

RESEARCH PAPER

Pollution Control of Azo Dye–Contaminated Water Using TiO₂/CNT/ZnCo₂O₄ Nanocomposite under Visible-Light Photocatalysis

Neran A. Al Naqeeb

Department of Biology, College of Science, University of Misan, Misan, Iraq

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ABSTRACT

Industrial dye effluents are a persistent source of water pollution, threatening aquatic ecosystems and undermining sustainable development goals. This study presents the development of TiO₂/CNT/ZnCo₂O₄ nanocomposite as an environmental photocatalyst for dye-polluted water, achieved through a two-step synthesis. The incorporation of CNTs and ZnCo₂O₄ enhanced TiO₂'s visible-light photoactivity by reducing charge carrier recombination and improving electron separation. This led to a shifted absorption edge toward the visible light region and a reduced band gap of 2.41 eV, compared to pure TiO₂'s 3.28 eV. The nanocomposite demonstrated a 92% degradation of acid blue 113 (AB113) under visible light after 180 minutes. Various operational parameters, including dosage, pH, and H₂O₂ concentration, were assessed for optimal degradation. Degradation occurred via hydroxyl radicals and photogenerated holes, as identified through radical scavenging tests. Also, the synthesized nanocomposite showed great reusability for accomplishing photodegradation of the AB113 over 5 consecutive reaction cycles, supporting its reusability and pollution-control potential. Overall, the TiO₂/CNT/ZnCo₂O₄ platform couples visible-light harvesting with durable performance, highlighting a practical route toward environmental remediation of dye-laden waters because the textile dye discharges are a persistent aquatic ecosystem stressor. By shifting TiO₂ activity into the visible-light window and achieving high, reusable performance against the anionic dye AB113, the TiO₂/CNT/ZnCo₂O₄ nanocomposite provides a practical photocatalytic option for dye-polluted wastewater, aligning with sustainable pollution-mitigation goals.

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INTRODUCTION

In the 21st century, rapid industrial development has been accompanied by an alarming increase in environmental degradation, including oil-contaminated soils, plastic accumulation and heavily polluted wastewaters. To address these issues, recent studies have explored sustainable remediation strategies such

* Corresponding Author Email: neranecology@uomisan.edu.iq

as bacterial biodegradation of oil-polluted soils for pollutant abatement in support of the Sustainable Development Goals [1], integrated management and treatment of plastic waste [2], and the valorization of diatomaceous earth as a green eco-coagulant for wastewater treatment [3]. The thinning of ozone layer, destruction of natural



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habitats, deforestation, pollution of soil and water are among the most threatening consequences of human activities [4, 5]. Specially, in developing regions, millions of tons of toxic pollutions including oil and petrochemical wastes, effluents from textile dyeing plants, and residues from overuse of pesticides and fertilizers in farms are discharged in water resources without adequate treatment [6, 7]. As the result, we have witnessed a surge of severe diseases across the world [8, 9]. Pollution of water is particularly concerning due to profound coherence of life sustainability to health of oceans [10]. In this context, sheer volume and persistent nature of these pollutants make common treatment methods not insufficient [11]. Therefore, new and efficient approaches are imperative for completely removing contaminates from aquatic environments [12, 13].

Photocatalytic process is known as a great solution for environmentally friendly elimination of contaminants from water using a proper photocatalyst material and a light source with energy as high as possible to activate photocatalyst [14, 15]. Common photocatalyst materials such as TiO_2 and ZnO require structural and surface engineering to enhance their sensitivity to visible light irradiation. Besides, structural improvement can decrease the recombination rate of electrons and holes, which is generated by light-excitation of photocatalyst [11, 16]. There are many strategies to help enhance photoactivity of TiO_2 . Among these, coupling TiO_2 with other photocatalyst material has gained tremendous attention [17, 18]. Electronic transition between different energy levels of two or more photocatalysts can improve separation of charges, and improve the photocatalytic performance [19, 20]. For examples, Thuy et al. reported improved photocatalytic degradation of dyes using tertiary heterojunction of $\text{ZnO}/\text{TiO}_2/\text{WO}_3$ [21]. Ho et al. synthesized $\text{TiO}_2\text{-CeO}_2$ nanocomposite using plant extracts for photodegradation of organic dye [22]. Furthermore, carbon nanotubes (CNTs) is a great candidate for enhancing the photocatalytic activity of TiO_2 [23, 24]. It has been found that CNTs can serve as a trap for electrons, hindering their recombination with the holes [25]. Besides, incorporation of CNTs into TiO_2 structure creates new energy levels associated with $2p$ orbitals of carbon, which effectively reduces the band gap of TiO_2 [26]. In this regard, Rahbar et al. reported highly efficient photocatalytic activity for Fe, B co-

doped $\text{TiO}_2/\text{CNTs}@/\text{WO}_3$ toward mineralization of petrochemical wastewater [27].

