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Fabrication of ZnO/$CdMoO_4$/T-GCN Ternary Nanocomposite using Neem Extract for Photocatalytic Degradation of Rhodamine Dye

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Abstract: ***This study successfully prepared a green-synthesized ZnO/$CdMoO_4$/T-GCN (ZCMG) nanocomposite using neem (Azadirachta indica) leaf extract through a microwave-assisted co-precipitation method. The neem extract functioned as a natural reducing, stabilizing, and capping agent, facilitating an environmentally sustainable and economical synthesis pathway. The rapid microwave irradiation facilitated uniform nucleation, enhanced crystallinity, and diminished particle agglomeration. The structural and morphological characteristics of the synthesized nanocomposite were validated through XRD, FTIR, SEM, and UV–Vis. The establishment of a heterojunction among ZnO, $CdMoO_4$, and T-GCN improved light absorption and inhibited electron-hole recombination. The photocatalytic efficacy of the ZCMG nanocomposite was assessed by the degradation of Rhodamine dye under ultraviolet (UV) exposure. The composite demonstrated considerably better degradation efficiency relative to the individual components, due to enhanced charge separation and synergistic interfacial interactions. The improved photocatalytic activity is assigned to the establishment of an efficient Z-scheme/heterojunction system, enhanced reactive oxygen species production, and a broadened light absorption spectrum. The study presents a sustainable methodology for the design of effective ternary nanocomposites designed for wastewater treatment applications.***
Keywords: Green Synthesis, Neem leaf extract, Rhodamine dye, heterojunction, ZnO/$CdMoO_4$/T-GCN Ternary Nanocomposite.

I. INTRODUCTION

Rapid industrialization and urbanization have resulted in the extensive release of synthetic dyes into aquatic systems, causing serious environmental and health issues. Rhodamine dye, prevalent in the textile, paper, and printing industries, is recognized for its significant stability, toxicity, and resistance to standard wastewater treatment methods. The presence of such dyes in aquatic ecosystems diminishes light penetration, affects photosynthetic processes, and endangers living organisms. Therefore, the advancement of efficient, sustainable, and economical techniques for dye degradation is of extreme importance [1-3]. Photocatalysis has emerged as a promising advanced oxidation process for the elimination of organic pollutants owing to its environmentally friendly characteristics, low energy consumption, and the ability to achieve complete mineralization of contaminants under light irradiation [4]. Semiconductor metal oxides like ZnO have attracted significant interest due to their high photosensitivity, robust oxidizing capacity, non-toxicity, and superior chemical stability [5]. ZnO is considered an attractive alternative for TiO_2 because of its similar band gap and high quantum efficiency.

The substantial band gap of ZnO (~3.2 eV) restricts its absorption primarily to the UV region, which constitutes only a small fraction of the solar spectrum [6]. The rapid recombination of photogenerated electron–hole pairs markedly diminish its photocatalytic efficiency under visible light irradiation [7]. Consequently, various modification strategies, including heterojunction formation, doping, and composite fabrication, have been investigated to improve its visible light activity and charge separation efficiency. Cadmium molybdate ($CdMoO_4$) is an interesting semiconductor material recognized for its phenomenal chemical stability, unique optical characteristics, and photocatalytic ability [8]. However, its practical application is limited by insufficient visible-light absorption and rapid recombination of photogenerated charge carriers, which diminish its overall photocatalytic efficiency.

Graphitic carbon nitride (T-GCN), a metal-free polymeric semiconductor, has attracted considerable attention due to its appropriate band gap (~2.7 eV), effective visible-light responsiveness, high thermal and chemical stability, and economical synthesis [9]. The establishment of heterojunction systems by combining ZnO and $CdMoO_4$ with T-GCN is regarded as an effective approach to improve charge separation efficiency, extend the light absorption spectrum, and inhibit electron–hole recombination.

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These heterostructured composites enhance synergistic interfacial charge transfer and improve photocatalytic efficiency under UV light exposure [10]. For example, g-C_3N_4/ZnO nanocomposites exhibited effective degradation of Rhodamine-B under sunlight, demonstrating the advantages of heterostructure formation for pollutant elimination [11]. Green synthesis methods using plant extracts offer an eco-friendly method for nanomaterial fabrication, eliminating the need for toxic chemicals and minimizing secondary pollution [12].

