



Morphology controlled fabrication of $\text{Fe}_2\text{O}_3/\text{GCN}$ composites: a comparative study of hydrothermal and sonochemical synthesis methods for efficient sunlight driven photocatalysis for environmental remediation

Chetan Harak¹ · Dilip Satpute¹ · Vinayak Kadam² · Nitin Kolhe¹ · Anil Wade¹ · Shital Balgude³ · Satish Mardikar⁴ · Sagar Balgude¹ · Hari Pawar¹

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Abstract

A novel bifunctional type of Z-scheme heterojunction with excellent adsorption and photocatalytic properties has been synthesized successfully using hydrothermal and sonochemical methods. The $\text{Fe}_2\text{O}_3/\text{g-C}_3\text{N}_4$ (GCN) nanocomposite, which has a distinct nanorod-like shape, has been characterized using various analytical techniques such as XRD, XPS, FESEM-EDS, TEM, and UV-DRS. The results of sunlight-driven photodegradation experiments indicate that the hydrothermally synthesized $\text{Fe}_2\text{O}_3/\text{GCN}$ nanocomposite achieved a 95% degradation of methylene blue (MB) dye within 45 min. The improved photocatalytic activity is believed to be due to the formation of a Z-scheme heterojunction system, as supported by active species trapping experiments. Additionally, the composite photocatalyst demonstrated high degradation efficiency and stability over four cycles. Trapping studies c $\cdot\text{OH}$ and $\text{O}_2\cdot^-$ were the principal active species in the photocatalytic process created by sunlight irradiation of a Z-scheme $\text{Fe}_2\text{O}_3/\text{GCN}$ photocatalyst.

Keywords Heterojunction · $\text{Fe}_2\text{O}_3/\text{GCN}$ · Graphitic carbon nitride · Photocatalysis

1 Introduction

Recently, visible-light active polymeric graphitic carbon nitride (GCN) with a narrow band gap of 2.7 eV has been reported as a novel metal-free photocatalyst [1]. GCN is a triazine-structured, metal-free semiconductor that is produced quickly by polymerizing inexpensive raw ingredients like thiourea, melamine, urea, and cyanamide [1]. Its exceptional physicochemical characteristics and use in a variety of applications, such as hydrogen production, carbon dioxide

reduction, antibacterial activity, gas sensors, drug delivery, and photocatalysis, have sparked a significant deal of interest in the scientific community [2–6]. It is widely accepted that $\text{g-C}_3\text{N}_4$ is an excellent photocatalyst for the production of hydrogen and the degradation of hazardous organic pollutants. Organic pollutants discharged into the environment by industry are a major concern due to their high toxicity and potential health concerns to living beings [3]. Among the various approaches, semiconductor-based photocatalysis is considered as a highly effective technology for the degradation of organic dyes with utilization of solar energy [6]. The technology could mineralize a wide range of organic pollutants into low-toxic organic moieties, CO_2 and H_2O at mild temperatures and pressures without use of expensive oxidants. $\text{g-C}_3\text{N}_4$ has received a lot of attention due to its good band structure and potential for photocatalytic destruction of organic pollutants. Despite significant research on heterojunction photocatalysts made of semiconductors and $\text{g-C}_3\text{N}_4$, the high rate of photogenerated electron and hole recombination limits the application of $\text{g-C}_3\text{N}_4$ for the degradation of hazardous organic pollutants [7]. There are numerous reports on different heterojunction photocatalysts in the literature, such as $\text{CeO}_2/\text{g-C}_3\text{N}_4$, $\text{ZnFe}_2\text{O}_4/\text{g-C}_3\text{N}_4$, $\text{WO}_3/\text{g-C}_3\text{N}_4$,

✉ Sagar Balgude
sagarbalgude88@gmail.com

✉ Hari Pawar
haripawar2010@gmail.com

¹ MES Abasaheb Garware College, Pune, Maharashtra, India

² MGVS Arts, Science and Commerce College, Nashik, Maharashtra, India

³ Shardabai Pawar Mahila Mahavidyalaya, Pune, Maharashtra, India

⁴ Smt. R S College, Sant Gadge Baba Amravati University, Amravati, Maharashtra, India

TiO₂/g-C₃N₄, Fe₂O₃/g-C₃N₄, and Bi₂O₃/g-C₃N₄ [8]. The Z-scheme heterojunction exhibits stronger oxidation and reduction abilities compared to those of single-component systems because of the more positive VB of one semiconductor and the more negative CB of another semiconductor [9]. Recently, enormous attention has been paid to the design and synthesis of g-C₃N₄-based Z-scheme photocatalysts to improve the activity of pristine g-C₃N₄. Compositing g-C₃N₄ with metal oxides results in a controlled band structure/band gap with enhanced visible light absorption and numerous active sites for photocatalytic reactions. For example, Zhao et al. successfully synthesized a g-C₃N₄/WO₃ heterojunction for the effective degradation of Rhodamine B dye under sunlight irradiation [9]. The synthesized heterojunctions were believed to channelize electrons from the conduction band of WO₃ to the valence band of g-C₃N₄, resulting in superior photocatalytic activity of the synthesized composites. In another report by Girish et al., g-C₃N₄ was composited with TiO₂, and they found that the photocatalytic efficiency of TiO₂ was significantly enhanced after making nanocomposites with g-C₃N₄ nanostructures [10]. Another Z-scheme photocatalyst composed of a g-C₃N₄/WS₂ heterojunction was reported by Nawaz et al. [11]. The composite catalyst was prepared by a one-pot hydrothermal fabrication method. The superior photoactivity was attributed to the p-n heterojunction formed due to the C₃N₄/WS₂ composite. In a report by Girish et al., a ZnO/g-C₃N₄ photocatalyst has been reported recently. The heterostructured photocatalyst was able to degrade dye as well as act as an HER catalyst [12]. The superior HER activity of the composite was ascribed to the increased charge-transfer rate owing to the better interfacial contact between the heterocomponents. The superior photoactivity was due to the nanoparticle nature of ZnO and g-C₃N₄ heterocomponents, which provided more exposed edge sites.

