



Sustainable Synthesis of Chitosan-SnO₂ Nanocomposite for Enhanced Visible-Light Photocatalytic Degradation of Crystal Violet Dye

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ABSTRACT

A chitosan–tin oxide (CS–SnO₂) nanocomposite was prepared by a co-precipitation-based approach and investigated for the visible-light-assisted removal of Crystal Violet (CV) dye from aqueous solution. The incorporation of chitosan was intended to provide additional adsorption sites and improve the dispersion of SnO₂ nanoparticles. The prepared nanocomposite was characterized using FT-IR, XRD, FE-SEM, EDS, TGA, and UV-Vis spectroscopy. XRD analysis indicated the formation of crystalline SnO₂ with an estimated crystallite size of 4.34 nm, while FE-SEM analysis showed an average particle size of 69.4 ± 13.6 nm. The optical band gap was estimated to be 3.40 eV for the CS–SnO₂ nanocomposite compared with 3.65 eV for SnO₂. The removal of CV was investigated as a function of catalyst dosage, initial dye concentration, temperature, pH, and irradiation time. A considerable contribution from adsorption was observed during the initial dark-equilibrium period. Under the investigated conditions, the highest CV removal efficiency of 97.5% was obtained at an initial dye concentration of 5 mg/L, catalyst dosage of 60 mg, pH 12, and 180 min of visible-light irradiation. The photocatalytic process followed an apparent pseudo-first-order kinetic model, with a rate constant of 0.0033 min⁻¹ at 5 mg/L CV. The catalyst also exhibited measurable reusability over four successive cycles, although a decrease in removal efficiency was observed during repeated use. The results demonstrate the potential of the CS–SnO₂ nanocomposite for combined adsorption and visible-light-assisted dye removal.

Keywords: CS–SnO₂ nanocomposite, Crystal Violet, Photocatalysis

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Introduction

The large release of synthetic dye-containing effluents from industries such as textiles, paper, leather, cosmetics, and printing endangers aquatic ecosystems and human health [1]. Many synthetic dyes have highly conjugated aromatic structures that make them chemically stable and resistant to biological breakdown, heat, and light [2]. Crystal violet (CV), a cationic triphenylmethane dye common in various applications, is particularly problematic because of its bioaccumulation, cytotoxicity, mutagenicity, and possible carcinogenic effects. At very low concentrations, it decreases light penetration in water, affecting photosynthesis and causing oxygen deficiency [3]. Thus, the development of sustainable techniques that allow complete degradation of these pollutants is essential. Of the many Advanced Oxidation Processes (AOPs), semiconductor photocatalysis stands out due to its capacity to completely mineralize organic pollutants to CO₂, H₂O, and inorganic ions through light [4]. Tin dioxide

(SnO₂) is an n-type semiconductor whose value lies in its chemical stability, non-toxic nature, affordability, and high electron mobility [5]. Nevertheless, the utilization of SnO₂ is restricted by its large bandgap (3.6–3.8 eV), which only allows its photoactivation through ultraviolet light and its rapid recombination of photogenerated electrons and holes (e⁻/h⁺) [6]

In order to rectify these deficiencies, a modification of SnO₂ by biopolymer-based supporting matrices was shown to be a very promising approach. The cationic polysaccharide called chitosan (CS) that is synthesized through the process of deacetylation of chitin has different functional groups such as -NH₂ and -OH [7]. The presence of functional groups increases the number of attachment sites for organic pollutants and helps in making strong chemical or electrostatic bonds with the surface of semiconductors. CS along with SnO₂ acts as a capping agent preventing from agglomeration and providing a decrease in the band gap of semiconductors under visible light [8]. While there are studies about the



properties of CS-metal oxides nanocomposites and their applications in the environment, the focus of this research will be placed on the preparation and physicochemical investigation of the CS-SnO₂ nanocomposite and its application in the degradation of Crystal Violet under visible light. This research in particular will examine the influence of catalyst amount, initial concentration of the dye, temperature, the value of pH of the solution, and the reaction time on the efficiency of dye degradation.

Results And Discussion

2.1.1 Materials

The chemicals used in this study include Tin II chloride dihydrate (SnCl₂ · 2H₂O (98.0% purity)), Chitosan (CS, 85.0% purity), Glacial Acetic Acid (CH₃COOH, 99.0% purity), Hydrochloric acid (HCl, 36.0% purity), Sodium Chloride (NaCl, 99.0% purity), Crystal Violet (CV, 90.0% purity), Sodium hydroxide (NaOH, 99.0% purity), and deionized water.

2.1.2. Instrumentation

The resulting materials were then characterized using Scanning Electron Microscopy (SEM; Jeol JCM-6000), EDX, and UV-Vis spectroscopy (UV-2450PC Pharmaspec Spectrophotometer). FT-IR spectra were recorded using a Rayleigh (WQF-510A) FTIR spectrophotometer. A Philips X-ray diffractometer with Ni-filtered Cu K α radiation was used to perform the X-ray diffraction measurements.