Phytochemicals found in neem (*Azadirachta indica*) leaf extract, including flavonoids, terpenoids, phenolic compounds, and reducing sugars, function as natural reducing, capping, and stabilizing agents, thus enabling sustainable nanoparticle synthesis [13]. The utilization of biological extracts enhances ecological compatibility while simultaneously improving particle stability and surface capability [14]. Additionally, microwave-assisted co-precipitation provides benefits including expedited volumetric heating, consistent nucleation, diminished reaction duration, and superior crystallinity, resulting in nanocomposites with enhanced structural and optical characteristics [15]. The combination of green synthesis and microwave irradiation has been indicated to yield highly efficient photocatalysts with enhanced activity [16]. This study focused on the synthesis of a ternary ZnO/$CdMoO_4$/T-GCN nanocomposite utilizing neem extract through microwave-assisted co-precipitation, following a systematic characterization. The photocatalytic efficacy of the synthesized material has been evaluated for the degradation of Rhodamine dye under ultraviolet (UV) exposure. The improved performance is due to efficient heterojunction formation, expanded ultraviolet (UV) absorption, and reduced electron-hole recombination, rendering the developed nanocomposite a viable option for sustainable wastewater treatment applications [17].

II. EXPERIMENTAL SECTION

A. *Chemicals required*

Zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$), cadmium nitrate tetrahydrate ($Cd(NO_3)_2 \cdot 4H_2O$), sodium molybdate dihydrate ($Na_2MoO_4 \cdot 2H_2O$), and Thiourea (T-GCN synthesis) served as precursor materials. Alternatively, fresh neem (*Azadirachta indica*) leaves were utilized for green synthesis. Rhodamine dye served as the representative organic contaminant. All chemicals were of analytical grade and used without any further purification. Throughout the experiments, double-distilled water was used.

B. *Preparation of Neem Extract*

Fresh neem leaves were carefully rinsed with tap water and subsequently with distilled water to remove dust and impurities. The purified leaves were shade-dried and thoroughly chopped. 10 grams of chopped leaves were subjected to boiling in 100 ml of distilled water at a temperature range of 80 °C for a duration of 1 hour. The mixture was cooled to the outside temperature and filtered through Whatman filter paper to generate a clear neem leaf extract, which was subsequently stored at 4 °C for future use.

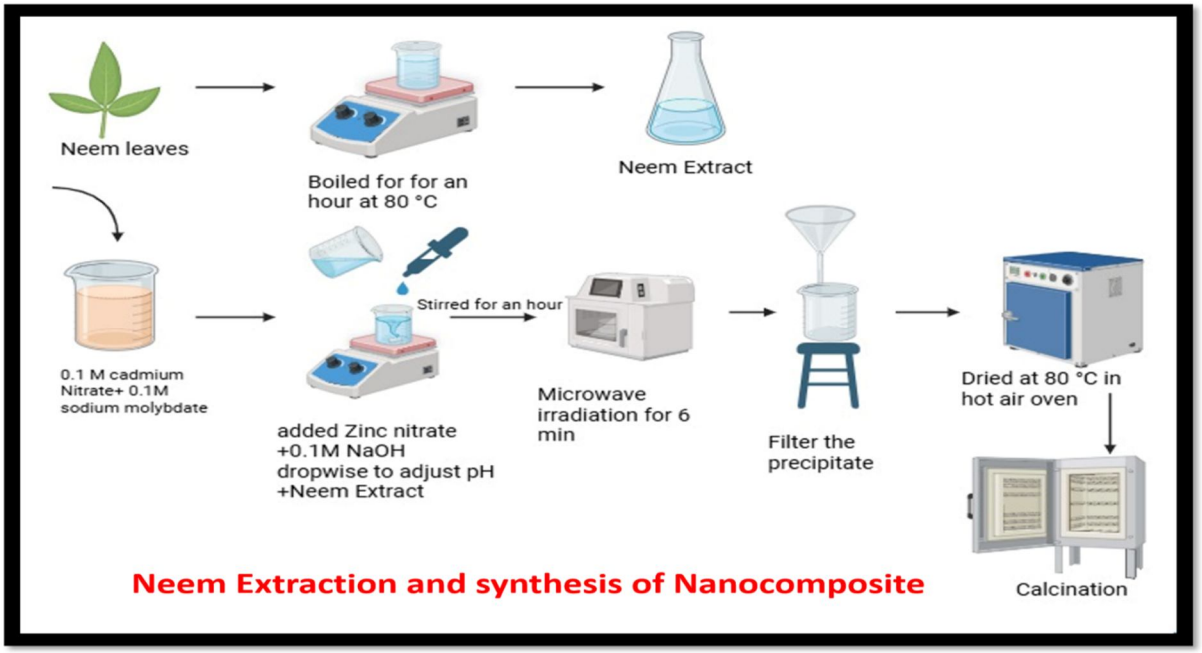


Fig. 1. Scheme of the synthesis of the nanocomposite via the microwave-assisted green co-precipitation method.