Magnetic metal oxide has recently gained popularity among researchers due to its ease of separation from the reaction mixture. Iron oxide (Fe₂O₃) is an important, non-toxic, abundant, low-cost material with narrow band gap [13]. The combination of Fe₂O₃ and g-C₃N₄ creates a heterojunction interface that effectively separates photogenerated electrons and holes [14]. These electron/hole pairs are crucial in photodegradation reactions. Several reports on the benefits of Fe₂O₃/g-C₃N₄ nanocomposite have been reported in the literature for various applications [8, 14–16]. These studies show that although significant progress has been made in improving the photocatalytic performance of Fe₂O₃/g-C₃N₄ nanocomposite, the photocatalytic performance still has to be improved. One efficient way to solve this problem is by morphologically modifying nanostructures, such as nanowires, nanosheets, nanorods, nanospheres, and nanoflowers, among others, with plenty of pores and large surface areas [17–19]. The objective of this

study is to propose a template-free technique for creating tunable morphology Fe₂O₃/g-C₃N₄ nanocomposite materials by adjusting reaction conditions.

In present paper, sonochemical and hydrothermal methods were used for the synthesis of Fe₂O₃/g-C₃N₄ nanocomposites. The effect of synthesis parameters on nanocomposite growth was investigated, and as-synthesized samples were examined using different characterization techniques. The photocatalytic activity of as-synthesized nanocomposites under natural sunlight irradiation was evaluated using MB dye solution as a model hazardous organic pollutant. Using Fe₂O₃/g-C₃N₄ nanocomposites, the plausible mechanism for the degradation of MB dye solutions was also examined.

2 Experimental

2.1 Materials and methods

All of the chemicals used in this work were obtained from Merck and used without further purification. X-ray diffraction (XRD) measurements were carried out on a Philips diffractometer using Cu K α radiation with a wavelength of 1.540 Å at RT. The surface morphologies and microstructures of the Fe₂O₃/GCN nanocomposite were examined by the field emission scanning electron microscopy (JEOL JSM-7600F, FEG-SEM). X-ray photoelectron spectra were obtained using a Prevac specifically designed ambient pressure XPS system outfitted with a VG Scienta SAX 100 emission controller monochromator and an Al K α anode (1486.6 eV) in transmission lens mode. The optical investigations were done using a Shimadzu UV-Vis spectrophotometer (Model 1800). The microstructure investigation of Fe₂O₃/GCN nanocomposite was performed using Transmission Electron Microscopy (JEOL JEM 2100 PLUS).

3 Synthesis of photocatalyst

Metal-free Graphitic carbon nitride (GCN) was synthesized by a co-polymerization method. Melamine was used as the precursor in the process of copolymerization. Typically, a silica crucible containing 5 g of melamine was heated to 550 °C for 5 h at a rate of 2 °C/min. After cooling the silica crucible to room temperature naturally, the yellow powder was ground to produce g-C₃N₄. The acquired sample was labelled as GCN and was further used to synthesize Fe₂O₃/GCN nanocomposites.

For the preparation of Fe₂O₃/GCN nanocomposite photocatalysts, a certain amount of iron (III) nitrate nonahydrate was first dissolved in 50 mL distilled water, and then 50 mg

GCN powder was dispersed in this solution by ultrasonication for 30 min. Subsequently, the pH of the above mixture was adjusted to 8–9 by dropwise adding of 0.2 M aqueous NaOH solution. The resultant mixture was treated sonochemically and hydrothermally independently after being magnetically agitated for 30 min at room temperature. For hydrothermal treatment, the reaction mixture was put into a 100-mL Teflon-lined stainless-steel autoclave, sealed, and kept at 140 °C for 24 h in oven. The autoclave was then allowed to cool naturally to ambient temperature. The precipitate was collected, centrifuged, washed, and dried in a 55 °C oven. The dry product was then calcinated for 4 h at 500 °C. The obtained sample was denoted as H-IGCN. Similarly, for sonochemical treatment, the magnetically stirred reaction mixture was exposed to ultrasonic probe radiation for 3 h (using a 5 s pulse ON and 2 s pulse OFF cycle with a 25% pulse amplitude). The precipitate was collected, centrifuged, washed and dried in a 55 °C oven. The dry product was then calcinated for 4 h at 500 °C. The obtained sample was denoted as S-IGCN.

4 Photodegradation and recycle experiments

The photocatalytic performance of as-synthesized $\text{Fe}_2\text{O}_3/\text{GCN}$ composite photocatalysts was evaluated by the degradation of MB under sunlight irradiation. A 250-mL Pyrex beaker was used as the photoreactor. In a typical experiment, 0.1 g of catalyst was dispersed in a 100 mL, 10 mgL^{-1} aqueous solution of MB for 15 min using sonochemical bath. Prior to visible-light irradiation, the suspension was kept on magnetic stirring in the dark for an hour to establish adsorption-desorption equilibrium of MB on the catalyst surface at room temperature. After an hour, the suspension was exposed to the sunlight. During photodegradation experiments, at a regulated interval, 4 mL aliquot was collected on and then centrifuged to remove the catalyst particles for analysis. The residual MB concentration during photodegradation was monitored using a UV-Vis spectrophotometer by measuring its light absorbance at 664 nm. After the photodegradation process, the nanocomposites were separated from the solution using a centrifuge operating at 3000 rpm for 20 min. Subsequently, they were thoroughly washed to eliminate any remaining MB residue adhered to the $\text{Fe}_2\text{O}_3/\text{GCN}$ surface. These nanocomposites were then dried in an oven at 100 °C for 2 h, resulting in a powdered form. This recovered catalyst powder was employed in a reusability experiment following the same photocatalytic method. This experiment was repeated for the first, second, and third cycles. To investigate the active species generated during the photocatalytic reaction, including holes (h^+), hydroxyl ($\text{OH}\bullet$) radicals, and superoxide ion ($\text{O}_2^{\bullet-}$) radicals, 1 mmol

L^{-1} of ethylene diamine tetraacetic acid (EDTA), isopropanol (IPA), and benzoquinone (BQ) were introduced into the MB dye solution.