2.2.1. Synthesis of chitosan - tin oxide (CS-SnO₂) nanocomposite

Chitosan (0.20 g) was dissolved in 25 mL of 5% (v/v) acetic acid solution and stirred for 30 min to obtain a homogeneous polymer solution. Separately, SnCl₂·2H₂O was dissolved in distilled water to obtain a 0.2 M tin precursor solution. The tin precursor solution was then gradually combined with the chitosan solution under continuous stirring at room temperature. The resulting mixture was maintained under stirring for 2 h to promote interaction between the tin precursor species and the functional groups of chitosan. The resulting solid was

separated from the reaction mixture, washed repeatedly with distilled water to remove soluble residual species, and dried at 100 °C for 1 h. The obtained material was subsequently characterized to investigate its structural, morphological, and physicochemical properties.

The proposed role of chitosan in the preparation is associated with its hydroxyl and amino functional groups, which can interact with tin-containing precursor species and contribute to the stabilization and dispersion of the inorganic component [9].

2.2.2. Photocatalytic degradation of dyes

The photocatalytic activity of CS-SnO₂ nanocomposites was analyzed by testing the dye CV degradation rate under visible light irradiation. Generally, 50 mg of the catalyst was introduced into 50 mL of dye solution having an initial concentration of 10 mg/L. The mixture was stirred in dark conditions for 30 minutes until adsorption/desorption equilibrium. Next, the mixture was exposed to visible light produced by the 50 W LED UFO lamp (7. STAR, model T150), which emits 6000 Lm having color temperature of 10000 K and peak wavelength in range of 450-460 nm. This stage lasted for 180 minutes. During the whole process 4 mL of the sample was extracted and centrifuged to eliminate catalyst particles. The degree of dye decomposition was estimated by absorbance measurement at 580 nm with UV-VIS spectrophotometer. Influence of temperature, dye concentration, catalyst concentration, pH of solution and irradiation time was studied and optimized.

Experimental

3.1. Characterization

Different physical parameters of CS-SnO₂ nanocomposites like the crystal structure, morphology, and thermophysical parameters were studied through several methods including XRD, FT-IR, FE-SEM, EDS, UV-Vis, and TGA. The crystalline nature of SnO₂ and efficient dispersion of nanoparticles in CS matrix were proved through XRD and FT-IR analysis, particularly for the 4000-400 cm⁻¹ region. Surface morphology and structure of particles were analyzed through FE-SEM, while the

optical bandgap of the composite was calculated through UV-Vis (200-800 nm).

3.1.1 FTIR analysis of CS-SnO₂ Nanocomposites

In the FTIR spectrum of chitosan (CS) as shown in Figure 1(a), there is an intensive band at 3330 cm⁻¹, which is due to O-H and N-H stretches [10]. The absorption bands at ~1638 cm⁻¹ and ~1426 cm⁻¹ correspond to amide I (C=O) and coupled OH/CH₂ bends, respectively. Moreover, the absorption band at ~1022 cm⁻¹ corresponds to C-O stretches, which confirm the polysaccharide nature of CS [11].

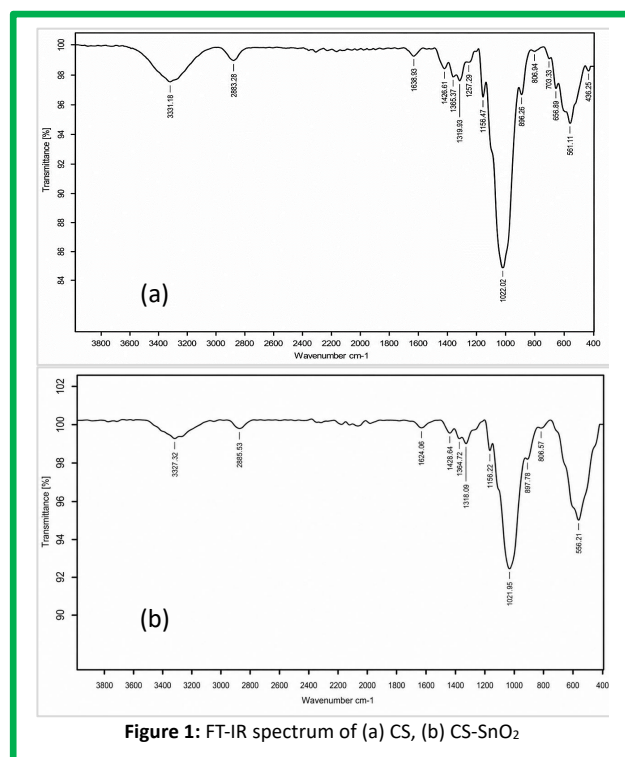


Figure 1: FT-IR spectrum of (a) CS, (b) CS-SnO₂

The FTIR spectrum for CS-SnO₂ in Figure 1(b) has a broad peak at about 3330 cm⁻¹ due to vibrations of O-H and N-H bonds. There is the presence of typical CS peaks at ~1638 cm⁻¹ (amide I) and ~1022 cm⁻¹ (C-O), though they have been slightly shifted compared to CS alone [12]. There is also a new peak in the range of ~560 to 650 cm⁻¹, which is due to Sn-O vibrations, thus proving the addition of SnO₂ into the sample.