C. Synthesis of T-GCN

Thiourea was used as the precursor for thermal polymerization of graphitic carbon nitride. In a typical synthesis, 10 g of thiourea was heated in a covered alumina crucible with a lid. The muffle furnace was used to heat the crucible to 550°C at a controlled rate of 5°C min^{-1} for 2 hours under ambient conditions. The thermal treatment of thiourea produced polymeric graphitic carbon nitride by stepwise decomposition and condensation reactions that evolved gaseous species like ammonia and sulphur-containing compounds. A yellow solid product was created after the furnace cooled naturally to room temperature. The product was finely ground with an agate mortar and pestle to a uniform powder and stored in an airtight container for nanocomposite fabrication.

D. Green Synthesis of ZnO/$CdMoO_4$/T-GCN Nanocomposite via Microwave-Assisted Co-precipitation

0.1 M sodium molybdate and 0.1M cadmium nitrate were dissolved in water under constant stirring for 15 min. Then, 0.1M of zinc nitrate solution was added. A calculated amount of pre-synthesized T-GCN was added to the reaction mixture while stirring. Next, 30 mL of neem leaf extract was slowly added to reduce and stabilize. To facilitate precipitation, the solution pH was adjusted (9–10) using NaOH solution. To accelerate nucleation and uniform crystallization, the mixture was microwaved for 6 minutes. After centrifuging, the precipitate was washed several times with distilled water and ethanol to remove impurities and dried overnight at 80 °C. The ZnO/$CdMoO_4$/T-GCN nanocomposite was obtained by calcining the dried powder at 500 °C for 2 hours.

III. RESULTS AND DISCUSSION

A. XRD Analysis

The crystalline structure and phase purity of the synthesized ZnO/$CdMoO_4$/T-GCN nanocomposite have been investigated using X-ray diffraction (XRD). The diffraction pattern exhibited distinct characteristic peaks associated with ZnO, $CdMoO_4$, and graphitic carbon nitride (T-GCN), thereby confirming the successful synthesis of the ternary composite. The notable diffraction peaks at 2θ values of approximately 31.7°, 34.4°, 36.2°, 47.5°, and 56.6° correspond to the (100), (002), (101), (102), and (110) planes of ZnO, respectively, signifying its hexagonal crystal structure. The distinctive peaks of $CdMoO_4$ were observed at 2θ values of approximately 29°,32°,34°, and 47°, indicative of its tetragonal phase. A recognized diffraction peak at approximately 27.4° was attributed to the (002) interlayer stacking plane of T-GCN, whereas a less prominent peak near 13° was associated with the (100) in-plane structural packing motif. No additional impurity peaks were detected in the XRD pattern, indicating the high phase purity of the synthesized material. The minor decrease in peak intensity in the composite relative to the pure components signifies robust interfacial interaction and effective heterojunction formation among ZnO, $CdMoO_4$, and g-C_3N_4. The average crystallite size of the nanocomposite was calculated using the Debye–Scherrer equation, validating its nanoscale crystalline characteristics. The XRD results demonstrate a successful synthesis of a well-crystallized ternary ZnO/$CdMoO_4$/g-C_3N_4 nanocomposite appropriate for photocatalytic applications. We estimated the average crystallite size of the ZnO/$CdMoO_4$/S-g-C_3N_4 nanocomposite using the Debye–Scherrer equation:

$$\text{Average particle size } (D) = K\lambda / \beta \cos\theta \quad (2)$$

Where D is the crystallite size, K is the shape factor (0.9), λ is the wavelength of Cu Kα radiation (1.5406 Å), β is the FWHM of the diffraction peak, and θ is the Bragg angle [18]. This equation yielded an average crystallite size of 12.4 nm, confirming the synthesized composite's nanocrystalline nature. Strong interfacial interaction between ZnO, $CdMoO_4$, and T-GCN is indicated by slight variations in peak intensity and broadening of the semiconductor phases' close contact should improve charge carrier transfer across the interface, suppress electron–hole recombination, and boost visible-light photocatalytic performance [19].