5 Results and discussion

The morphological investigation of GCN, S-IGCN, and H-IGCN composites was performed using field emission scanning electron microscopy (FE-SEM). Figure 1 depicts FESEM images of as-synthesized GCN, S-IGCN, and H-IGCN composites, respectively. It can be observed from Figure 1a, b that bare GCN exhibits a typical layered structure with wrinkles. Figure 1d, e shows FESEM images of an S-IGCN composite fabricated by sonochemical treatment. The high magnification image (Figure 1d) reveals the formation of unevenly shaped crystals with variable size. Figure 1e shows that these unevenly shaped nanoparticles are aggregated on the surface of graphitic carbon nitride. Similarly, the formation of nanorods on the surface of graphitic carbon nitride produced during hydrothermal treatment can be observed in the case of H-IGCN composite (Figure 1g). A close examination of these composites reveals the nanorod-like shape of the as-produced H-IGCN composite (Figure 1h). These uniformly developed nanorods exhibits a diameter in the range of 10–20 nm and a length ~20–60 nm. It is evident from Figure 1 that the fabrication method has a considerable impact on the morphology evolution of the $\text{Fe}_2\text{O}_3/\text{GCN}$ nanocomposite and plays a key role in its growth. Furthermore, corresponding EDS elemental mapping of as-synthesized GCN, S-IGCN, and H-IGCN composites are depicted in Figure 1c, f, i, respectively. The results are in close agreement with the synthetic architecture utilized for synthesis of bare GCN and composites of GCN with Fe_2O_3 viz. S-IGCN and H-IGCN.

The investigation of the microstructure of a H-IGCN nanocomposite was conducted using transmission electron microscopy (TEM), as depicted in Figure 2. This revealed the presence of nanorod-like morphology of H-IGCN nanocomposite (Figure 2a, b), which is good agreement with FESEM results (Figure 1g, h). The inset of Figure 2c displays lattice fringes with a spacing of 0.37 nm, corresponding to the (012) lattice plane of Fe_2O_3 . The selected area electron diffraction (SAED) pattern of the H-IGCN nanocomposite, as shown in Figure 2d, clearly indicates its crystalline nature.

The X-ray diffraction patterns for the as-synthesized GCN, S-IGCN, and H-IGCN composites are shown in Figure 3. As can be seen from Figure 3a, the XRD peaks for bare GCN appearing at 27.4° corresponds to the (002) stacking layered structure. These results are in accordance with JCPDS card No. 87-1526 [20]. Furthermore, the XRD patterns of S-IGCN and H-IGCN composites are depicted

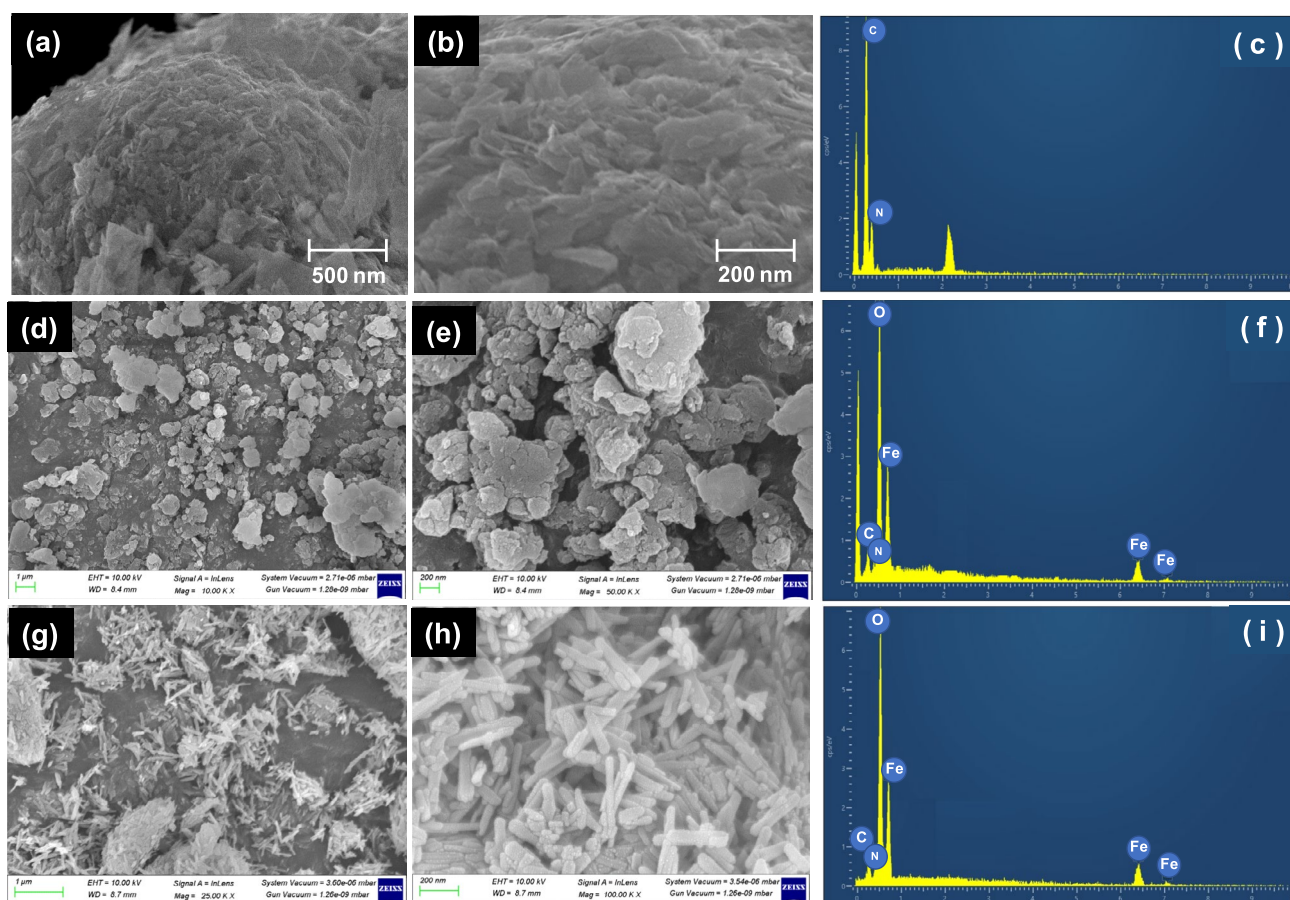


Fig. 1 FESEM images of GCN, S-IGCN, and H-IGCN (**a, b, d, e, g, h**) along with their corresponding EDS elemental mapping (**c, f, i**)