3.1.2. XRD analysis of CS-SnO₂ Nanocomposites

X-ray diffraction analysis pattern of the nanocomposite CS-SnO₂ (Figure 2) shows that it is both amorphous and crystalline, which confirms the synthesis of a hybrid material. A broad XRD pattern at 2θ ≈ 20° proves the semi-crystalline nature of chitosan, while the peak at 27.1513° represents the (110) crystal plane of rutile SnO₂ in the composite [14]. Peaks at 27.15°, 34.46°, 51.70°, and 63.91° represent the (110), (101), (211), and (301) crystal planes of tetragonal rutile SnO₂, respectively, proving the presence of SnO₂ NPs in the nanocomposite and indicating the retention of crystallinity in them even after embedding in the polymer without any secondary phase formation [15]. The decrease in peak intensity and broadening of peaks indicate proper dispersion of SnO₂ NPs and their interaction with the functional groups of chitosan. Also, the low intensity peak at 15.62° shows the amorphous region of chitosan, implying the disruption of polymer chain organization owing to incorporation of nanoparticles [16].

Further, the mean crystallite size of the nanocomposite CS-SnO₂ was calculated using the Debye-Scherrer formula.

$$D = K \lambda / \beta \cdot \cos(\theta) \quad (1)$$

Where K is the shape factor (0.9), λ is the wavelength of X-rays (Kα Cu = 0.15406 nm), β is the broadening of the peak at full width half maximum converted into radians, and (θ) is the angle of Bragg diffraction in specific consideration of the (110) plane at 2θ = 27.1513° with a measured FWHM of 1.9680°. Thus the average crystallite size was around 4.34 nm. The drastic decrease in the crystallite size of SnO₂ in the composite (4.34 nm) as opposed to the size of the pure SnO₂ NPs (20.74 nm) clearly suggests that the chitosan polymer matrix acts as an excellent capping and stabilization material during the synthesis process. This ensures that there is no overgrowth and agglomeration of the oxide crystals, which results in the lattice strain and peak broadening, while at the same time retaining the rutile crystalline structure of SnO₂.

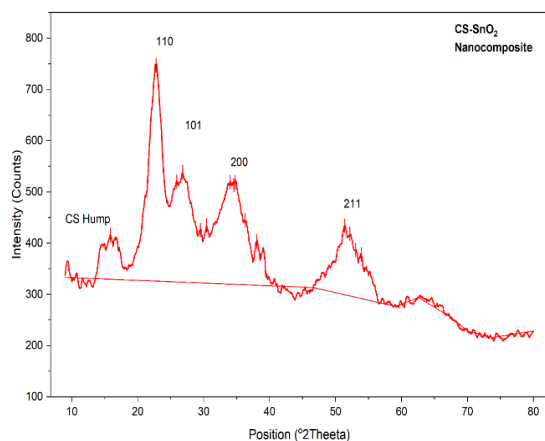


Figure 2: XRD pattern of CS-SnO₂ nanocomposite

3.1.3. Morphological and EDX Analysis of CS-SnO₂ nanocomposite

As seen from FE-SEM micrographs of CS-SnO₂ nanocomposite (Figure 3a), there are quasi-spherical particles having slightly tendency for agglomeration, which might be related to the fact that surface energy of SnO₂ NPs is extremely high [17]. Nevertheless, the nanoparticles are homogeneously dispersed in the chitosan matrix, which proves that chitosan successfully acts as a stabilizer and prevents excessive agglomeration of particles [8]. Analysis of particle size (Figure 3b) showed that the average size of CS-SnO₂ nanocomposite was (69.4±13.6 nm), which clearly shows the crucial role of polymer in nanoparticle growth and homogeneous distribution. This is probably due to strong electrostatic and coordination interactions between chitosan functional groups (amino and hydroxyl groups) and metal oxide surface.

The EDS analysis shown in **Figure 4** reveals the presence of O (43.10%), C (29.44%), Sn (19.12%), Cl (5.75%), and N (2.58%). The high oxygen content is attributed to the formation of SnO₂ NPs, while carbon and nitrogen originate from the chitosan matrix, which contains carbon-rich polymer chains and amino (–NH₂) functional groups [18]. The presence of tin confirms the

successful incorporation of SnO₂ into the composite structure. A minor amount of chlorine is also observed, which can be attributed to residual precursor salts (such as SnCl₂) used during the synthesis process [19]