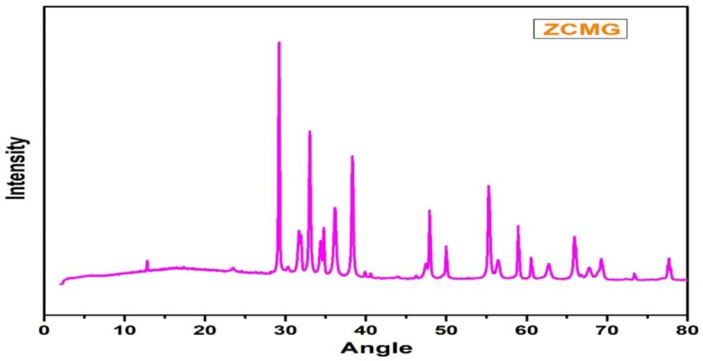


Fig. 2. X-Ray Diffraction plot of ternary ZnO/$CdMoO_4$/S-g-C_3N_4 nanocomposites.

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B. UV-DRS (UV-Visible Diffuse Reflectance Spectroscopy)

The ZnO/$CdMoO_4$/T-GCN nanocomposite showed a shift in the absorption edge compared to its components, indicating better light harvesting. The Tauc's plot from the Kubelka–Munk function determined the bandgap energy:

$$(\alpha hv) = A(hv\text{-}Eg)^n \quad (3)$$

Where α is the absorption coefficient, hν is photon energy, and E_g is the band gap energy, and n represents the electronic transition [20]. The calculated band gap of the nanocomposite was 3.1 eV, slightly lower than pure ZnO. The band gap decreases due to a heterojunction between ZnO, $CdMoO_4$, and S-g-C_3N_4, as well as possible sulphur-induced electronic changes.

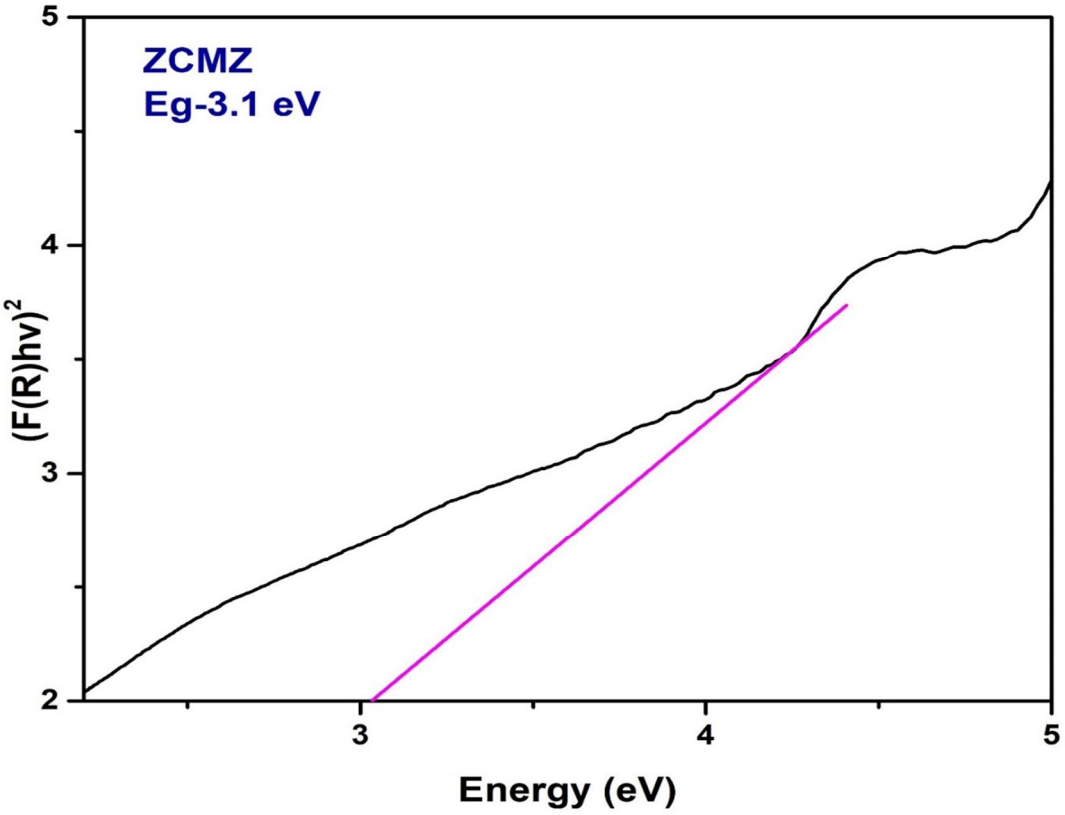


Fig. 3. Tauc plot of ZnO/$CdMoO_4$/S-g-C_3N_4 nanocomposite.