in Figure 3b. For H-IGCN, the diffraction peaks appearing at 23.98° , 29.99° , 33.03° , 35.50° , 40.78° , 49.27° , 53.93° , 57.35° , 62.28° , 63.81° , and 71.66° corresponds to (012), (002), (104), (110), (113), (024), (116), (122), (214), (030), and (220) planes. The presence of Fe_2O_3 and GCN peaks in the H-IGCN composites demonstrates that Fe_2O_3 was uniformly integrated onto crystalline GCN surface [13]. A similar XRD pattern was observed for the S-IGCN sample, with higher peak intensity than the H-IGCN sample. In both the composites, besides diffraction peaks of GCN and Fe_2O_3 , no diffraction peaks related to any impurities were observed, indicating the formation of highly pure $\text{Fe}_2\text{O}_3/\text{GCN}$ nanocomposites.

We also looked at the optical characteristics of the as-synthesized GCN, S-IGCN, and H-IGCN composites. The UV-vis diffuse reflectance spectra of bare GCN and the composite catalysts were recorded between 200 and 800 nm and are depicted in Figure 4a. From figure it can be seen that the bare GCN shows an absorption edge at about 433 nm. Furthermore, it is remarkable that the absorption curves of S-IGCN and H-IGCN composites are similar to bare GCN with higher intensity and shifted towards longer

wavelength. The optical spectra of S-IGCN shows absorption edge at ~ 551.86 nm, which is typically reported in the literature [21], whereas the H-IGCN displays a potent absorption band at ~ 590.68 nm. Meanwhile, the colors of the composites change from yellow for bare GCN to crimson and maroon for S-IGCN and H-IGCN, respectively, indicating the modification of the optical properties of $\text{Fe}_2\text{O}_3/\text{GCN}$ composites. The representative schematic pertaining to color change of bare GCN after compositing with Fe_2O_3 via individual hydrothermal and sonochemical processing in present investigation is depicted in Figure 5.

Furthermore, the UV-vis diffused reflectance data was used to calculate band gap energies of composite photocatalysts by using the following equation and the results are depicted in Figure 4b:

$$\alpha h\nu = A(h\nu - E_g)^{n/2} \quad (1)$$

where α , h , ν , A , and E_g represent the absorption coefficient, Planck constant, light frequency, proportionality constant, and band gap energy, respectively. The value of n is determined from the nature of optical transition. $n=2$