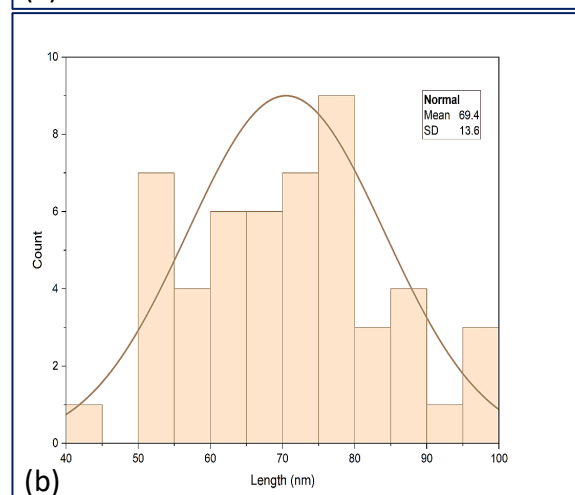
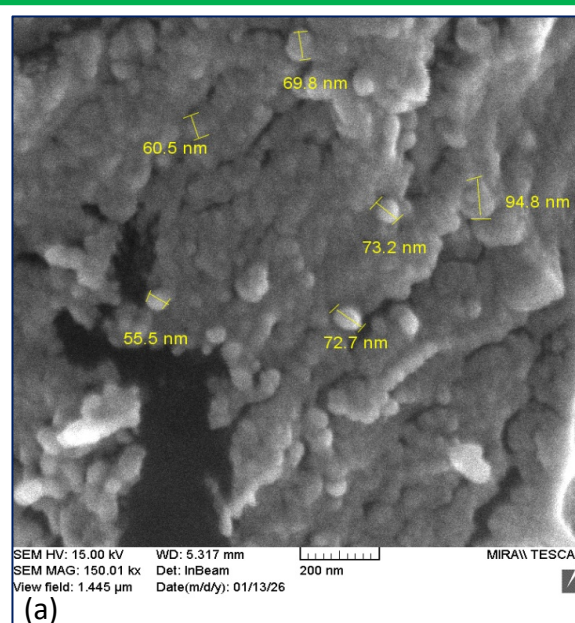


Figure 3. FE-SEM images of CS-SnO₂ at (a) 200 nm and (b) Particle size distribution histogram with a Gaussian normal distribution

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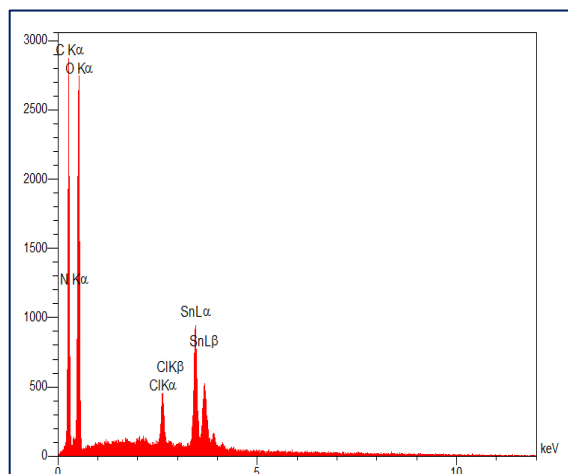


Figure 4. EDS spectrum of the CS-SnO₂ nanocomposite

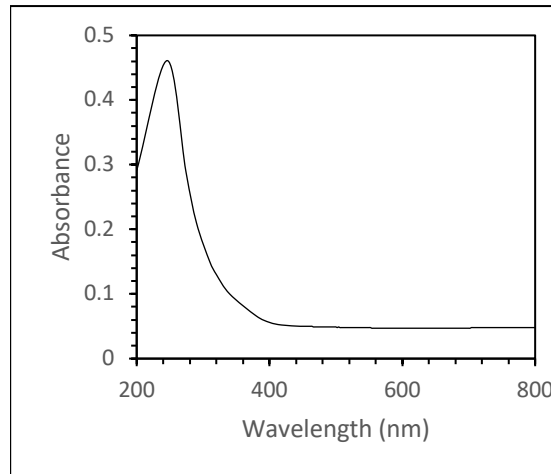


Figure 5. UV-Vis spectra of CS-SnO₂ nanocomposite

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3.1.4. UV-Vis Spectral Analysis of CS-SnO₂

Figure 5 illustrates CS-SnO₂ nanocomposite and a noticeable red shift with increased absorption in the visible range from the UV-Vis spectra. This can be due to the interaction between SnO₂ nanoparticles and functional groups of chitosan, namely –NH₂ and –OH [20]. Interaction leads to the formation of additional energy levels in the forbidden bandgap, which in turn allows for more electronic transitions. One example of such transition is $n \rightarrow \pi^*$ transition caused by lone pair of electrons from nitrogen atoms in the amino groups. Moreover, charge transfer complexes ensure better charge carrier separation [21]. The result is a reduced energy gap that allows increased absorption in the visible range. In this regard, chitosan serves as a sensitizer and an electron donor.

3.1.5. Thermogravimetric Analysis (TGA) of CS-SnO₂

The TGA–DTG analysis of the CS-SnO₂ nanocomposite

(Figure 6) reveal three major stages of thermal degradation. The first stage, from 73 to 148 °C, where the DTG maximum occurs at 92.66 °C, corresponds to the loss of physically and loosely absorbed water due to the presence of hydrophilic groups of chitosan, as has been widely described in literature [23]. The second stage is from 195 to 274 °C, where the DTG maximum occurs at 215.03 °C, corresponding to the initial decomposition of chitosan, which typically takes place at temperatures between 200 and 300 °C [24].

