

# Improving Visible Light Driven Photocatalytic Performance of $\text{ZnWO}_4/\text{g-C}_3\text{N}_4$ Nanocomposites for RhB Dye Degradation

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**Abstract**—This study attempts to break down Rhodamine dye with  $\text{ZnWO}_4/\text{CN}$  nanocomposites for environmental use. The nanocomposites were made using a simple hydrothermal process and examined for optical, structural, and morphological properties. XPS, SEM-EDX, UV-DRS, and PL are examples. RhB dye degradation in water was employed to test photocatalytic activity. The study found that  $\text{ZnWO}_4@0.5\%$  CN has superior photocatalytic performance compared to pure  $\text{ZnWO}_4$ ,  $\text{ZnWO}_4@0.1\%$  CN, and  $\text{ZnWO}_4@1.0\%$  CN nanocomposites. The synergistic effect results from efficient photoinduced electron-hole pair separation and transfer. As an excellent electron conductor, activated carbon increases charge separation and extends charge carrier life. Photocatalytic experiments indicated  $\text{ZnWO}_4@0.5\%$  CN nanocomposites destroyed 97% RhB dye in 150 minutes. Quenching investigations confirmed that the degradation kinetics followed a pseudo-first-order model, indicating that reactive oxygen species (ROS) formed efficiently and abundantly caused dye degradation.

**Keywords**—Reactive oxygen species, Kinetics, Charge separation, Synergistic effect, Rhodamine dye

## I. INTRODUCTION

Industrialization and population growth have contributed to environmental pollution and energy shortages caused by organic pollutants, which have become major barriers to economic and social progress [1, 2]. The adoption of pollution-free technologies and the identification of clean energy sources are crucial for achieving sustainable development [3, 4]. In contrast, organic dyes used widely in sectors like food, cosmetics, and paint industries are thought of as a significant source of water pollution [5]. Rhodamine B (RhB) is a yellow anthracene dye that is very soluble, has excellent brightness, and finds widespread use in biological applications and industrial production processes [6, 7]. Several health problems, including nausea, skin irritation, and vomiting, can be caused by exposure to high concentrations of RhB [8, 9]. Chemically durable, hardly biodegradable, and extremely toxic even at low

concentrations—RhB is a synthetic organic dye. They are extremely dangerous for people and the environment [10]. So, to degrade organic contaminants, we need an effective, inexpensive method. Due to its capacity to fully mineralize the organic contaminants, semiconductor photocatalyst have garnered tremendous interest among researchers [11]. A wide band gap and a fast electron-hole (e-h) pair recombination rate are two limitations of the widely used semiconductor photocatalyst that contribute to its poor efficiency in converting light into energy [12]. Developing photocatalytic technology currently hinges on perfecting the synthesis of photocatalysts, which must possess attributes including low production costs, wide light absorption ranges, high cycle stability, and outstanding catalytic activity [13]. Consequently, it is of the utmost importance to develop an environmentally friendly and exceptionally effective photocatalyst through the use of standard techniques such as elemental doping, morphological control, and the creation of an appropriate heterojunction structure. A number of parameters determine how well they work, including dimensions, surface charge, shape, thermal characteristics, and optical characteristics [14]. Since narrow band gap oxides absorb a greater amount of visible light and are thought to be better photocatalysts owing to their wide solar light area, researchers are concentrating on employing these materials for photocatalysis [15]. Zinc tungstate ( $\text{ZnWO}_4$ ) stands out among narrow band gap semiconductors due to its exceptional optical, electrical, structural, and physiochemical characteristics; these qualities make it a great candidate for use in composites and open up new avenues for environmental applications [16]. Research on  $\text{ZnWO}_4$ , a wolframite tungstate metal oxide with a  $d10s2-d10$  electronic configuration, has been conducted because of its potential uses in water splitting and the breakdown of organic pollutants in water when exposed to UV light [17, 18]. The photocatalytic activity of  $\text{ZnWO}_4$  is inefficient because it does not absorb visible light [19]. There have been numerous

attempts to increase  $\text{ZnWO}_4$ s photocatalytic activity by harvesting visible light [20, 21].