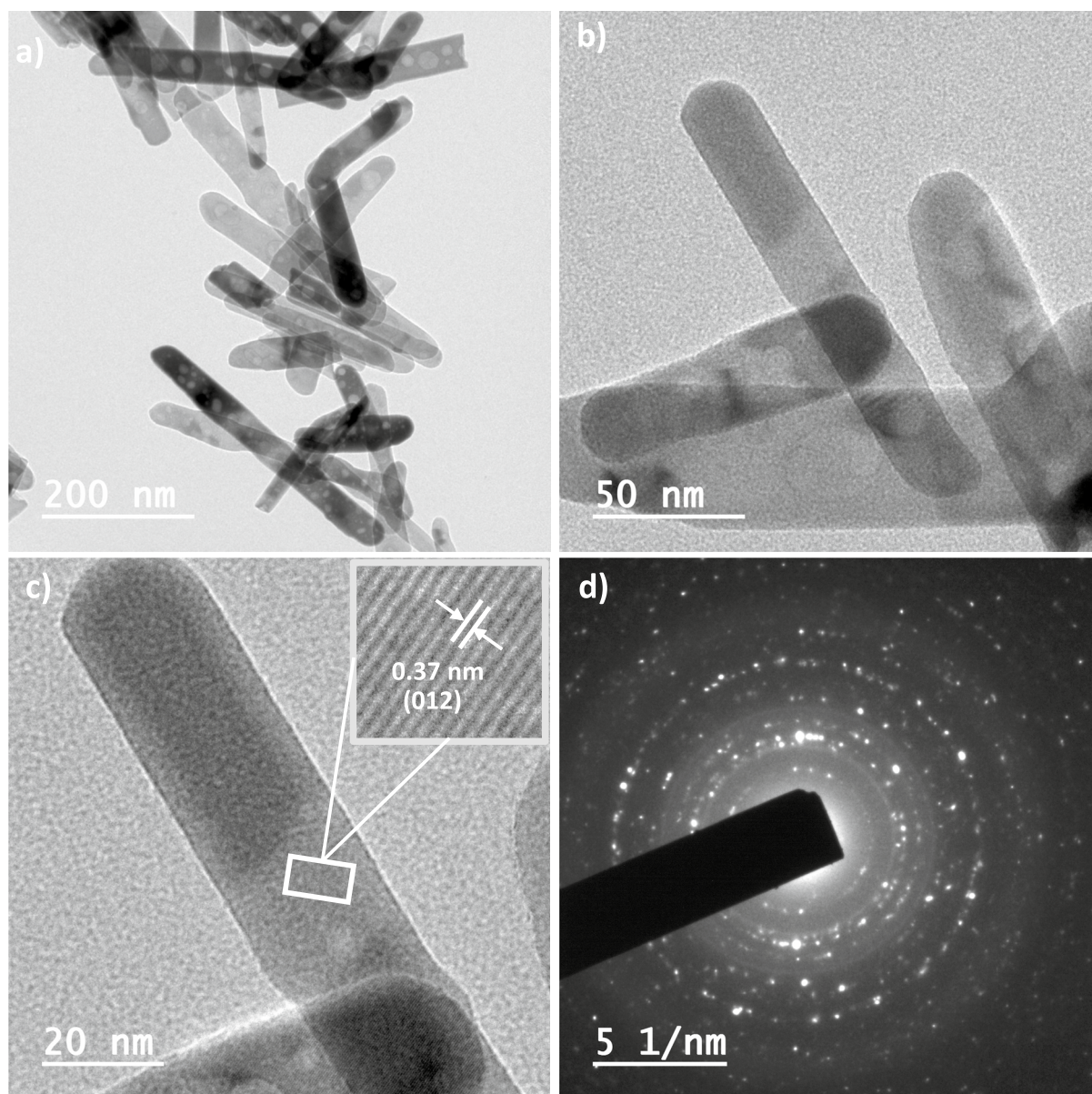


Fig. 2 a, b & c TEM images and d SAED pattern of H-IGCN

Fig. 3 XRD pattern of as-synthesized a GCN and b $\text{Fe}_2\text{O}_3/\text{GCN}$ composites, respectively

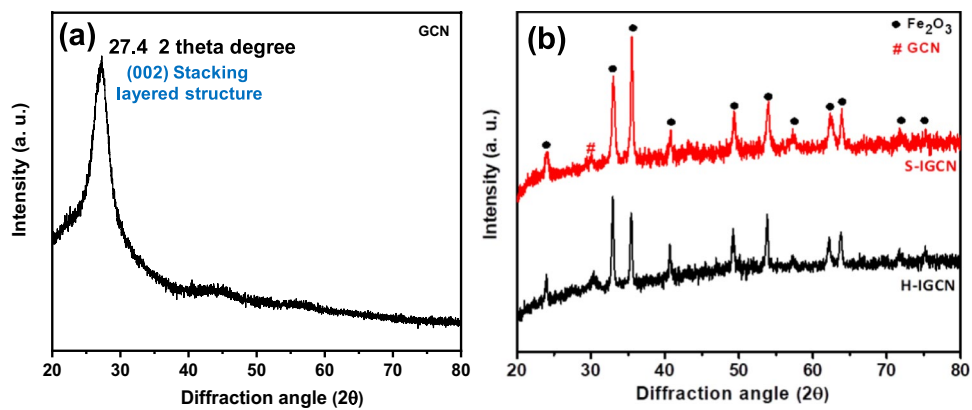


Fig. 4 **a** UV-vis diffuse reflectance spectra of GCN, S-IGCN, and H-IGCN composite photocatalysts. **b** Plot of $(\alpha h\nu)^2$ vs. photon energy ($h\nu$) for GCN, S-IGCN, and H-IGCN composite photocatalysts

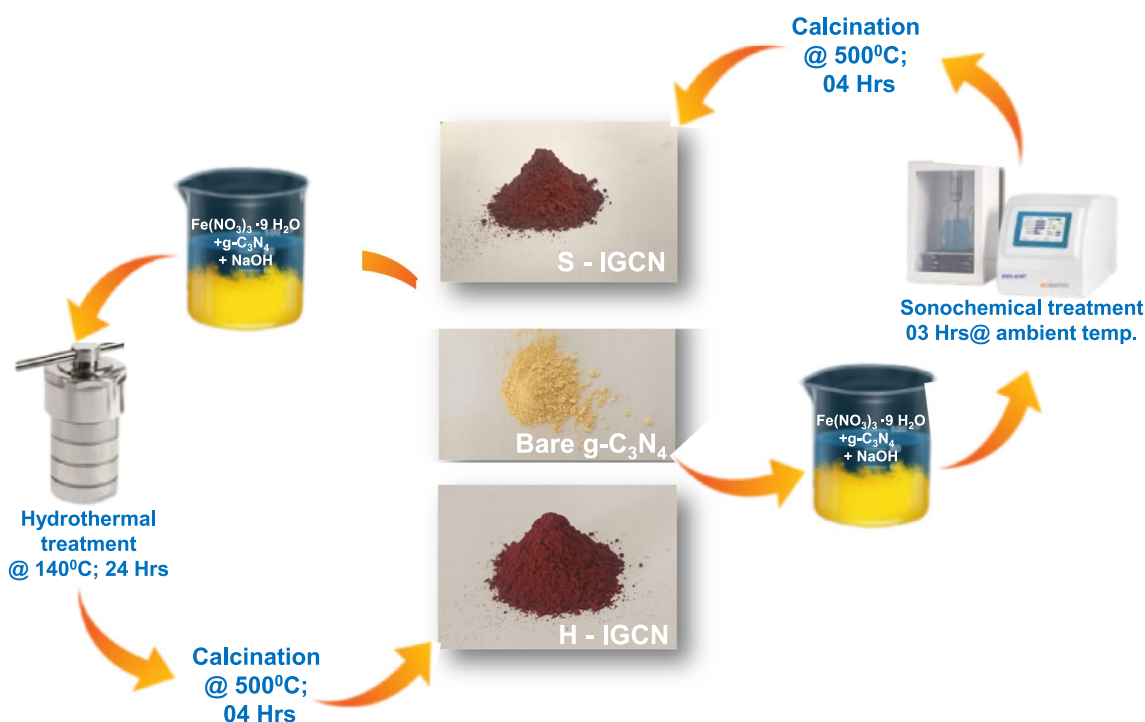
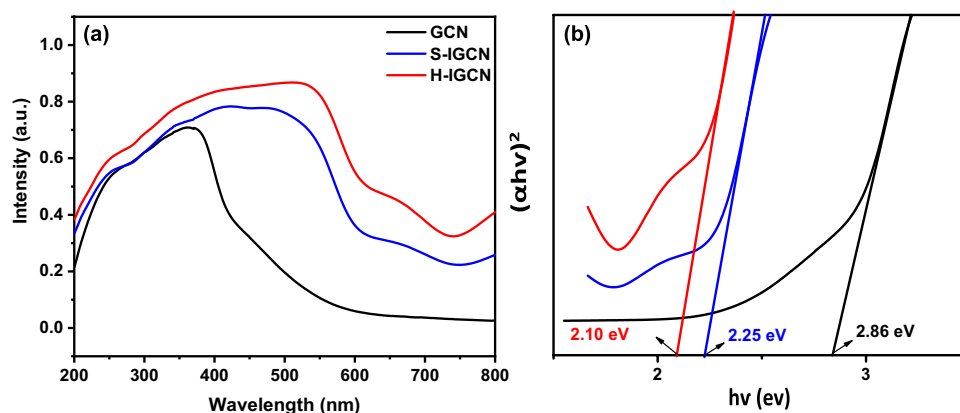


