth and uniformly produced surface. The SEM image reveals that the grains are spherical in shape and uniformly distributed all over the surface of the substrate. It is observed that the CNT-TiO₂ nanoparticles in the polymer matrix are distributed homogeneously over the film surface. However, slight agglomeration is observed in certain localized regions, which may be attributed to the high surface energy of nanoparticles and their tendency to cluster. Despite this, the overall morphology indicates good dispersion with minimal aggregation. The SEM image was recorded at a magnification of 3000 $\times$, and the morphology was analyzed using a scale bar of 10 μ m.

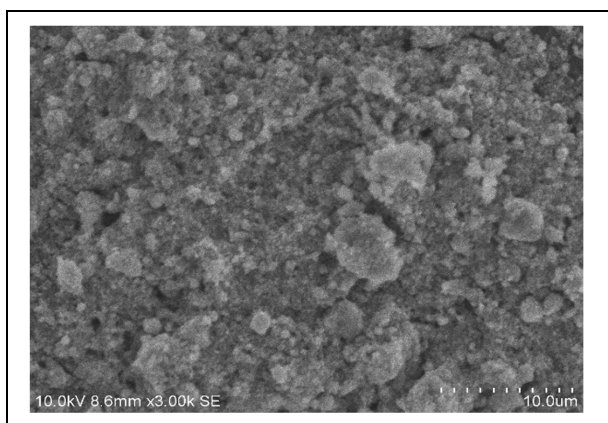


Fig. 7: SEM image of the CNT-CPT bio-nanocomposite film

3.6 Photocatalytic Degradation of MB

Fixed surface area (2.5 inch $\times$ 2.5 inch) of the CNT-CPT bio-nanocomposite film (prepared using 10

mg of CNT-TiO₂) was selected for photodegradation studies and kept in a petri dish. The MB stock solution with a concentration of 10 ppm was prepared by dissolving 10 mg of MB in 1000 mL of distilled water. From this solution, 25 mL was taken and placed in a Petri dish. The immersed film was then allowed to equilibrate in dark conditions for 30 min to achieve adsorption-desorption equilibrium between photocatalysts and dye molecules. After equilibrium was attained, the solution was exposed to direct solar irradiation between 11:00 and 14:00 at a temperature of 32 $\pm$ 2 $^{\circ}$ C. The degradation efficiency of the bio-nanocomposite film was evaluated by measuring the absorption every 30 min using a UV-Vis spectrophotometer. The experiment was carried out for 180 min.

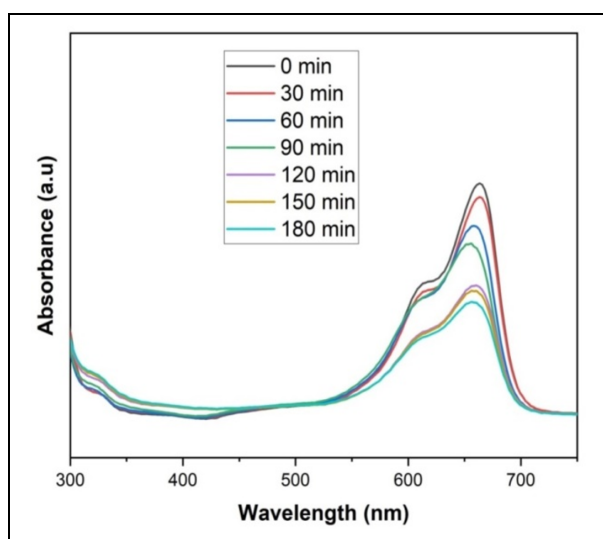


Fig. 8: Visible light-induced photocatalytic degradation of MB using CNT-CPT bio-nanocomposite

3.7 Effect of Immersion Time

The photocatalytic degradation of MB using the CNT-CPT bio-nanocomposite under solar irradiation is shown in Fig. 9. The results indicate that the concentration of MB gradually decreased as the reaction time increased. As illustrated in Fig. 8, a progressive decline in the absorption intensity of MB was observed after the addition of the CNT-CPT bio-nanocomposite under solar-light irradiation. It is found that photodegradation gradually increased with the increasing immersion time. Furthermore, the photodegradation behavior exhibited no variation in the absorption band after an incubation period of 180 min under solar light in the presence of the CNT-CPT bio-nanocomposite. The obtained data agreed with the calculated photodegradation rate/efficiency. The relation between the photodegradation efficiency and the immersion time was calculated using Eq 1.

The degradation efficiency can be defined as follows (Li *et al.* 2008).

$$\text{Degradation Efficiency (\%)} = (C_0 - C_e) / C_0 \times 100 \quad \dots (1)$$

In this equation, C_0 indicates the initial dye concentration and C_e refers to the concentration of the dye after photodegradation, both expressed in mg/L.

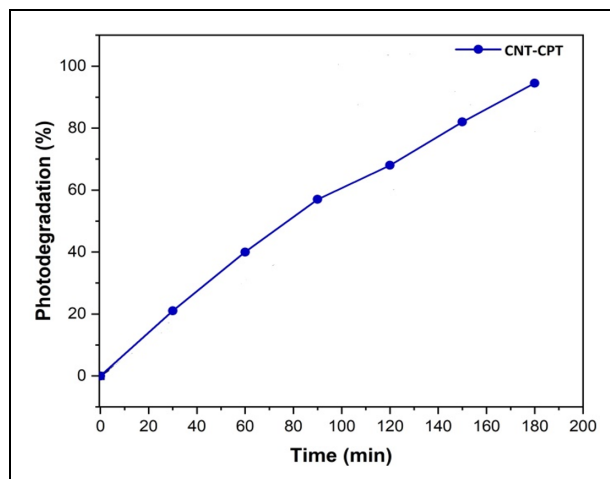
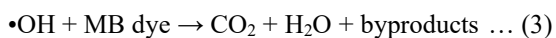


Fig. 9: Photocatalytic degradation of MB using CNT-CPT bio-nanocomposite

As illustrated in Fig. 9, the degradation of MB was minimal during the first 30 min of immersion. However, the photodegradation efficiency progressively increased with increasing immersion time. The CNT-CPT bio-nanocomposite exhibited a maximum degradation efficiency of 94.5% for MB after 180 min.