Under visible light irradiation,  $\text{g-C}_3\text{N}_4$  may efficiently catalyze photochemical processes because to its low bandgap of about 2.8 eV [22, 23]. Attaching Co nanoclusters to  $\text{g-C}_3\text{N}_4$  allowed Shang et al. to investigate their potential for  $\text{CO}_2$  reduction with great success [24, 25]. By adding nitrogen vacancies to  $\text{g-C}_3\text{N}_4$ , Dong et al. were able to attain high activity in photocatalytic nitrogen fixation [26]. The issue that single-component  $\text{ZnWO}_4$  can only use ultraviolet energy is solved when  $\text{g-C}_3\text{N}_4$  and  $\text{ZnWO}_4$  are combined because the light absorption range of  $\text{ZnWO}_4$  is broadened. And since their energy band structures are so similar,  $\text{g-C}_3\text{N}_4$  is an excellent material for creating heterojunctions with  $\text{ZnWO}_4$  [27]. The photogenerated carriers will be efficiently segregated and transported under the action of an internal electric field that forms at the composite interface after the composition of  $\text{g-C}_3\text{N}_4$  and  $\text{ZnWO}_4$  [28]. Furthermore, a great deal of reducing gases, including  $\text{NH}_3$ , are produced during the annealing process in a controlled atmosphere when  $\text{g-C}_3\text{N}_4$  is synthesized using melamine as the precursor. There are a lot of oxygen vacancies in this atmosphere because the oxidation status of W atoms fluctuates [29]. These vacancies can protonate water molecules. Consequently, the combination of  $\text{g-C}_3\text{N}_4$  and  $\text{ZnWO}_4$  is able to facilitate effective deprotonation of water molecules, increase the separation of photogenerated carriers, and prolong the light absorption of single-component  $\text{ZnWO}_4$  [30].

At this time Zinc oxide/graphene oxide heterojunction nanofibers for highly effective photocatalytic nitrogen fixation: Deprotonation of water molecules and photogenerated electrons work in tandem to produce a synergistic impact.

Our goal is to create a composite material by combining  $\text{ZnWO}_4$  with two-dimensional graphitic carbon nitrate using a hydrothermal process. This approach is heavily influenced by the literature stated earlier. Various analytical techniques were used to thoroughly investigate the proposed catalyst  $\text{ZnWO}_4/\text{g-C}_3\text{N}_4$ . Under visible light irradiation, our catalyst  $\text{ZnWO}_4/\text{g-C}_3\text{N}_4$  continuously degraded 97% RhB dye in 150 minutes. The purpose of the scavenger trapping study was to determine whether reactive species were involved in the degradation of the dye. The  $\text{ZnWO}_4/\text{g-C}_3\text{N}_4$  photocatalyst is highly effective in degrading RhB dye in water samples, thanks to its high catalytic efficiency.

## II. XRD ANALYSIS

The 2theta range of  $5^\circ$  to  $80^\circ$  was used to record the XRD profiles of the nanocomposites in figure 1. These profiles include  $\text{ZnWO}_4$ ,  $\text{g-C}_3\text{N}_4$ ,  $\text{ZnWO}_4@0.1\%\text{CN}$ ,  $\text{ZnWO}_4@0.5\%\text{CN}$ , and  $\text{ZnWO}_4@1.0\%\text{CN}$ . On planes (100) and (002), respectively, the two diffraction patterns of graphitic carbon nitrate are located at  $13^\circ$  and  $27.7^\circ$ . The JCPDS (03-0401) was entirely in agreement with it [13]. One phase of monoclinic wolframite from the JCPDS (15-0774) has the sharp diffraction peaks of  $\text{ZnWO}_4$  [23]. The diffraction pattern of the nanocomposite made of  $\text{ZnWO}_4$  at different mass ratios (0.10% CN, 0.5% CN, and 1.0% CN) shows peaks for  $\text{ZnWO}_4$ , whereas the planes (100), (002), that belong to graphitic carbon nitrate, are absent. We validate the  $\text{g-C}_3\text{N}_4$  interacts with composite material production in subsequent characterizations.

The broadening of the diffraction peaks, caused by loading activated carbon with  $\text{ZnWO}_4$  at concentrations of 0.1%, 0.5%, and 1%, reveals the interaction of graphitic carbon nitrate with  $\text{ZnWO}_4$ . The two phases and high crystallinity of  $\text{ZnWO}_4@CN$  nanocomposites were shown by the conspicuous and well-reflected images. Additionally, the absence of any other peaks that are not shown serves as confirmation of the processed materials' purity.