Fig. 5 Representative schematic illustrating color change of bare GCN after compositing with Fe_2O_3 via individual hydrothermal and sonochemical processing in present investigation

or 3 for indirect allowed and indirect forbidden transition, respectively, and $n = 1/2$ or $3/2$ for direct allowed and direct forbidden transition, respectively. The calculated band gaps for the GCN, S-IGCN, and H-IGCN composites were found to be 2.86, 2.25 eV, and 2.10 eV, respectively. In the case of H-IGCN composite compared to S-IGCN, the band gap was seen to be narrowing. This band gap narrowing may be attributed to nanorods-like morphology of H-IGCN [17]. When H-IGCN are exposed to sunlight, 1D hierarchical nanostructures that resemble nanorods cause light dispersion, which increases light harvesting compared to S-IGCN nanostructures. This could be advantageous for the photocatalytic activity of H-IGCN [22].

The chemical binding state of each atom in H-IGCN was confirmed by X-ray photoelectron spectroscopy. The high-resolution spectra of C, N, Fe, and O elements were depicted in Figure 6. The high-resolution spectra of C 1s show two primary peaks in Figure 6a, at 288.86 and 285.05 eV, which were attributed to C-(N)₃ and C-C bonding in the GCN lattice, respectively [23]. The peak observed at 286.62 eV could be the result of SP^2 hybridized carbon atoms present in C=N, which could be detected by graphitic domains in a CN matrix [24]. The high-resolution spectra of N 1s of the H-IGCN were deconvoluted in the peaks at 398.67, 399.30, and 400.21 eV, which were assigned to C-N-C, N-C₃, and C-N-H groups in the triazine rings of GCN (Figure 6b) [25].

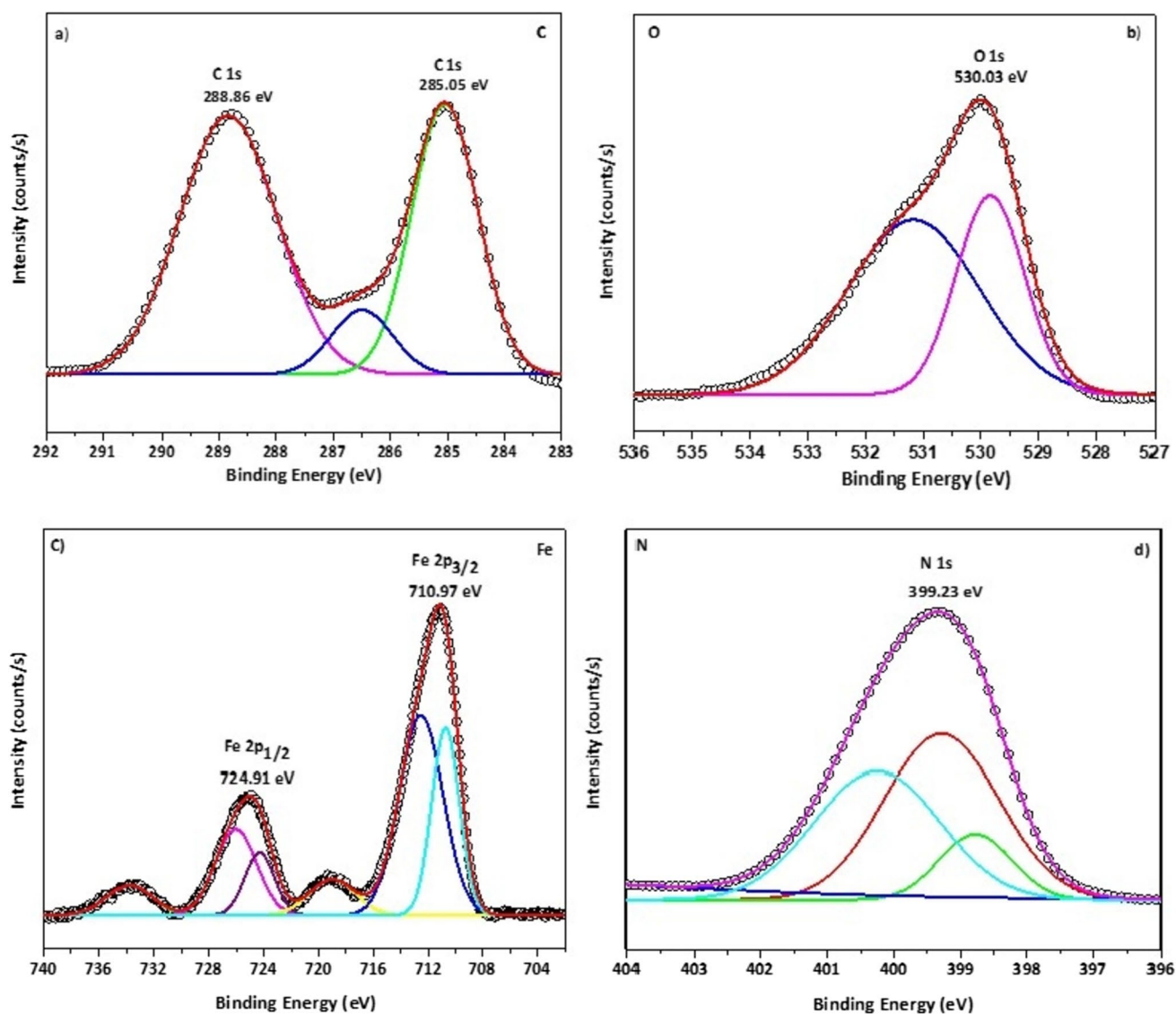


Fig. 6 High resolution XPS spectra of **a** C 1s, **b** O 1s, **c** Fe 2p, and **d** N 1s for representative H-IGCN composite