3.8 Effect of pH

The pH of the solution influences the surface charge distribution of the photocatalyst and the electrostatic interactions between the MB dye molecules and the photocatalyst surface in an aqueous medium. Therefore, determining the optimal pH range for maximum dye degradation is essential (Fig. 10). The removal efficiency of MB dye gradually increased as the pH rose from 3 to 11, indicating improved photocatalytic activity under alkaline conditions. According to Eskelinen *et al.* (2010), higher pH conditions can reduce the recombination of photo-generated electrons and holes, thereby improving photocatalytic performance. Thus, an enhancement of the catalytic activity occurred (Eskelinen *et al.* 2010). Another reason mentioned by Meydan *et al.* (2024) was that the increase in the pH led to the ease of formation of $\bullet\text{OH}$ radicals, as given below (Andal *et al.* 2019).



Maximum degradation efficiency of the studied catalysts was achieved under alkaline conditions at a pH

of 8–9, confirming the role of hydroxyl radical formation in the degradation process (Zhu *et al.* 2019).

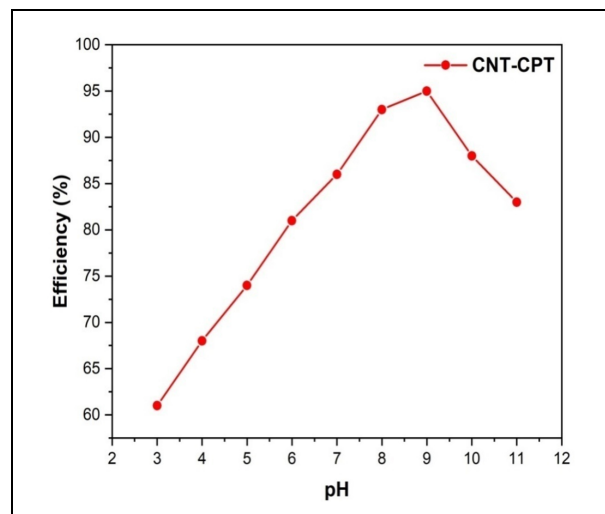


Fig. 10. Effect of pH on degradation efficiency

3.9 Comparison of CNT Blended and Unblended Bio-nanocomposites

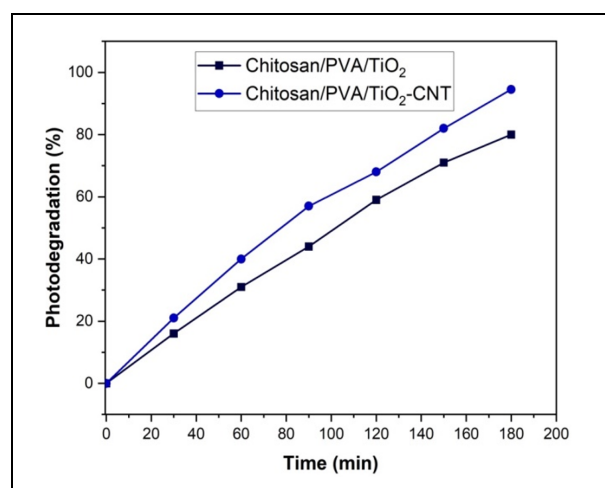


Fig. 11: Photocatalytic degradation of MB using CNT-CPT and CPT bio-nanocomposites

The enhanced photocatalytic performance of CNT-CPT compared to CPT can be attributed to the presence of CNTs, which act as electron acceptors and facilitate charge separation. The high electrical conductivity of CNTs improves electron transport and suppresses recombination losses, thereby increasing the availability of reactive species for degradation. The minimum degradation efficiency observed at pH = 3 may be attributed to increased charge-carrier recombination and the reduced availability of hydroxyl ions under acidic conditions. The results are consistent with those of previous studies (Seralathan *et al.* 2021; Wang and Mano, 2008), although the improved performance in the present

work is more pronounced due to the synergistic effect of CNT incorporation.

4. CONCLUSION

In this work, the prepared CNT–CPT bio-nanocomposite was qualitatively characterized using PL, FTIR, and XRD analyses. The SEM analysis of the bio-nanocomposite thin films revealed a smooth surface morphology and a uniform distribution of particles. The bio-nanocomposite prepared with 10 mg of CNT–TiO₂ exhibited the best performance because of the lower charge-carrier recombination rate observed in the photoluminescence studies. The CNT-doped bio-nanocomposite demonstrated efficient photocatalytic degradation of methylene blue under solar irradiation, achieving a maximum degradation efficiency of 94.5% within 180 minutes. The enhanced performance is attributed to improved charge separation and electron transport facilitated by CNTs, which promote the generation of reactive species such as hydroxyl (•OH) radicals. The results also indicate that thin-film photocatalysts exhibit better performance than powder photocatalysts and that CNT incorporation significantly enhances photocatalytic efficiency compared to undoped systems. However, factors such as long-term stability, reusability, and large-scale applicability need further investigation. Future work may focus on enhancing visible-light activity, optimizing the composition, and evaluating applications in real wastewater treatment. Therefore, CNT-doped chitosan/PVA/TiO₂ bio-nanocomposite films exhibit strong potential as efficient and sustainable photocatalysts for wastewater treatment applications.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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