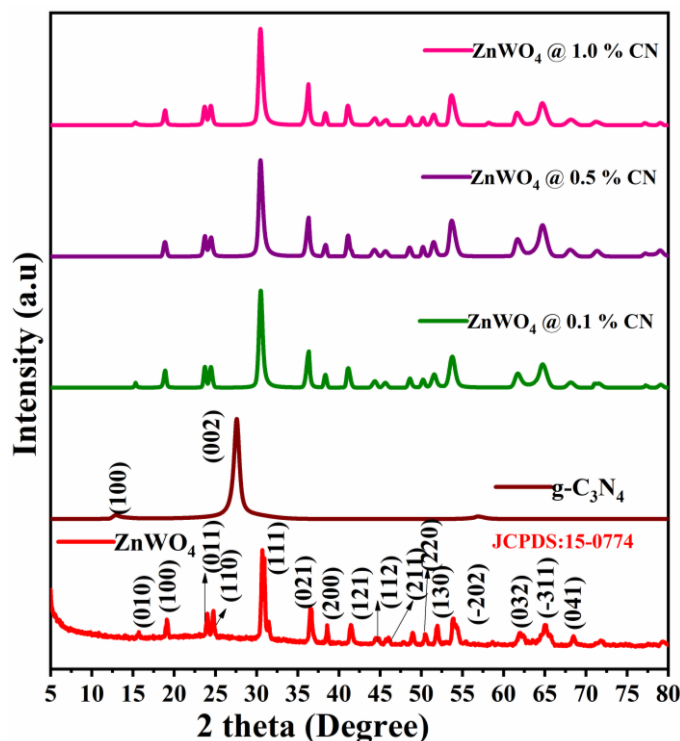


Figure 1. XRD diffraction pattern of  $\text{ZnWO}_4$ ,  $\text{g-C}_3\text{N}_4$ ,  $\text{ZnWO}_4@0.1\%\text{CN}$ ,  $\text{ZnWO}_4@0.5\%\text{CN}$ ,  $\text{ZnWO}_4@1.0\%\text{CN}$  nanocomposite

## III. XPS ANALYSIS

The elemental composition and surface chemical bonding of the as-prepared  $\text{ZnWO}_4@1.0\%\text{AC}$  crystalline material were shown by the XPS spectra. The survey spectra of the  $\text{ZnWO}_4@1.0\%\text{CN}$  nanocomposite are shown in Figure 2(a), which validates the presence of carbon, oxygen, zinc, and tungstate. The Zn 2p deconvoluted spectra are shown in Figure 2(b). Zn 2p<sub>3/2</sub> and Zn 2p<sub>1/2</sub> belong to the XPS peaks at 1044.5 eV and 1021.5 eV, respectively, according to references [31]. The two main peaks observed in the O1s XPS spectrum at 531.4 eV and 530.4 eV, respectively, are hydroxyl groups, Zn-O-C links between graphitic carbon nitrogen surfaces and  $\text{ZnWO}_4$ , and metal-oxygen-metal bonds, as shown in Figure 2(c) [32]. The high-resolution XPS doublet peaks, which are assigned to W(VI) in WO and can be seen in Figure 3(d) at around 37.1 eV for W 4f 5/2 and 35.2 eV for W 4f 7/2, are presented. At the binding energies of 398.2eV, 399.4 eV, and 400.5 eV, the trinity peaks of N1s are situated [33]. The sp<sup>2</sup> hybridized aromatic nitrogen atom bound into carbon (C=N-C) and characterized as Pyrrolic -N is represented by the first peak. When the tertiary carbon atom forms a bond with the nitrogen atom, as shown by the second peak, the bond is N-

(C)3 [34]. In Figure 3(e), the N-H groups in the graphitic carbon nitrate are shown by the third peak. 70 and 71Two prominent XPS peaks, C=O and C-C, having binding energies of 286.5 eV and 284.6 eV, respectively, make up a C1s spectra shown in Figure 3(f) [35]. In addition, our XPS study verifies that the produced  $\text{ZnWO}_4@1.0\%$  CN nanocomposite interacts with graphitic carbon nitrogen surfaces.

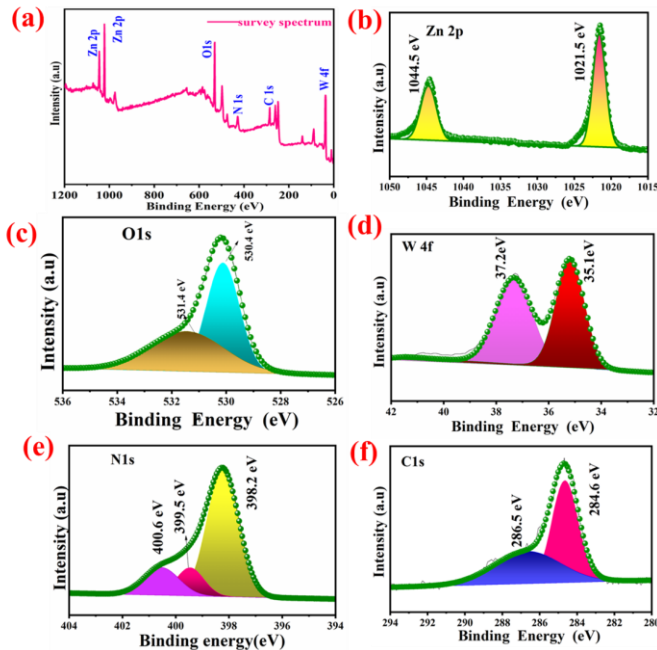


Figure 2. (a) Survey spectra (b-e) Deconvoluted spectra of Zn 2p, O 1s, W 4f, N 1s, C 1s of  $\text{ZnWO}_4@0.5\%$  CN nanocomposite.