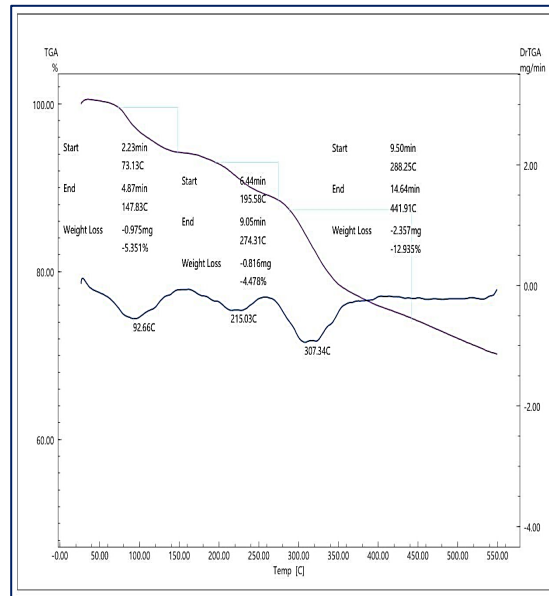


Figure 6. TGA–DTG analysis of the CS-SnO₂ nanocomposites



The third stage (288–442 °C), characterized by a DTG peak at 307.34 °C, marks the primary degradation of the polymer backbone and the formation of carbonaceous residues, suggesting a multi-step degradation process. The residual mass at higher temperatures confirms the presence of thermally stable SnO₂, thereby improving the composite's overall thermal stability [25].

3.2. Photodegradation of CV dye

Photocatalytic activity was determined based on dye CV degradation. It is affected by parameters like pH of the solution, temperature, concentration of the dye, amount of the catalyst, and illumination time, which are shown in Figure 7a-f.

3.2.1. Effect of irradiation time

The effect of irradiation time on the CV photodegradation of the CS-SnO₂ composite (10 mg L⁻¹) is demonstrated in Figure 7a. With prolonged irradiation time, the degradation efficiency increased. At 30 minutes in the dark, the degradation efficiency (D%) of 27.65% was measured, suggesting moderate adsorption. Under visible light, the efficiency increased steadily until it peaked at 180 minutes with a value of 52.89%. This result is due to the combination of CS adsorption via electrostatic forces and the photocatalytic activity of SnO₂. In the later stages, stability is achieved due to the shielding effect of the adsorbed dye and competition from intermediates for the active sites [26].

3.2.2. Effect of catalyst amount

As depicted in Figure 7(b), the percent photodegradation of CV increases as the amount of CS-SnO₂ nanocomposites increases from 40 to 60 mg. The important thing is that the percent of degradation changes from 17.05% to 66.91%. This is due to the fact that with an increase in the amount of the catalyst, the surface area of the nanocomposites increases [27].

3.2.3. Effect of CV concentrations on dye

Figure 7c shows the effect of the initial CV concentration on its photodegradation using CS-SnO₂ (60 mg) nanoparticles. As the initial CV concentration increased from 5 to 15 mg L⁻¹, the photodegradation of CV decreased from 72.89% to 36.72%. As the number of

color molecules increased, the ratio of vacant sites on the catalyst surface to color molecules decreased. Thus, at low concentrations, all CV molecules were available and accessible on the nanoparticle surface. At higher initial dye concentrations, active regions of the absorbent surfaces become saturated, and the percent photodegradation decreases [28].

3.2.4. Effect of temperature

The impact of temperature on the optical degradation of CV in 25, 35, and 45 °C was demonstrated in Figure 7d. According to the findings, as the temperature increases, the photocatalytic degradation of CV dye becomes increasingly ineffective. In other words, at 25 °C, adsorption of the dye on the catalyst is very effective. However, increasing the temperature to 35 and 45 °C causes a disturbance in the adsorption-desorption equilibrium of the dye on the catalyst surface, hence reducing the effectiveness of adsorption. Higher temperatures may cause electron-hole recombination, resulting in fewer reactive oxygen species [29].

3.2.5. Effect of the initial pH

Figure 7e demonstrates the importance of pHZPC (point of zero charge) for photocatalysts. In cases when the pH value of solution is higher than the pHZPC, the surface of catalyst will have negative charge and, therefore, there will be electrostatic attraction of positively charged ions, for example, of cationic dyes. If the pH value of the solution is less than the pHZPC, then the surface will have positive charge and there will be adsorption of anions and repulsion of cations. pHZPC was obtained via pH adjustment. It was carried out in closed Erlenmeyer flasks in 50 mL of 0.01 M NaCl solution at pH 2, 4, 6, 8, 10, and 12, respectively, prepared by 0.1 M HCl or 0.1 M NaOH. In each flask, 60 mg of catalyst was added and mixture was stirred for 48 hours [30]. Afterwards, the pH_f was measured, and $\Delta\text{pH} = \text{pH}_i - \text{pH}_f$ was calculated and plotted against the pH_i. The point of zero charge was found in the point of crossing of the curve with the X axis ($\Delta\text{pH} = 0$). For CS-SnO₂ nanocomposite the pHZPC was equal to 7.

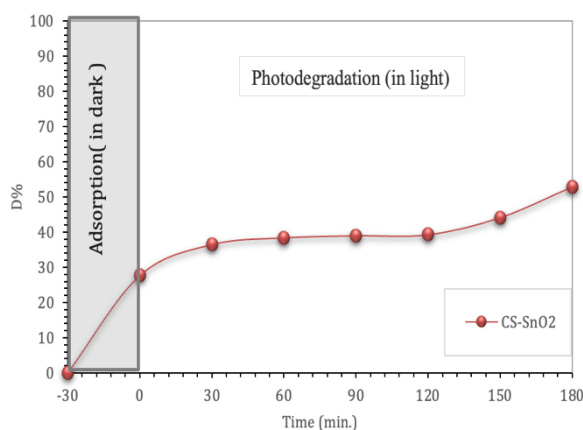


Figure 7a: Photocatalytic degradation of CV dye using CS-SnO₂ under visible-light irradiation.