In this work, we have synthesized $\text{TiO}_2/\text{CNTs}/\text{ZnCo}_2\text{O}_4$ nanocomposite as the viable photocatalyst under the visible light irradiation. Characterizations nanocomposite were carried out using X-ray diffraction pattern (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), diffuse reflectance (DRS) and photoluminescence (PL) spectroscopy. Also, the photocatalytic activity of the nanocomposite was assessed using visible light photodegradation of acid blue 113 (AB113). In addition to their chemical interest, visible-light-responsive photocatalysts are directly relevant to environmental pollution management. Azo dyes such as Acid Blue 113 are representative priority pollutants in textile wastewater, where color, chemical oxygen demand (COD), and potential ecotoxicity challenge conventional treatment. Embedding environmental context, this work positions $\text{TiO}_2/\text{CNT}/\text{ZnCo}_2\text{O}_4$ as a remediation material designed to harvest abundant visible photons to abate dye pollution, thereby supporting water-quality protection and sustainable environmental management.

MATERIALS AND METHODS

Synthesis of ZnCo_2O_4 nanoparticles

Co-precipitation method was employed to synthesize ZnCo_2O_4 nanoparticles. First, 1.0 g of polypropylene glycol (PPG) was dissolved into 50 mL of deionized water. Then, 1.0 mmol of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 2.0 mmol of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was added and pH of the solution was adjusted to about 9 by adding of NaOH (0.01 M). Then, the solution was stirred for 2 h. After that, the solid was separated using centrifugation at 6000 rpm for 15 min. The collected solid was washed twice with deionized water and then dried in an oven at 120°C overnight.

Synthesis of $\text{TiO}_2/\text{Carbon nanotubes}/\text{ZnCo}_2\text{O}_4$ nanocomposite (TO/CNT/ZCO)

The TO/CNT/ZCO nanocomposite was synthesized using facile and straightforward sol-gel method. To this end, first 0.01 g of CNTs were dispersed into 50 mL of absolute ethanol using ultrasonic bath. Then, 1.0 g of PPG was dissolved into the above solution. Next, 1.0 mmol of tetrabutyl orthotitanate (TBOT) was added dropwise and the solution was stirred for 1 h.

After that, 10 wt% (0.05 g) of the as-synthesized ZnCo₂O₄ nanoparticles were added and stirring was followed for 1 h. The solution was kept in an oven at 80 °C overnight to completely evaporate the ethanol solvent and obtain dried gel. Finally, dried gel was calcined under an argon atmosphere at 500 °C for 4 h.

Photocatalytic activity

The photocatalytic activity of the synthesized TO/CNT/ZCO nanocomposite was studied using photodegradation of acid blue 113 (AB113) aqueous solution under visible light irradiation. AB113 (50 mg L⁻¹) was chosen as a model anionic dye pollutant typical of textile effluents, and the pH range examined reflects environmentally relevant waters. The use of H₂O₂ serves as a green co-oxidant to enhance •OH generation within an advanced oxidation framework suitable for wastewater treatment. A constant concentration of AB113 (50 ppm) to remove the typographical carry over while keeping all results intact was used in all the conducted photocatalytic reaction.

A white color LED lamp (100 W) was used as the visible light source which was fixed at 20 cm away from the dye solution container. To reach the adsorption/desorption equilibrium between dye molecules and photocatalyst particles, the AB113 solution was stirred in the presence of specific amount of the nanocomposite for 30 min in darkness. Different dosages of the nanocomposite were dispersed into AB113 solution and the photocatalytic degradation efficiency was investigated at the constant irradiation time of 180 min. Every 30 min, the absorption of the AB113 solution was determined using UV/Vis spectrophotometer at maximum wavelength of AB113 ($\lambda_{\max} = 566$ nm). Furthermore, the effect of different parameters such as different concentrations of H₂O₂ and different pH of the AB113 solution were studied to establish the efficient utilizing of the nanocomposite. From an environmental-remediation perspective, the observed 92% reduction of AB113 absorbance under visible light evidences effective decolorization and pollutant abatement relevant