#### IV. UV-DRS

The optical features of the  $\text{ZnWO}_4$ ,  $\text{ZnWO}_4@0.1\%$  CN,  $\text{ZnWO}_4@0.5\%$  CN, and  $\text{ZnWO}_4@1.0\%$  CN nanocomposites were analyzed using UV-visible absorption spectroscopy. The absorption edge of  $\text{ZnWO}_4$ ,  $\text{ZnWO}_4@0.1\%$  CN,  $\text{ZnWO}_4@0.5\%$  CN, and  $\text{ZnWO}_4@1.0\%$  CN nanocomposites is observed at 355 nm, 400 nm, 483 nm, 417 nm respectively. Figure 3(a, c, e, g) shows the absorption spectra of the photocatalyst. Furthermore, the band-gap values were determined utilizing Tauc's plot. The band-gap value for  $\text{ZnWO}_4$  is 3.5 eV, while the values for  $\text{ZnWO}_4@0.1\%$  CN,  $\text{ZnWO}_4@0.5\%$  CN, and  $\text{ZnWO}_4@1.0\%$  CN nanocomposites are 3.7 eV, 2.72 eV, 2.6 eV, 2.82 eV correspondingly [43,44]. The coupling of g-C<sub>3</sub>N<sub>4</sub> diminishes the band gap of  $\text{ZnWO}_4$  yet enhances the visible light absorption of the  $\text{ZnWO}_4@0.5\%$  CN nanocomposite which is shown in figure 3(b, d, f, h). The interaction contact between g-C<sub>3</sub>N<sub>4</sub> and  $\text{ZnWO}_4$  facilitated the frequent and efficient separation of electrons and holes, attributed to the pronounced optical absorption of the  $\text{ZnWO}_4@0.5\%$  CN nanocomposite. This leads to enhanced photocatalytic activity and more effective use of the solar spectrum's increased visible light absorption [35].

#### V. SURFACE AREA ANALYSIS

$\text{N}_2$  adsorption-desorption experiments facilitated the analysis of the pore structure and specific surface area of the  $\text{ZnWO}_4@0.5\%$  CN nanocomposites. Figure 4(a-b) illustrates the pore size distributions and  $\text{N}_2$  adsorption-desorption isotherms of the synthesized photocatalysts. Figure 5a indicates that the samples exhibit  $\text{ZnWO}_4@0.5\%$  CN nanocomposites characteristics of a typical type-IV isotherm. Simultaneously, under IUPAC classifications, the materials exhibit mesoporous structures, evidenced by the isotherm displaying an H3-type hysteresis loop [36]. Moreover, multi-layer adsorption may transpire with increased pressure, while single-molecule