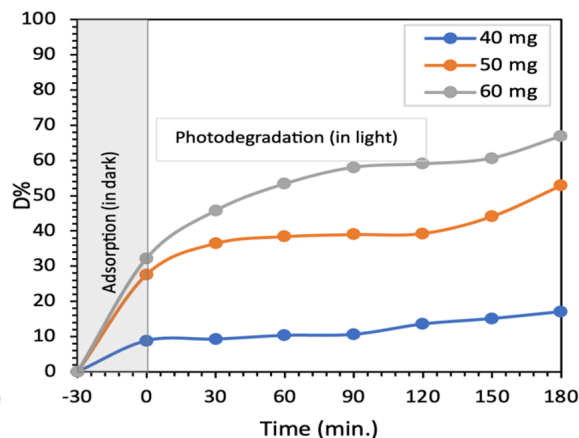


Figure 7b: The effect of catalyst weight on the photocatalytic degradation of CV dye using CS-SnO₂

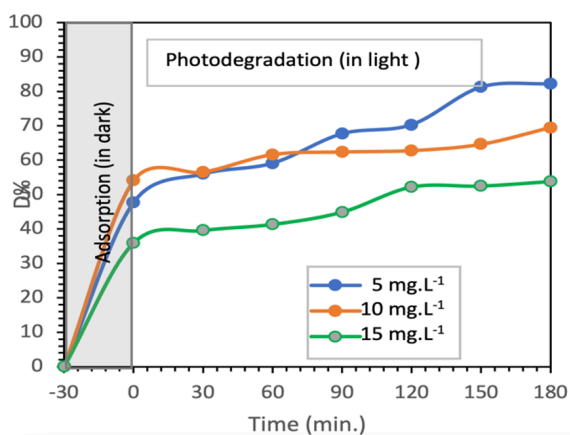


Figure 7c: The effect of CV dye concentration on photocatalytic degradation using CS-SnO₂.

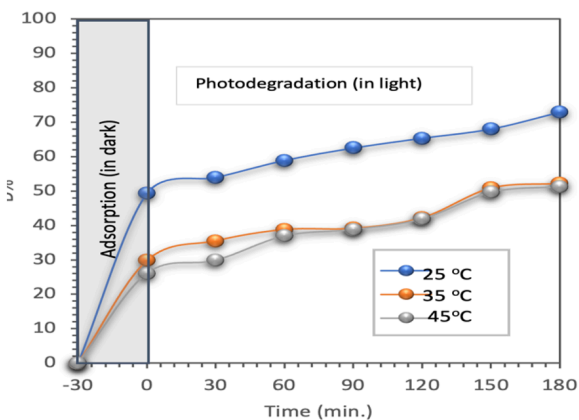


Figure 7d: Effect of temperature on the photocatalytic degradation of CV dye (5 mg L⁻¹) using CS-SnO₂ nanocomposite.

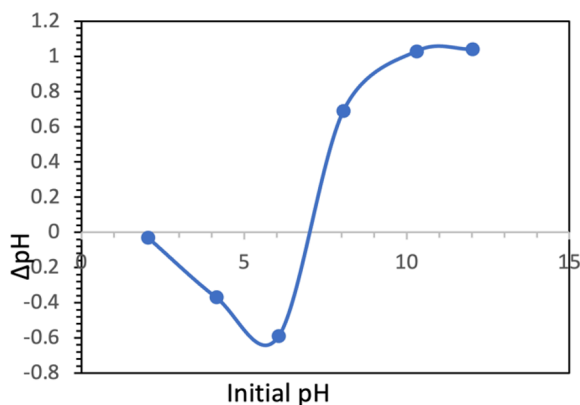


Figure 7e: Determination of the point of zero charge (pHPZC) for CS-SnO₂ using the solid addition method.

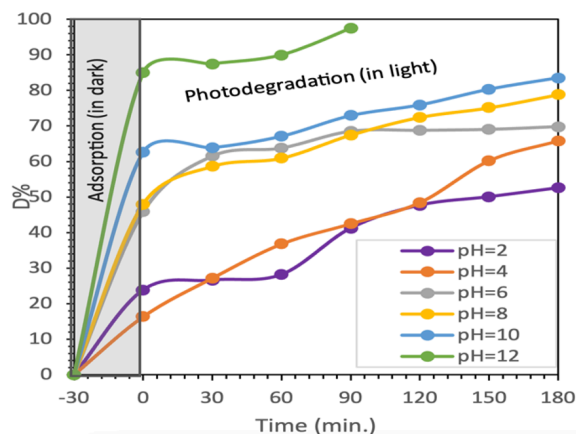


Figure 7f: Effect of pH (2-12) on photocatalytic degradation of CV dye (5 mg L⁻¹) by CS-SnO₂ nanocomposite at 25 °C

The explanation of the removal process of cationic dyes can be considered as:

For solution pH values above 7, the CS-SnO₂ nanocomposite surface mostly carries a negative charge. This leads to an enhanced attraction towards cationic (positive) dye molecules. Therefore, pH values above the pHZPC value should be considered better since the higher number of negatively charged sites enhances dye ion adsorption through electrostatic interactions and increases color removal efficiency.