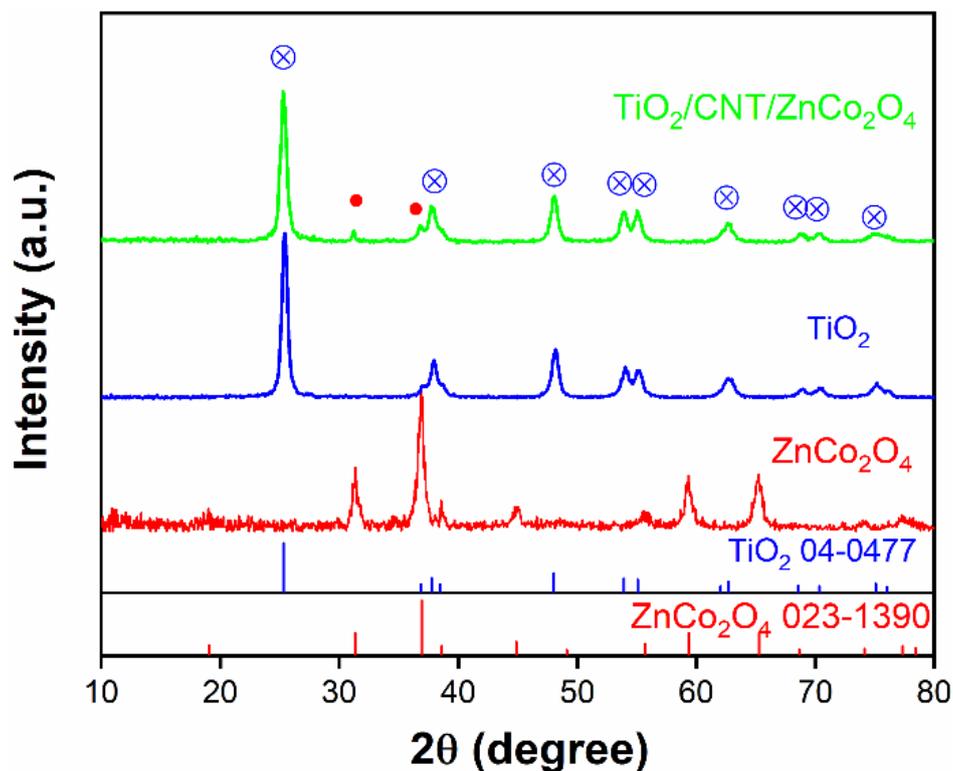


Fig. 1. XRD patterns for TiO₂, ZnCo₂O₄, and TiO₂/CNT/ZnCo₂O₄.

to water-pollution control. The marked kinetic enhancement ($k \approx 0.403 \text{ min}^{-1}$ vs 0.058 min^{-1} for TiO_2) indicates more efficient use of benign

light sources, lowering potential energy demand in treatment scenarios. The demonstrated reusability over five cycles further supports the