IV. PHOTOCATALYTIC RESULTS

A. Evaluation of Photocatalytic Performance for Rhodamine B Dye Degradation

The photocatalytic degradation of Rhodamine dye was performed using the synthesized ZnO/$CdMoO_4$/T-GCN nanocomposite under ultraviolet (UV) exposure. A stock solution of Rhodamine dye was initially prepared in double-distilled water and subsequently diluted to achieve a working concentration of 10 mg/L. 100 mL of the dye solution was transferred into a 100 mL borosilicate beaker, and then 50 mg of the photocatalyst was added. Before light exposure, the suspension was magnetically stirred in the dark for 30 minutes to establish adsorption-desorption equilibrium between the dye molecules and the catalyst surface. After establishing equilibrium, the reaction mixture was exposed to an ultraviolet light source while being continuously stirred to ensure uniform irradiation and distribution of the catalyst particles. At regular intervals of 20 minutes, small aliquots were extracted and centrifuged to separate the catalyst. The transparent supernatant was examined with a UV–Visible spectrophotometer at the peak absorption wavelength of Rhodamine dye (approximately 554 nm). The degradation efficiency was determined using the formula

$$(C_0 - C_t)/C_0 \times 100$$

where C_0 represents the initial dye concentration and C_t denotes the concentration at time t. The degradation kinetics were subsequently assessed employing the pseudo-first-order kinetic model.

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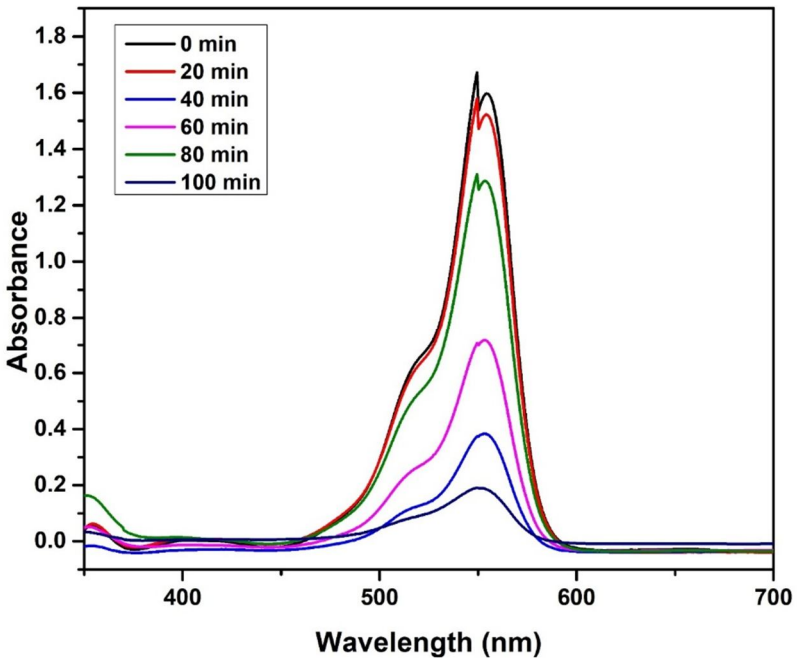


Fig. 4. Photocatalytic degradation of Rhodamine B dye with ZnO/$CdMoO_4$/T-GCN nanocomposite under ultraviolet (UV) exposure.