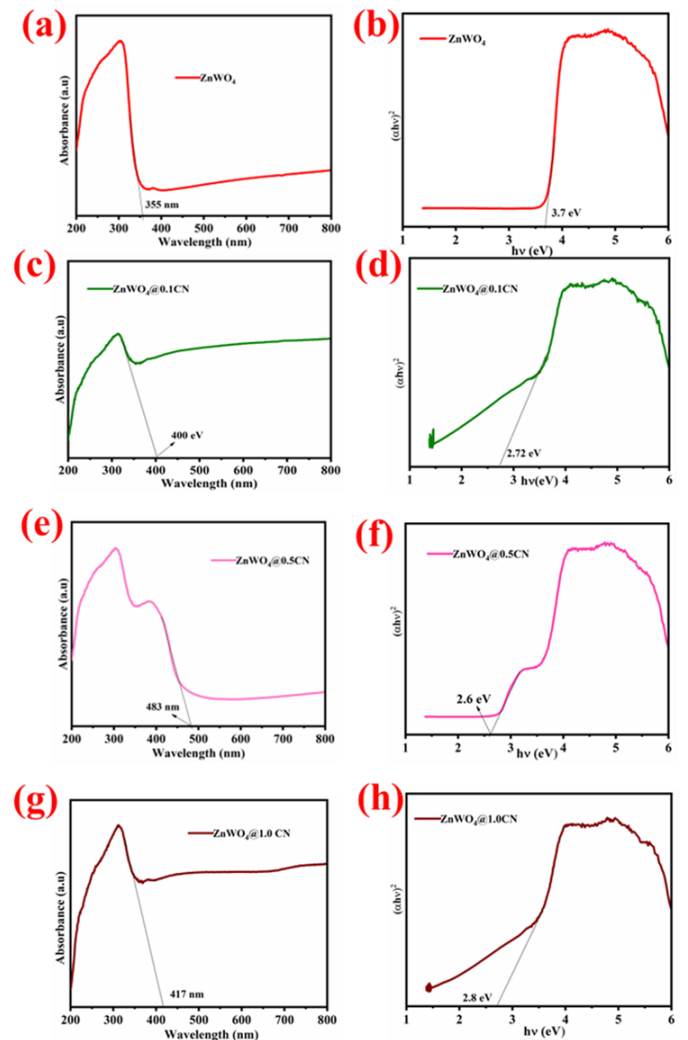


Figure 3. (a-h) UV-DRS and Tauc plot of  $\text{ZnWO}_4$ ,  $\text{ZnWO}_4@0.1\%$  CN,  $\text{ZnWO}_4@0.5\%$  CN,  $\text{ZnWO}_4@1.0\%$  CN nanocomposite

adsorption occurs at lower relative pressures. Additionally, the isotherm-derived pore diameter, pore volume, and specific surface area are included. Research indicates that the  $\text{ZnWO}_4@0.5\%$  CN nanocomposite possesses a specific surface area of 94.956 m<sup>2</sup>/g, with pore diameters and volumes measuring 3.368 nm and 0.079 cc/g, respectively. The mesoporous structure of  $\text{ZnWO}_4@0.5\%$  CN nanocomposites enhances the adsorption capacity for organic pollutants in wastewater,



accomplished by uniformly distributing  $\text{ZnWO}_4$  over  $\text{g-C}_3\text{N}_4$  sheets [37].

Using scanning electron microscopy (SEM), the surface morphology of the synthetic samples was analyzed, as illustrated in Figure 5 (a-i). In the pure  $\text{ZnWO}_4$  nanoparticles shown in figure 5 (a-d), the surface morphology is rough and porous, and the grains are agglomerated in an uneven shape. Due to the high surface energy of  $\text{ZnWO}_4$ , the particles show non-uniform distribution, suggesting random development and partial aggregation. On the other hand, the  $\text{ZnWO}_4/\text{g-C}_3\text{N}_4$  composite is displayed in the figures 5(e-i), where  $\text{ZnWO}_4$  nanoparticles are interconnected with sheet-like  $\text{g-C}_3\text{N}_4$  structures. With these thin stacked sheets present, the surface

minutes of exposure to visible light. At 150 minutes of irradiation, however, the  $\text{ZnWO}_4@0.5\%\text{CN}$  photocatalyst demonstrated remarkable degradation efficiency. Based on these results, it seems that the heterojunction of  $\text{ZnWO}_4$  and  $\text{g-C}_3\text{N}_4$  speeds up the breakdown of RhB. Figure 6(a-d) shows the photodegradation rate of RhB for  $\text{ZnWO}_4$ ,  $\text{g-C}_3\text{N}_4$ ,  $\text{ZnWO}_4@0.1\%\text{CN}$ ,  $\text{ZnWO}_4@0.5\%\text{CN}$ , and  $\text{ZnWO}_4@1.0\%\text{CN}$  nanocomposites after 150 minutes of light irradiation; Figure 7 (a-b) shows the corresponding rate constant for the same nanocomposites under visible light illumination and figure 6(a-d) shows the photodegradation rate for  $\text{ZnWO}_4$ ,  $\text{g-C}_3\text{N}_4$ ,  $\text{ZnWO}_4@0.1\%\text{CN}$ ,  $\text{ZnWO}_4@0.5\%\text{CN}$ , and  $\text{ZnWO}_4@1.0\%\text{CN}$  nanocomposites.

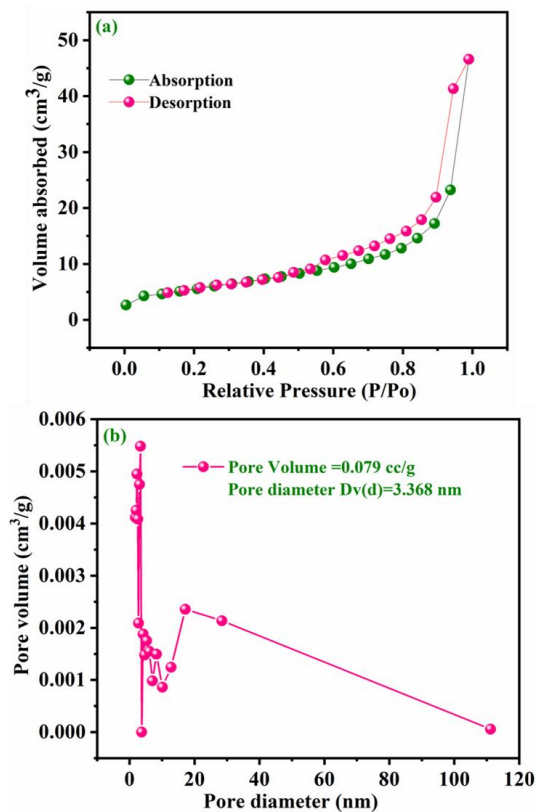


Figure 4. (a, b) Surface area and pore size distribution of  $\text{ZnWO}_4@0.5\%\text{CN}$  nanocomposites

contact area is increased and the  $\text{ZnWO}_4$  particles are distributed evenly over the  $\text{g-C}_3\text{N}_4$ . The successful integration of  $\text{ZnWO}_4$  and  $\text{g-C}_3\text{N}_4$  into a heterostructure is confirmed by the morphological alteration. The goal of designing this structure is to enhance photocatalytic activity and make charge separation more efficient.