High-resolution spectra of the Fe 2p in H-IGCN are shown in Figure 6c, with the characteristic peaks of Fe 2p_{3/2} and Fe 2p_{1/2} being seen at binding energies of 710.97 and 724.91 eV, respectively [26]. There are two satellite peaks for Fe³⁺ in the H-IGCN, located at 719.23 and 733.45 eV. The characteristic peaks of O 1s were observed at 530.03 eV of binding energy (Figure 6d), which could be deconvoluted into two peaks. The Fe-O binding energy of Fe₂O₃ is shown by the peak at 529.36 eV, whereas the carbon-oxygen binding energy is represented by the peak at 531.25 eV [24]. In the comprehensive XPS investigation, no impurity peak was identified, indicating the formation of a highly pure Fe₂O₃/GCN composite.

The photocatalytic activity of as-synthesized H-IGCN and S-IGCN for the methylene blue degradation of the

model textile dye was evaluated in the presence of sunlight irradiation. Figure 7a depicts the absorption of aqueous MB dye under various testing conditions and time periods. The graph demonstrates that in the presence of the photocatalysts, the absorbance of aqueous solutions of MB dye declined steadily as the illumination time increased. The pure GCN exhibits a poorer response to MB dye degradation compared to Fe₂O₃/g-C₃N₄ composites. The H-IGCN photocatalyst degraded more than 95% of the MB dye after 45 min, compared to 84% for the S-IGCN photocatalyst during the same time period. The fact that the rate of MB dye degradation was observed to be higher in case of H-IGCN compared to S-IGCN clearly demonstrates that band structure and nanorod-like morphology have a significant impact on the efficiency of MB dye degradation under the comparable



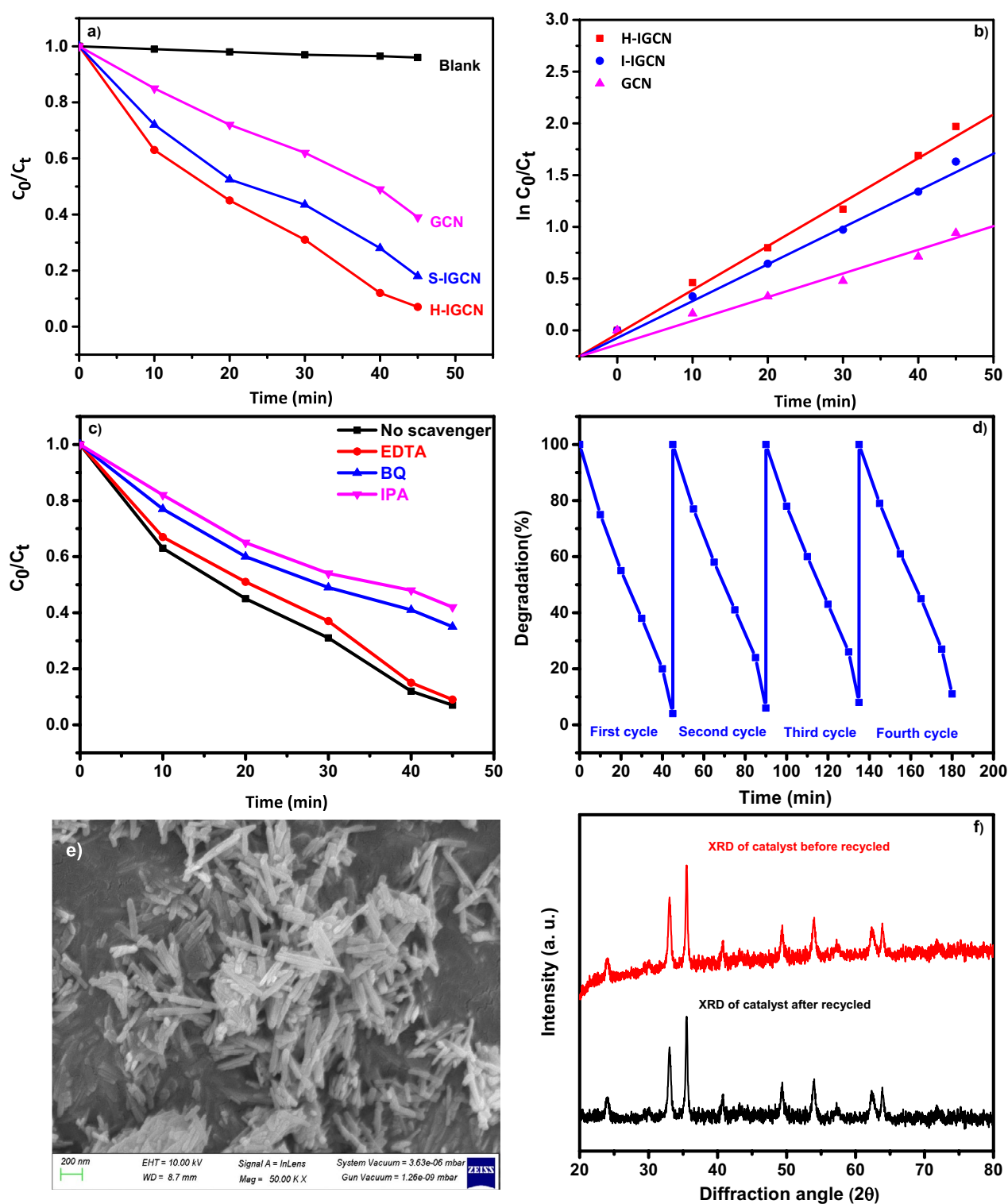


Fig. 7 **a** Photocatalytic degradation of MB dye in aqueous solution using GCN, H-IGCN, and S-IGCN composites; **b** plot of $\ln(C_0/C_t)$ vs. reaction time for the photocatalytic MB degradation using GCN, H-IGCN, and S-IGCN composites; **c** effect of scavengers on MB