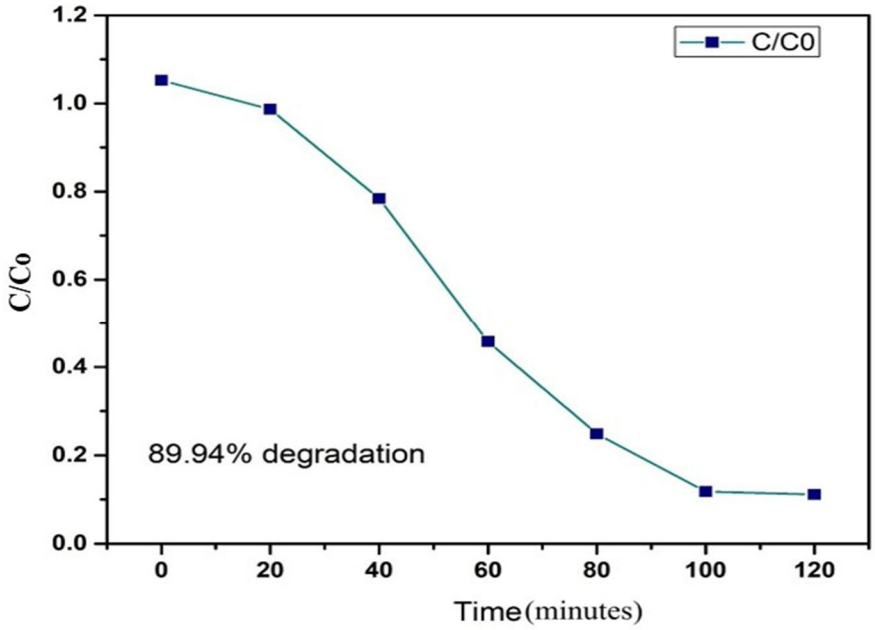


Fig. 5. C/C_0 versus time plot of the photocatalytic activity.

B. Photocatalyst Recyclability and Stability

The recyclability and stability of the ZnO/$CdMoO_4$/T-GCN nanocomposite were assessed to determine its practical utility in wastewater treatment. Following each photocatalytic degradation cycle of Rhodamine dye under UV irradiation, the catalyst was retrieved via centrifugation, meticulously rinsed with distilled water and ethanol to eliminate residual dye molecules, and subsequently dried at 80 °C prior to reuse in the ensuing cycle.

The photocatalyst underwent several consecutive degradation cycles under uniform experimental conditions. The results exhibited a minimal reduction in degradation efficiency following repeated use, signifying commendable stability and reusability of the nanocomposite.

The slight decrease in activity may be ascribed to negligible material loss during recovery or partial surface contamination by intermediate products. Moreover, the structural integrity of the recycled catalyst was validated through XRD analysis, which revealed no substantial alterations in the diffraction patterns relative to the fresh sample, thereby affirming that the crystalline structure remained unaltered following multiple photocatalytic reactions. The robust interfacial interaction among ZnO, $CdMoO_4$, and T-GCN enhances durability and photo corrosion resistance. The results indicate that the synthesized ZnO/$CdMoO_4$/T-GCN nanocomposite exhibits remarkable stability and recyclability, positioning it as a viable and sustainable photocatalyst for prolonged wastewater treatment applications.



Fig. 6. Percentage degradation for four cycles as a measure of recyclability data.

C. Radical Trapping Experiment

Radical trapping experiments were carried out under UV light irradiation to determine the active species responsible for the photocatalytic degradation of Rhodamine dye using the ZnO/$CdMoO_4$/T-GCN nanocomposite. Particular scavengers were introduced into the reaction system to determine the predominant reactive oxygen species involved in the degradation process. Isopropanol (IPA) served as a scavenger for hydroxyl radicals (•OH), benzoquinone (BQ) acted as a scavenger for superoxide radicals ($\bullet O_2^-$), and ethylenediaminetetraacetic acid (EDTA) functioned as a scavenger for holes (h^+). In a standard experiment, a suitable quantity of each scavenger was incorporated into the Rhodamine dye solution containing the photocatalyst before light irradiation. The degradation efficiency was subsequently compared to that of the control experiment conducted in the absence of any scavenger. A notable reduction in photocatalytic efficiency upon the introduction of a particular scavenger signifies the critical involvement of the associated reactive species.

The findings indicated that the incorporation of benzoquinone significantly diminished degradation efficiency, implying that superoxide radicals ($\bullet O_2^-$) are pivotal in the photocatalytic mechanism. The presence of EDTA significantly inhibited degradation, suggesting the participation of photogenerated holes (h^+). The incorporation of isopropanol exhibited a relatively minor effect, indicating a secondary influence from hydroxyl radicals (•OH).

It can be concluded from these observations that $\bullet O_2^-$ radicals and h^+ are the main active species responsible for the degradation of Rhodamine dye, whereas •OH radicals serve an additional role. The increased production of reactive oxygen species is attributed to the effective charge separation and transfer facilitated by the heterojunction structure of the ZnO/$CdMoO_4$/T-GCN nanocomposite.