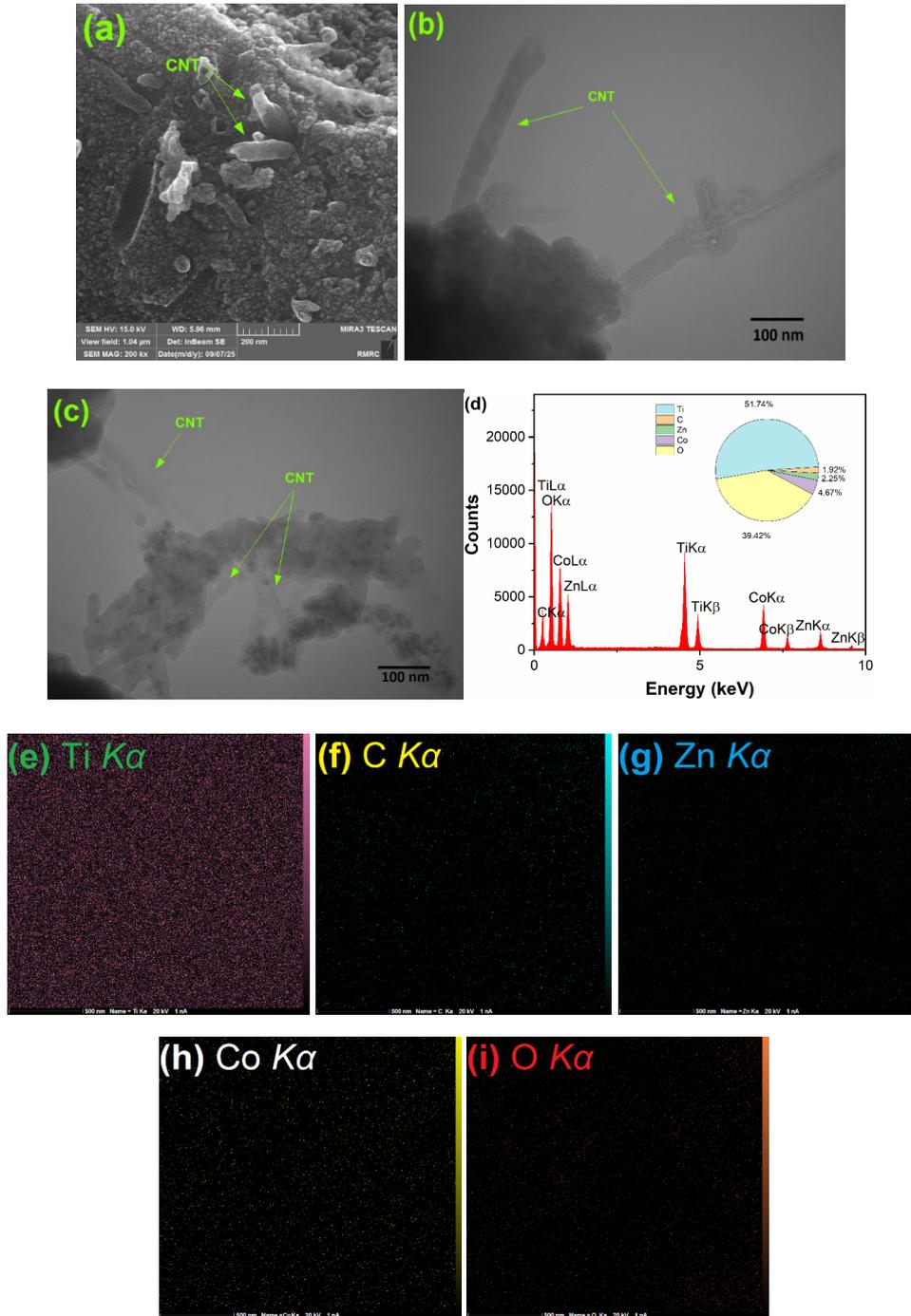


Fig. 2. FE-SEM image (a), TEM images (b, c), EDX spectrum (d), and EDX-mapping images (e: Ti, f: C, g: Zn, h: Co, and i: O) for $\text{TiO}_2/\text{CNT}/\text{ZnCo}_2\text{O}_4$ nanocomposite.

material's applicability to sustainable wastewater operations.

Characterization

Phase and composition of the nanocomposite were studied using X-ray diffraction (XRD) pattern by Philips X'pert Pro MPD diffractometer (Cu K α = 1.54 Å). The Morphology and structure of the nanocomposite were studied using transmission electron microscopy (TEM) (Carl Zeiss) and field emission scanning electron microscopy (FE-SEM) (TESCAN Mira 3). Optical properties of the nanocomposite were investigated using diffuse reflectance (DRS) (JASCO UV-Vis spectrophotometer) and photoluminescence (PL) (Perkin Elmer LS 55) spectroscopy.

RESULTS AND DISCUSSION

XRD patterns

Fig. 1 shows the XRD patterns of the TiO₂, ZnCo₂O₄, and TiO₂/CNT/ZnCo₂O₄ nanocomposite. As shown, the diffraction patterns of TiO₂ and ZnCo₂O₄ exhibit the planes which are in good agreement with anatase phase of titanium dioxide (JCPDS. File no. 04-0477) and cubic phase of zinc cobalt oxide (JCPDS file no. 023-1390). As for the nanocomposite, the peaks at 2 θ = 25.3°, 37.7°, 48.1°, 53.9°, 55.1°, 62.1°, 62.7°, 68.5°, 70.3°, 75.1°, and 76.1° (marked with \otimes) are attributed to the TiO₂ phase and diffraction peaks at 2 θ = 31.2° and 36.8° (marked with \bullet) are assigned to the ZnCo₂O₄ phase. The (002) characteristic reflection of carbon (JCPDS file no. 008-0415) associated with the presence CNTs is not observed in the XRD pattern

of the nanocomposite. This absence is attributed to the overlap with the strong diffraction peak of TiO₂ at 25.3°.

TEM and FE-SEM images

Fig. 2a shows the FE-SEM image for the synthesized TO/CNT/ZCO nanocomposite. As can be seen, the metal oxide nanoparticles are aggregated around strands of the CNTs. The TEM images (Fig. 2b and 2c) clearly exhibit the internal structure of the nanocomposite, revealing that the CNTs are surrounded by TiO₂ and ZnCo₂O₄ nanoparticles.

The EDX spectrum of the nanocomposite is shown in Fig. 2d, which confirms the presence of the constituents of the nanocomposite with relative amounts (wt%) of Ti (51.74%), Zn (2.25%), Co (4.67%), C (1.92%), and O (39.42%). Additionally, the EDX mapping images in Fig. 2e-i show the uniform distribution of the components throughout the nanocomposite.