## VI. PHOTOCATALYTIC APPLICATIONS

Using Rhodamine B dye molecules as a model pollutant, the photocatalytic degradation efficacy was investigated under visible light. The photodegradation of RhB by various photocatalysts under visible light irradiation from 0 to 150 minutes is illustrated in Figure 6(a-d). Before light irradiation, the dye solution and catalyst combination were placed in darkness for 30 minutes to achieve adsorption-desorption equilibrium. The photocatalysts, whether they were pure  $\text{ZnWO}_4$  exhibited very little photocatalytic activity during 150

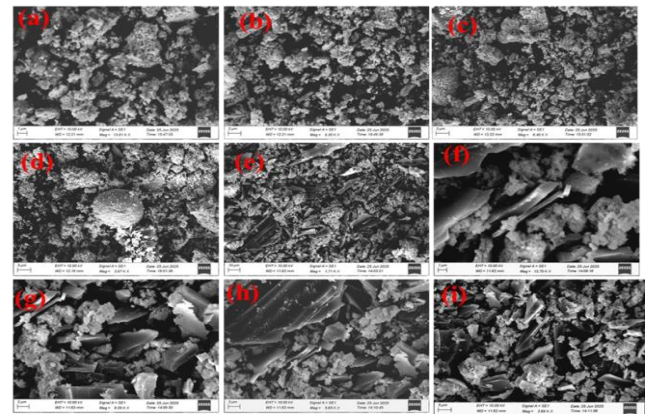


Figure 5. SEM images for (a-d)  $\text{ZnWO}_4$ , (e-i)  $\text{ZnWO}_4@0.5\%\text{CN}$  nanocomposites

After fitting the degradation data using a pseudo first order kinetic model, the following formula might be used to compute the degradation rates of the different photocatalysts: For every given value of  $C_0/C$ , the rate constant  $kt + x = -\ln(C_0/C)$ . A first-order kinetic model is shown in Figure 7b, which depicts the photocatalytic degradation of RhB, after which the relationship between irradiation time (t) and  $\ln(C_0/C)$  is determined. The kinetic parameter of the  $\text{ZnWO}_4@0.5\%\text{CN}$  nanocomposites was determined to be 0.0123  $\text{min}^{-1}$  comparison to pure  $\text{ZnWO}_4$  (0.0059  $\text{min}^{-1}$ ),  $\text{ZnWO}_4@0.1\%\text{CN}$  (0.0121  $\text{min}^{-1}$ ) and  $\text{ZnWO}_4@1.0\%\text{CN}$  (0.0112  $\text{min}^{-1}$ ), the  $\text{ZnWO}_4@0.5\%\text{CN}$  nanocomposites exhibited the highest photocatalytic activity (Figure 7b).

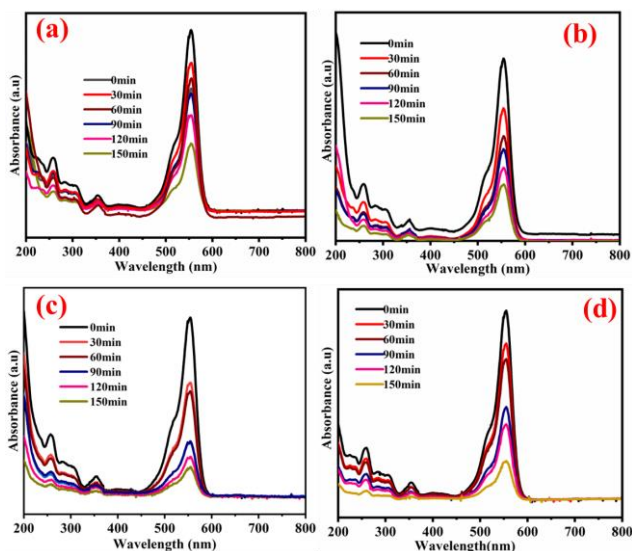


Figure 6. Photocatalytic performance of (a)  $\text{ZnWO}_4$  (b)  $\text{ZnWO}_4 @ 0.1 \% \text{CN}$  (c)  $\text{ZnWO}_4 @ 0.5 \% \text{CN}$  (d)  $\text{ZnWO}_4 @ 1.0 \% \text{CN}$  nanocomposite