$$O_2^{-}\bullet + BQ \longrightarrow BQ\bullet + O_2 \quad (5)$$

$$OH\bullet + IPA \longrightarrow IPA\bullet + H_2O \quad (6)$$

$$h^+ + EDTA \longrightarrow EDTA^+ \quad (7)$$

D. Photocatalytic Mechanism

A possible photocatalytic mechanism for the degradation of Rhodamine dye using the ZnO/$CdMoO_4$/T-GCN nanocomposite is proposed based on the UV–DRS results (Eg ≈ 3.1 eV) and radical trapping experiments. Upon exposure to ultraviolet (UV) radiation, T-GCN is efficiently excited owing to its appropriate band structure, resulting in the generation of photogenerated electrons (e^-) in the conduction band (CB) and holes (h^+) in the valence band (VB).The charge transfer is proposed to be linked to a Z-scheme heterojunction mechanism, based on the comparative band edge potentials of ZnO, $CdMoO_4$, and T-GCN. In this system, photogenerated electrons in the conduction band of ZnO recombine with holes in the valence band of T-GCN via close interfacial contact. This recombination pathway maintains the strong oxidative holes in the valence band of ZnO and the highly reductive electrons in the conduction band of T-GCN. The produced reactive oxygen species ($\bullet O_2^-$ and •OH), in conjunction with photogenerated holes (h^+), play a crucial role in the degradation and mineralization of Rhodamine dye into CO_2, H_2O, and other innocuous by products. The efficient separation of charge carriers, robust redox potential maintained in the Z-scheme structure, and enhanced ultraviolet absorption from S-doping collectively enhance the photocatalytic efficiency of the ZnO/$CdMoO_4$/T-GCN nanocomposite.

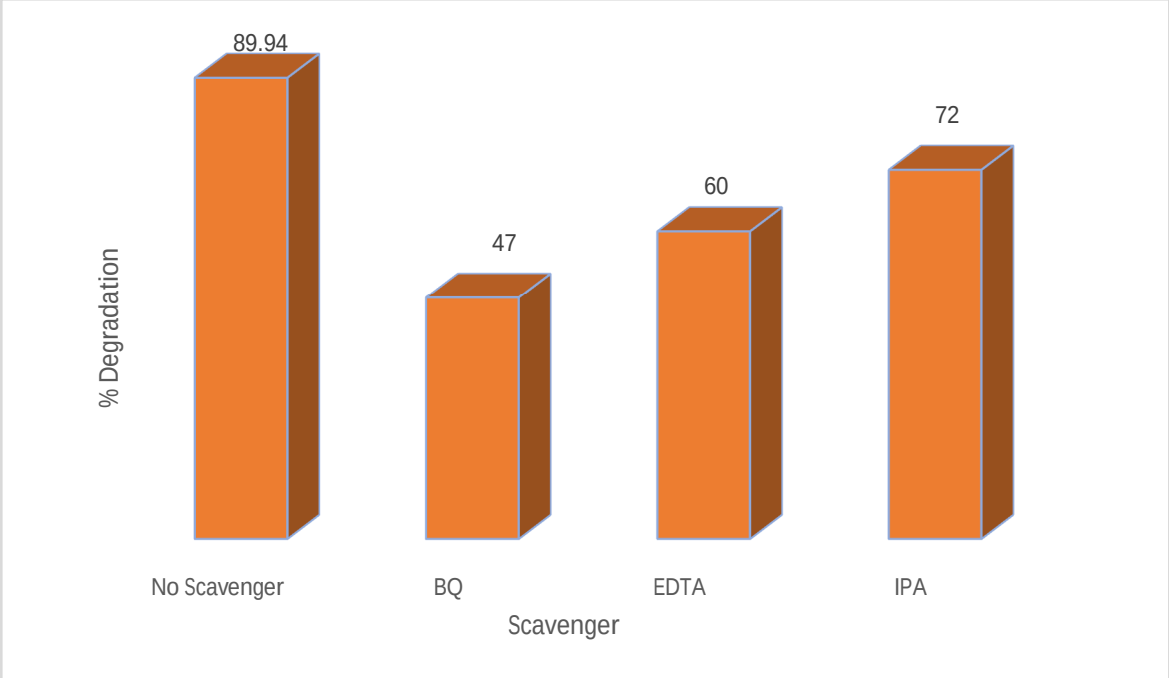


Fig. 7. Bar Graph showing degradation percentage for the scavenger test.

V. CONCLUSION

This study successfully synthesized and investigated a ZnO/$CdMoO_4$/T-GCN nanocomposite via a green synthesis method using neem (*Azadirachta indica*) leaf extract for the photocatalytic degradation of Rhodamine dye under ultraviolet (UV) exposure. Neem extract served as a natural reducing, stabilizing, and capping agent, providing the synthesis process environmentally friendly, economical, and sustainable. UV–DRS analysis demonstrated an optical band gap of 3.1 eV, indicating improved light absorption relative to the individual components. The establishment of a heterojunction between ZnO, $CdMoO_4$, and T-GCN markedly enhanced charge separation efficiency and reduced electron-hole recombination. Scavenger studies revealed that superoxide radicals ($\bullet O_2^-$) and photogenerated holes (h^+) were the primary reactive species in the degradation process, whereas hydroxyl radicals (•OH) assumed a secondary role. The proposed Z-scheme mechanism elucidated the improved redox capacity and effective charge transfer within the composite system.