DRS and PL spectra

To verify the positive effect of the CNTs and ZnCo₂O₄ incorporation on enhancing the visible-light absorption ability of the TiO₂, the DRS analysis was carried out, which is shown in Fig. 3a. The pure TiO₂ shows the insignificant absorption in the visible light region, implying that the synthesized pure TiO₂ is unable to serve as the efficient visible-light photocatalyst. However, the incorporation of the CNTs and ZnCo₂O₄ nanoparticles increased the absorption of the light in the range of 400-700 nm. Also, the inset to the Fig. 3a represents

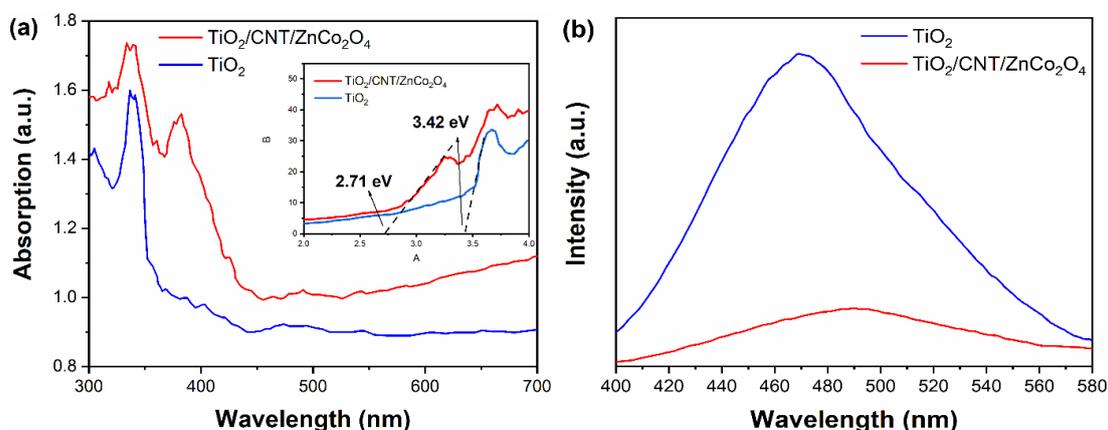


Fig. 3. DRS (a) and PL (b) spectra for the synthesized TO/CNT/ZCO nanocomposite and TiO₂. Inset to the Fig. 3a shows Tauc plots for determination band gap of the samples.

the Tauc plots for the pure TiO₂ and TO/CNT/ZCO nanocomposite, which confirmed that the synthesized nanocomposite possesses the narrower band gap compared to the pure TiO₂. The determined band gap values are 2.71 and 3.42 eV for the TO/CNT/ZCO nanocomposite and pure TiO₂, respectively. In addition, the recombination rate of the photo-generated electrons and holes was assessed for the TO/CNT/ZCO nanocomposite with respect to the pure TiO₂. For this purpose, PL analysis of the samples were recorded at excitation wavelength of 300 nm. As shown in Fig. 3b, the pure TiO₂ shows the emission peak at 470 nm, whose intensity decreases as the result of the CNTs and ZnCo₂O₄ incorporation. Also, the emission peak of the nanocomposite is slightly red-shifted compared to that of the pure TiO₂. These observations disclose that the electron transitions between TiO₂ and ZnCo₂O₄ nanoparticles patently reduce the recombination rate of the charges [28]. Moreover, the CNTs capture the electrons, thereby further improving the separation the charges [26, 29]. Concurrently, the formation of Ti-O-C bonds help to reduce the band gap and shift the edge of absorption toward the visible light region [27, 30, 31].

Photocatalytic activity

The photocatalytic activity of the synthesized TO/CNT/ZCO nanocomposite was investigated for degradation of the AB113 solution. Fig. 4a shows the photocatalytic degradation of the AB113 using the nanocomposite and pure TiO₂. As can be seen from Fig. 4a, the AB113 solution was degraded by 24% using the pure TiO₂, whereas the

TO/CNT/ZCO nanocomposite provided the 92% of the photocatalytic degradation of the AB113 solution. Also, the degradation of the AB113 is negligible without addition of the nanocomposite, confirming that the AB113 is not degraded under no-catalyst condition. Moreover, in the absence of light beams (no-light condition), the degradation level was trivial. The kinetics of the photocatalytic degradation using the synthesized nanocomposite was also studied, shown in Fig. 4b. It was found that the kinetics of the AB113 degradation reaction complied with the first order reaction: $-\ln(C/C_0) = Kt$; C and C_0 are the concentration of dye solution after and before the photocatalytic reaction, K is the rate constant, and t is the irradiation time. Fig. 4b shows the plot of $-\ln(C/C_0)$ versus irradiation time then the rate constant (K) was determined from the slop the curve. The rate constant was determined to be 0.403 and 0.058 min⁻¹ for the TO/CNT/ZCO nanocomposite and pure TiO₂, respectively.