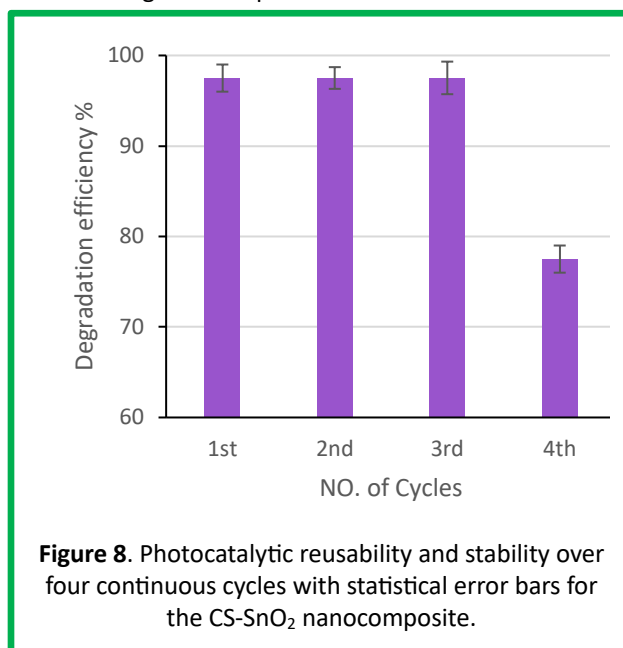
For pH values less than 7, the surface will carry a positive charge that leads to electrostatic repulsion against the cationic dye molecules. It decreases the efficiency of color removal in an acidic environment.

As seen from figure 7f, the percent photodegradation of CV increased as the pH value increased. As the pH value increased, there was dissociation of the functional groups in the adsorption surface, thus increasing electrostatic attraction between the positive dye functional groups and the negative surface groups. As a result, there was a significant increase in the percent photodegradation of the dye from 52.59% for pH = 2 to 97.5% for pH = 12 [31].

3.2.6. Recyclability test

Long-term photocatalyst efficiency is extremely important for its practical application, since it can be considered an indicator of material stability. Hence, the photodegradation efficiency of CV solution in aqueous medium with the use of the CS-SnO₂ catalyst was studied during four cycles of visible light irradiation. In every cycle, the catalyst was immediately washed with 0.01 M HCl, cleaned with distilled water and dried at 80°C for one hour. The pH value and the concentration of the CV solution were kept constant at 5 mg L⁻¹. It should be noted that the nanocomposite showed very good stability and durability, as its photocatalytic degradation efficiency was determined within four repeated cycles. In the first three cycles, the catalyst showed a very good and stable degradation performance of 97.5%, The chitosan matrix prevents aggregation of SnO₂ NPs in the catalyst, thus decreasing photocorrosion effects and maintaining the high efficiency of degradation. However,

in the fourth cycle, the catalyst showed some drop in efficiency of 77.5%, which could be caused either by catalyst weight loss during separation process, or by accumulating reaction products on active centers.



3.2.7. Kinetic Study of CV Dye Photocatalytic Degradation

In order to determine the dye decay rate due to CS-SnO₂ nanocomposite, the photocatalytic degradation results were analyzed based on the pseudo-first order kinetics, which was represented by the equation below:

$$-\ln (C/C_0) = kt \quad (2)$$

Wherein, C₀ and C are the initial and final concentration, respectively, while k is the apparent rate constant per minute [32].

The experimental data were correlated to the pseudo-first order kinetics model. The determined rate constant values decreased as the initial CV concentration increased, as presented in Table 1. This might be caused by increased occupancy of available surface sites and poor light penetration into the dye solution due to high concentration [33].

Table 1: Pseudo-first-order rate constants (k) and determination coefficients (R^2) for CV dye degradation at various concentrations

Catalyst	Initial dye Conc. (mg L^{-1})	Rate constant, (min^{-1}) k	Correlation coefficient, R^2
CS-SnO ₂	5	0.0033	0.9846
	10	0.0029	0.9341
	15	0.0016	0.8309

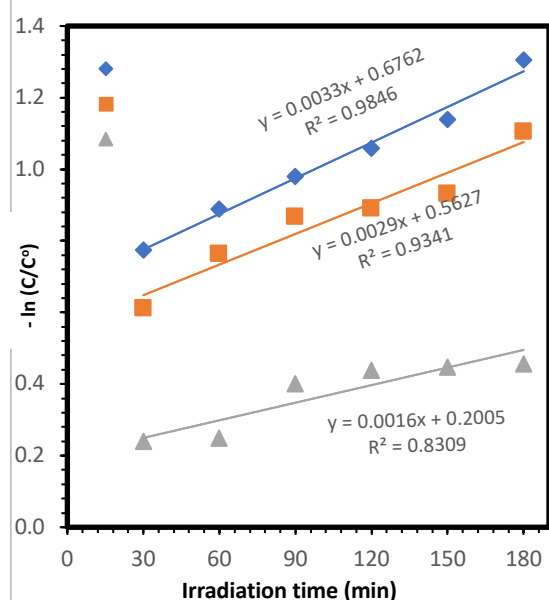


Figure 9: Pseudo-first-order kinetic plots for CV dye degradation using CS-SnO₂ nanocomposite