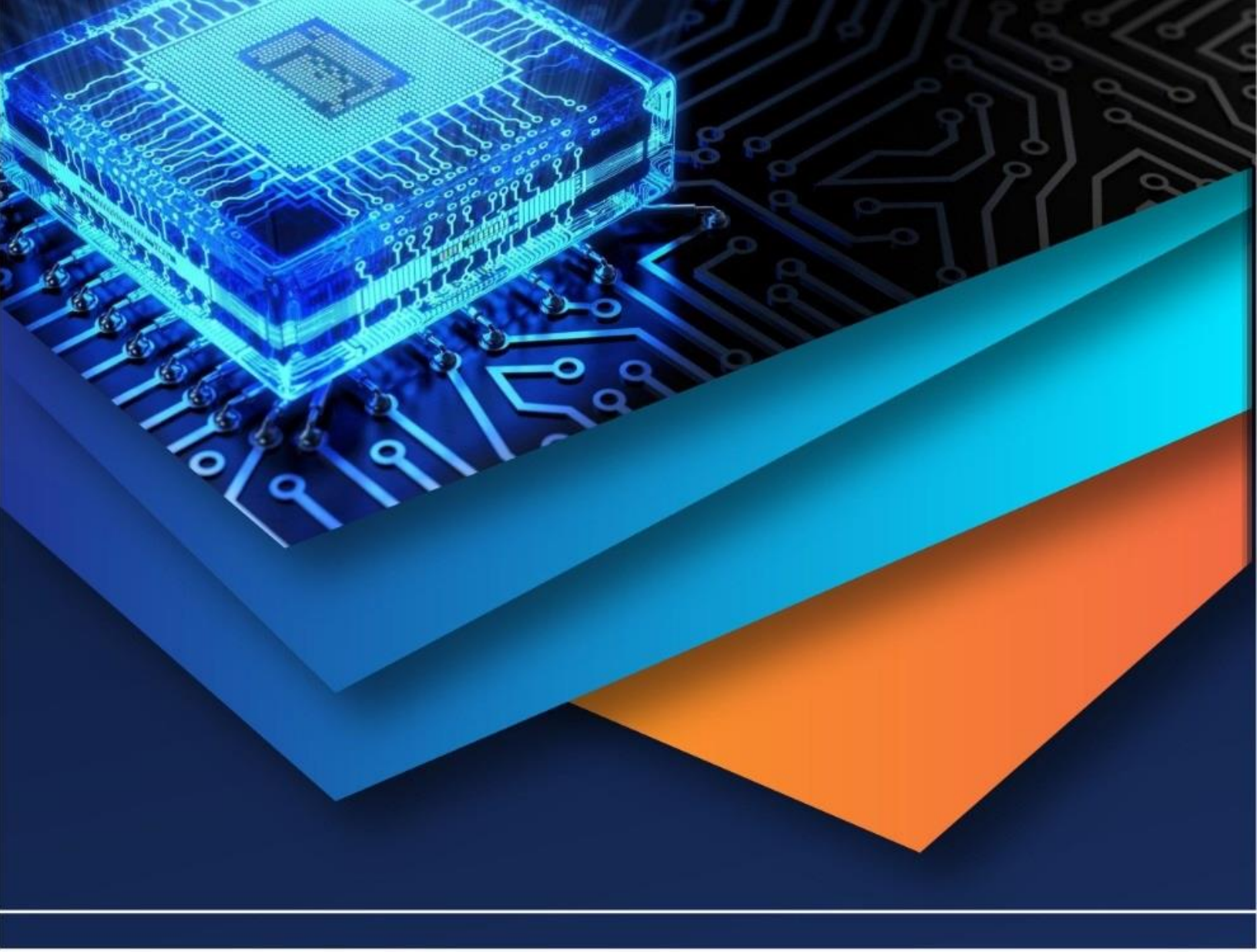
Moreover, the photocatalyst demonstrated significant degradation efficiency, coupled with outstanding recyclability and structural stability across numerous cycles, thereby affirming its durability and practical utility in wastewater treatment. The green-synthesized ZnO/$CdMoO_4$/T-GCN nanocomposite exhibits considerable promise as an effective, stable, and eco-friendly photocatalyst for sustainable environmental remediation applications.

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