Effect of different determinants

Effect of photocatalyst amount

Because real effluents vary in solids content, pH, and oxidant levels, evaluating catalyst dosage, solution acidity, radical scavengers, and H₂O₂ concentration provides process knobs to tailor the photocatalyst for environmental treatment trains. The different amounts of the TO/CNT/ZCO nanocomposite were loaded into the AB113 solution and then photocatalytic degradation was studied under the similar irradiation time (180 min). Fig. 5a represents that the AB113 photodegradation decreased with increasing

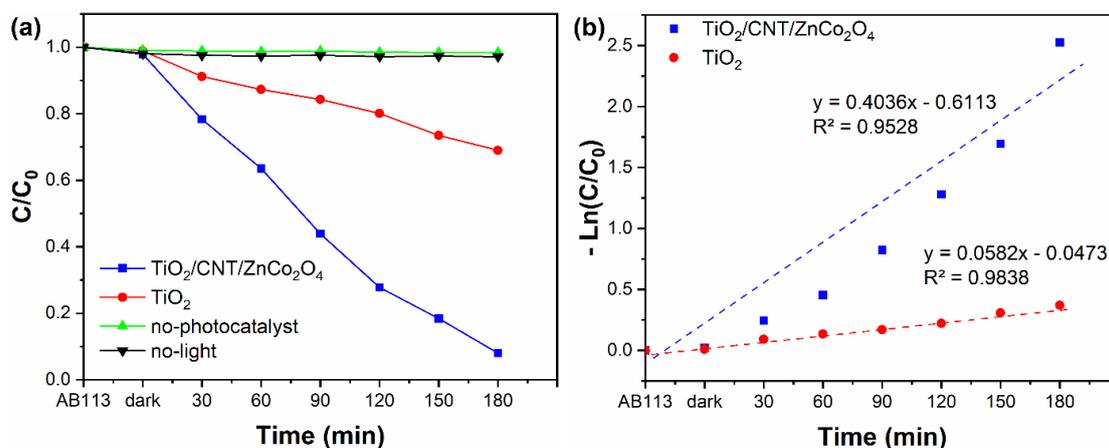


Fig. 4. Photocatalytic efficiency of the TiO₂/CNT/ZnCo₂O₄ vs. TiO₂ (a) and kinetics curves (b) for visible-light degradation of AB113.



amount of the nanocomposite. The maximum photocatalytic efficiency was attained by 0.04 g of the synthesized nanocomposite. Further amounts led to decrease the photodegradation level of the AB113 solution. It has been confirmed that the excess amount of photocatalyst limits the transmittance of the light beam through the dye solution, resulting in the reduced activation of photocatalyst and photocatalytic activity [32].

Effect of pH

Besides, the effect of pH of the AB113 solution was investigated on the photocatalytic efficiency of the synthesized nanocomposite. As shown in Fig. 5b, the AB113 solution is prone to the higher level of the photodegradation under acidic condition compared to alkaline solution. This is because AB113 is an anionic dye [33], which has the affinity to adsorb on the positively charged surface of

photocatalysts under acidic conditions. Therefore, more than 96% of the AB113 solution was degraded using the TO/CNT/ZCO nanocomposite at pH of 5. However, the degradation efficiency decreased to 72% by increasing the pH at 9.

Effect of radical scavenger agents

The photocatalytic degradation of the AB113 solution was investigated using various radical scavenger agents. In this regard, isopropyl alcohol [34], ethylenediaminetetraacetic acid (EDTA) [35], and L-ascorbic acid [36] were used as scavenging agent for hydroxyl radical (•OH), photo-generated holes (h⁺), and superoxide radical (•O₂⁻), respectively. Fig. 5c exhibits that the photodegradation level of the AB113 dramatically decreased in the presence of EDTA (2 mM) and isopropyl alcohol (2 mM), revealing that the photo-generated holes and •OH radicals are the oxidative