3.2.8. Optical Bandgap Determination via Tauc Plot Analysis

The optical band gap energy (E_g) was determined for pure SnO₂ nanoparticles and the CS-SnO₂ nanocomposites in order to investigate the effect of the incorporation of biopolymers on the electronic structure of the developed nanomaterials. The UV-Vis absorbance spectra were analyzed using Tauc's method and the formula below:

$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (3)$$

Where α is the optical absorption coefficient, A is a constant which is independent of energy, $(h\nu)$ is the photon energy and E_g is the optical band gap energy. Photon energy ($h\nu$) was calculated from the wavelength (λ) using the formula

$(h\nu) = 1240 / \lambda$. The value of E_g was determined by plotting $(\alpha h\nu)^2$ against $(h\nu)$, and by extrapolating the linear part of the graph to the point where $(\alpha h\nu)^2 = 0$.

According to Figure 10a, pristine SnO₂ NPs exhibit a broad optical bandgap of 3.65 eV,

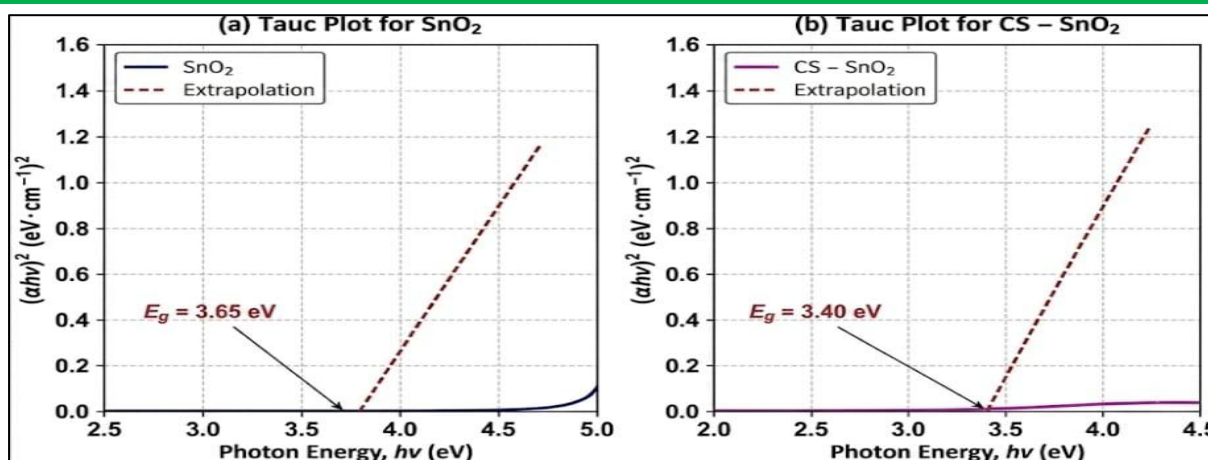


Figure 10: Tauc plots for determining the optical bandgap (E_g) of: (a) pure SnO₂ NPs, and (b) CS-SnO₂ nanocomposite.



consistent with the values obtained from other reports in literature on quantum confined tin oxide nanostructures. Upon functionalization and incorporation into the (CS) biopolymer matrix, the absorption edge was shifted towards longer wavelengths [34]. Consequently, the optical bandgap of the CS-SnO₂ nanocomposite was remarkably reduced to 3.40 eV, as shown in Figure 10b. This change from 3.65 to 3.40 eV suggests that the integration of chitosan biopolymer matrix has a significant impact on the electronic structure of SnO₂ NPs. The reason behind this could be the interaction of SnO₂ nanoparticles' surface with -OH and -NH₂ groups in the biopolymer [35]. The interaction changes the electronic environment, causing low energy transitions and hence increasing the visible light absorbance.

Conclusion

In this study, a chitosan-SnO₂ nanocomposite was prepared and characterized using FT-IR, XRD, FE-SEM/EDS, UV-Vis spectroscopy, and TGA. The characterization results support the incorporation of SnO₂ within the chitosan matrix and indicate interactions between the inorganic component and the functional groups of chitosan. The composite exhibited a combination of adsorption and visible-light-assisted activity toward Crystal Violet removal. The removal efficiency was strongly influenced by catalyst dosage, initial dye concentration, pH, temperature, and irradiation time. The highest removal efficiency of 97.5% was obtained under the specified alkaline and low-dye-concentration conditions. The kinetic analysis showed apparent pseudo-first-order behavior, while the reusability experiment demonstrated retention of activity over four cycles, accompanied by a gradual decrease in efficiency. Overall, the results indicate that the CS-SnO₂ nanocomposite has potential for dye removal from aqueous systems; however, further investigations are required to clarify the detailed photocatalytic mechanism and long-term stability of the material.

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