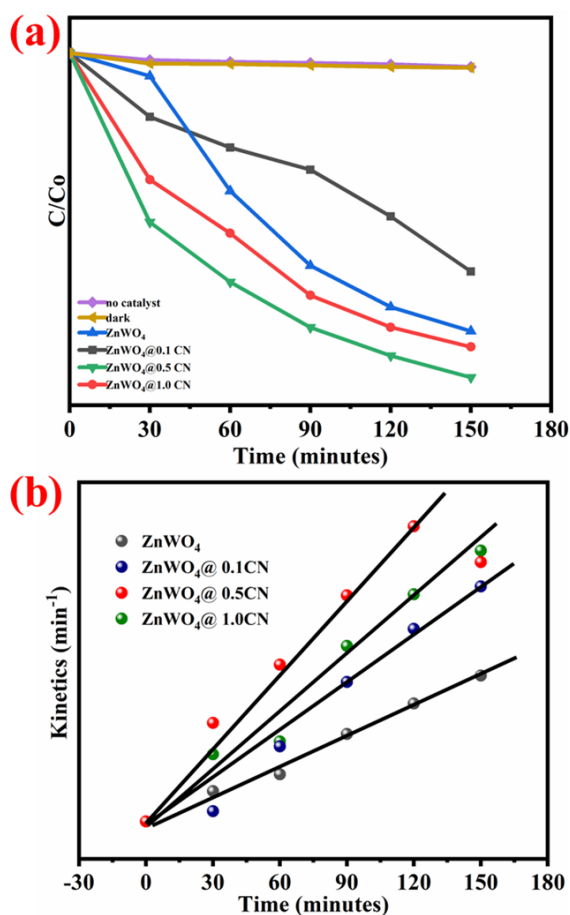


Figure 7. (a)  $C/C_0$  plot for degradation (b) Kinetics rate constant of  $\text{ZnWO}_4 @ 0.5 \% \text{CN}$  nanocomposites

## VII. DURABILITY TEST

For real-world applications, the materials' durability in their prepared state was crucial. Gathering, washing, and drying the material followed the deterioration process. For the following five cycles, the material was exposed to visible light. Also displayed in figure 8 shows the deprivation efficacy, which was determined for the first through fifth cycles in the following order: 97%, 96.8%, 95.4%, 94.8%, and 94.2%. Regarding degrading efficiency, there is a notable disparity. The stability and reusability experiments showed promising results for the  $\text{ZnWO}_4 @ 1.0 \% \text{AC}$  nanocomposite, which might be used for energy recovery and wastewater treatment for environmental remediation [38].

## VIII. SCAVENGER TEST

The impact of radical trapping agents on the photodegradation of RhB dyes is examined, as depicted in figure 9. The existence of scavengers in the RhB dye solution signifies the participation of reactive species, implying a progressive decline in degradation efficiency. The use of EDTA significantly diminished the removal effectiveness of the combined dyes from 97% to 26.4% for RhB. The photodegradation of RhB dye, with the inclusion of IPA, BQ and  $\text{AgNO}_3$ , has diminished the degradation efficiency to 39.4%, 74.1% and 80.2%, and respectively. The findings indicate that  $\text{h}^+$  significantly contributed to the degradation of

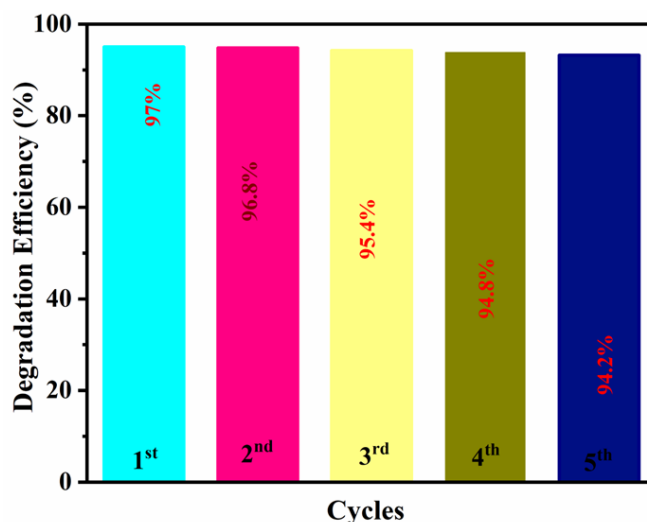


Figure 8. Durability test for  $\text{ZnWO}_4 @ 0.5 \% \text{CN}$  nanocomposites

RhB dye solution. The contribution of active species to the removal of mixed dyes by the  $\text{ZnWO}_4 / \text{g-C}_3\text{N}_4$  photocatalyst is ranked as follows:  $\text{h}^+ > \bullet\text{OH} > \bullet\text{O}_2 > \text{e}^-$  [39].