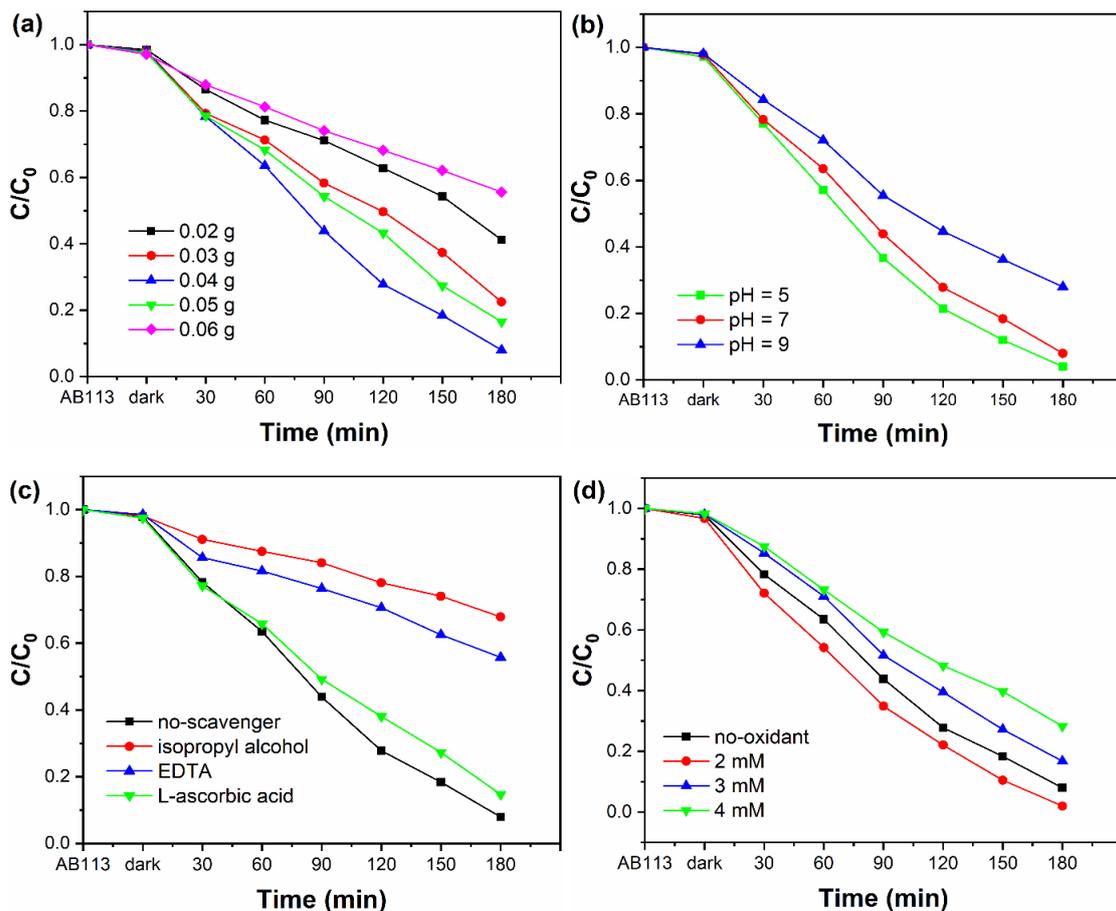


Fig. 5. Effects of photocatalyst amount (a), pH of AB113 (b), radical scavenger agent (c), and concentration of H₂O₂ (d) on degradation of AB113 using TiO₂/CNT/ZnCo₂O₄ nanocomposite.



species which degraded AB113 molecules during photo-excitation of the nanocomposite. In contrast, addition of ascorbic acid (2 mM) has no significant effect on the photocatalytic efficiency of the nanocomposite.

Effect of H₂O₂ concentration

Moreover, the effect of H₂O₂ as the oxidant agent was studied on the photocatalytic activity of the synthesized TO/CNT/ZCO nanocomposite. Fig. 5d shows the degradation extent of the AB113 increases to 98% by adding 1 mL of 2 mM H₂O₂. However, the addition of more concentrated H₂O₂ solution, 3 mM and 4 mM, led to decrement of the photo-degradation of AB113. It can be concluded that the moderate concentration of H₂O₂ promotes the formation of •OH radicals. On the other hand, excessive concentrations of H₂O₂ impedes the •OH radicals to contribute in the degradation reaction. It has been suggested that the more concentration of H₂O₂ reacts with •OH radicals to produce water molecules [37].

Reusability experiments

To study the reusability properties of the nanocomposite, the photocatalytic degradation was conducted at multiple reactions. Fig. 6 shows

the reusability properties of the nanocomposite at 5 consecutive reaction cycles. As shown, the TO/CNT/ZCO nanocomposite possesses the great reusability, so that the photodegradation of the AB113 was decreased by 4.16% after 5th photocatalytic reaction. This result clearly revealed the prominent structural stability. Maintaining activity over successive runs limits secondary solid waste from frequent catalyst replacement, an important consideration for environmental sustainability in full-scale pollution-control systems. At the regional scale, robust and reusable photocatalysts are particularly important in light of recent evidence on environmental stressors impacting both agroecosystems and food safety. Water salinity markedly suppresses germination some plants [38], water parasitological contamination that directly endangers consumers [39], and air pollutants induce characteristic morphological injuries in plant leaves, revealing the broader ecological burden of atmospheric contamination [40]. These issues underline that advanced water-treatment strategies such as the TiO₂/CNT/ZnCo₂O₄ system developed here can contribute to protecting crop productivity, securing the food chain and limiting human exposure to environmental pollutants.

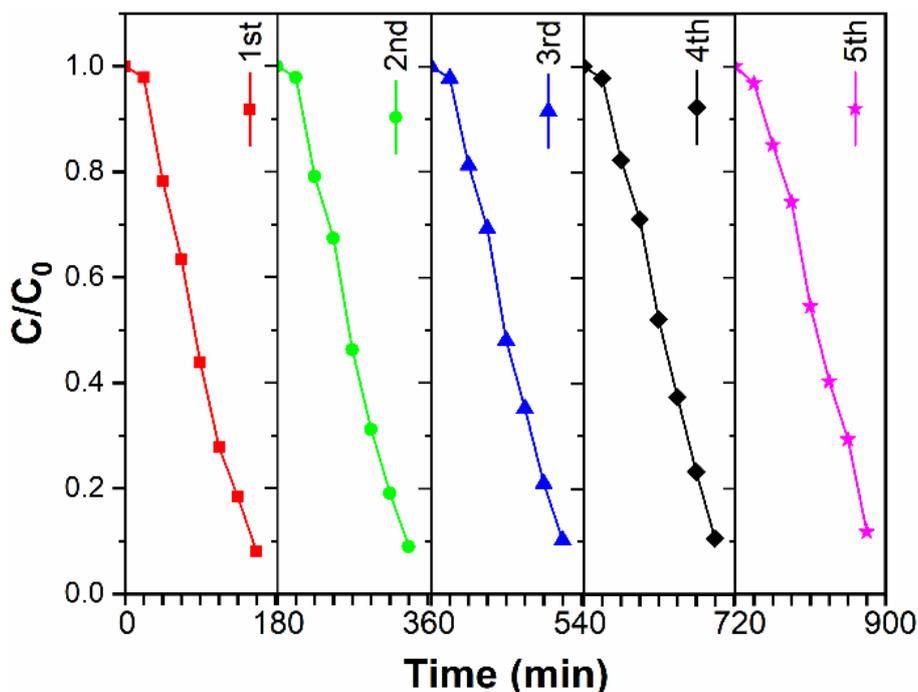


Fig. 6. Reusability tests for photodegradation of AB113 using the TiO₂/CNT/ZnCo₂O₄ nanocomposite.

Beyond material innovation, integrating artificial intelligence (AI) into water-treatment strategies offers a promising pathway for smarter pollution control. Recent reviews highlight the role of AI in predicting aquatic pollution trends and optimizing operational parameters. Coupling AI-based predictive tools with advanced photocatalysts such as the TiO₂/CNT/ZnCo₂O₄ system developed here could enhance efficiency, reduce resource consumption, and support adaptive, sustainable wastewater management [41].

CONCLUSION

We report a TiO₂/CNT/ZnCo₂O₄ nanocomposite engineered for visible-light environmental photocatalysis targeting dye-polluted water. The incorporation of CNT and ZnCo₂O₄ into TiO₂ nanostructure exhibited the undeniable enhancement of the visible-light harvesting. The band gap of the nanocomposite was calculated to be 2.71 eV, which is significantly lower than that of the pure TiO₂ (3.42 eV). The photocatalytic performance of the synthesized nanocomposite was evaluated by degradation of acid blue 113 aqueous solution. Photo-activation of the nanocomposite took place by the visible light illumination for 180 min. After this time, it was revealed that the AB113 solution was degraded more than 92%, whereas the synthesized pure TiO₂ showed about 24% photoactivity. It was suggested that the photogenerated electrons can be prevented to recombine with the holes through migration to the coupled ZnCo₂O₄ nanoparticles. Additionally, the electrons captured by the CNTs, thereby the photoactivity further improved to attain maximum degradation level of AB113 solution. Optimum amount of the loaded nanocomposite (0.04 g) and other experimental conditions, including pH of AB113 (~ 5), and H₂O₂ concentration (1mL of 2mM), were found via conducting the photocatalytic reactions under different operational parameters. Furthermore, it was confirmed that the photo-generated holes and hydroxyl radicals play pivotal role in degrading the AB113 solution. Besides, the synthesized nanocomposite was collected and reused over 5 reaction cycles, showing the significant reusability of the nanocomposite without noticeable loss of its photo-efficiency. Overall, the material's high visible-light activity, operational tunability (pH, H₂O₂, dosage), and five-cycle durability highlight its promise as a practical component

in environmental remediation and wastewater-pollution control workflows.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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