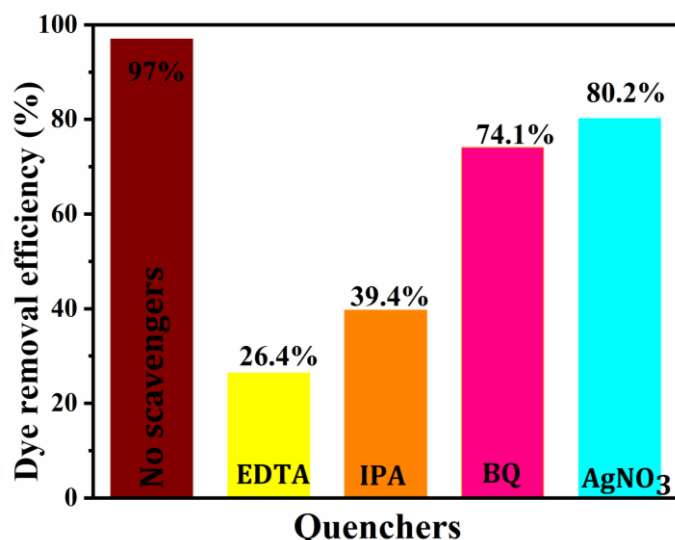


Figure 9. Scavenger test for ZnWO<sub>4</sub>@0.5%CN nanocomposites

### IX. STABILITY ANALYSIS

The stability of the materials was assessed by examining the elemental composition and distinctive peaks of both fresh and used ZnWO<sub>4</sub>@0.5%CN before to and following the photocatalytic reaction. Figure 10 demonstrates that the distinctive peak remains mostly unaltered following cycling, indicating the stability of the material's structure and the absence of major modification [40].

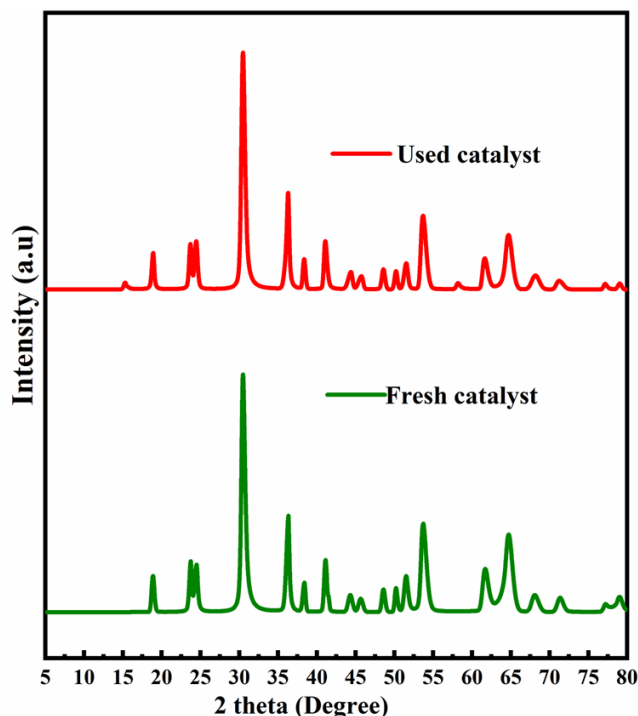


Figure 10. Structural stability test for ZnWO<sub>4</sub>@0.5% CN nanocomposites

### X. CONCLUSION

Photocatalytic materials including ZnWO<sub>4</sub> and ZnWO<sub>4</sub>/g-C<sub>3</sub>N<sub>4</sub> are synthesized in this study with composite ratios of 0.1%, 0.5%, and 1%. Compared to the pure ZnWO<sub>4</sub> phase, the g-C<sub>3</sub>N<sub>4</sub> loaded composite material has a lower prohibited

bandwidth and greater visible light absorption and utilization. When using a photocatalyst to break down RhB dye, ZnWO<sub>4</sub>@0.5%CN shows the highest catalytic activity. The consumption of the catalyst causes a modest decline in the catalytic effect after five cycles, demonstrating high stability. All of the active groups contribute to the degradation of pollutants, as  $\cdot\text{O}_2^-$ ,  $\cdot\text{OH}$ ,  $\text{h}^+$ , and  $\text{e}^-$  are the primary active species in the catalytic process. To sum up, our as-prepared ZnWO<sub>4</sub>@0.5%CN nanocomposite is environmentally friendly, contains suitable catalytic sites, is easily recyclable, and exhibits remarkable stability. This opens up a new avenue for practical research into the production of visible-light active materials and their potential use in antimicrobial and environmental remediation applications.

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