degradation over H-IGCN composite; **d** recyclability for photodegradation of MB dye by H-IGCN; **e** FESEM of H-IGCN after photocatalytic reaction; and **f** XRD spectra of H-IGCN before and after photocatalytic reaction

conditions. For comparison, a controlled experiment was also conducted without a catalyst. The MB dye scarcely decomposed in the absence of a catalyst. Therefore, exposure to light has a significant impact on the degradation of aqueous MB dye.

In addition, the kinetics of the reactions of the GCN, H-IGCN, and S-IGCN photocatalysts in the degradation of MB were studied. Figure 7b depicts pseudo first-order kinetic graphs of $\ln(C_0/C_t)$ vs. time for MB degradation. The rate constants for the GCN, H-IGCN, and S-IGCN composites were found to be 0.0123, 0.0178, and 0.0163 min^{-1} , respectively. The results suggest that under identical conditions, the H-IGCN composite should have superior photocatalytic activity than the S-IGCN composite. The comparative results of MB dye degradation are shown in Table 1 [12, 27–31]. The highest degradation of methylene blue (MB) was successfully accomplished using H-IGCN, primarily owing to its narrow band gap and the presence of a well-defined crystalline nanorod-like structure. The narrow band gap falls within the visible spectrum, allowing for the absorption of a maximal amount of sunlight and thereby generating the highest number of electron-hole pairs. These findings unambiguously show that morphologically controlled synthesis of Fe_2O_3 with $\text{g-C}_3\text{N}_4$ considerably improves photocatalytic activity [32].

The contribution of various active radicals to the degradation of MB dye using the H-IGCN photocatalyst has been examined using radical trapping studies. To capture holes, hydroxyl radicals, and superoxide radicals in radical trapping assays, 1 mmol of ethylenediaminetetraacetic acid (EDTA), isopropanol (IPA), and Benzoquinone (BQ) are introduced to the photocatalytic system [33]. Figure 7c depicts the photocatalytic activity of H-IGCN in the presence of several trapping agents. From figure, the results demonstrate that the photocatalytic activity of the H-IGCN nanocomposite was significantly reduced in the presence of IPA and BQ, indicating that the principal oxidative species involved in the degradation process are $\bullet\text{OH}$ and $\text{O}_2^{\bullet-}$. The photocatalytic activity is unaffected by the addition of EDTA to trap h^+ , proving that holes are not the main active species in the system at hand. The obtained results demonstrated that the

rate of MB dye degradation is significantly influenced by the concentration of hydroxyl and superoxide radicals.

The reusability investigation of the photodegradation of MB dye utilizing H-IGCN composite is shown in Figure 7d. Figure shows that the performance of the H-IGCN composite remained consistent after four cycles with very little performance variation caused by catalyst loss during handling. These results revealed that the H-IGCN composite was a stable and effective photocatalyst when compared to previously published reports [33–36]. Following four cycles, the catalyst demonstrates nearly identical degradation effectiveness. Figure 7e displays the FESEM image of the photocatalyst after recovery, revealing a structure similar to that shown in Figure 1. This observation underscores the photocatalyst's excellent stability, further confirming its suitability for extended use. Furthermore, the crystal structure of H-IGCN remains unchanged throughout the photocatalytic degradation reactions (as shown in Figure 7f), suggesting that H-IGCN is a reliable catalyst activated by sunlight and therefore well-suited for practical uses in the industry.

On the basis of the above explanation, a probable mechanism for MB photocatalytic degradation employing the H-IGCN composite under sunlight illumination can be proposed. Figure 8 shows the possible mechanism of photodegradation of MB dye in the presence of H-IGCN ($\text{Fe}_2\text{O}_3/\text{GCN}$) nanocomposite. The charge transfer mechanism between the interfacial phase could be regulated by the Z-scheme [37]. After sunlight is irradiated on the surface of the photocatalyst, electron and hole pairs are formed. These photogenerated e^-/h^+ pairs are responsible for photodegradation of MB dye. The h^+ in VB of Fe_2O_3 react with H_2O molecule to generate radicals. While electrons in the CB of Fe_2O_3 migrate to the VB of GCN, this mechanism is advantageous for separating e^-/h^+ pairs on different sides of the $\text{Fe}_2\text{O}_3/\text{GCN}$ composite. The transferred electron in the CB of GCN can easily convert O_2 to $\text{O}_2^{\bullet-}$ radical. The radical scavenger experiment provided evidence for the generation of $\bullet\text{OH}$ and $\text{O}_2^{\bullet-}$ active radicals (Fig. 7c). These photogenerated active hydroxyl/superoxide radicals can then cause the degradation of the MB dye molecule [22].

Table 1 Comparison of photocatalytic MB dye degradation with some other photocatalysts

Photocatalysts	Light source	Dye concentration	Amount of catalyst (g/L)	Time (min)	% degradation	References
$\text{ZnO@g-C}_3\text{N}_4$	250 W Xenon lamp	5 ppm	0.4	120	91.5%	[12]
P25	Sunlight	10 ppm	0.5	70	94%	[27]
$\text{g-C}_3\text{N}_4@ \text{TiO}_2$	Solar light	10 ppm	0.02	100	94.92%	[28]
$\text{CdMoO}_4@ \text{g-C}_3\text{N}_4$	35 W Xenon arc lamp	10 ppm	0.01	145	93.70%	[29]
$\text{g-C}_3\text{N}_4@ \text{MoS}_2$	Sunlight	5 ppm	0.1	80	98.7%	[30]
$\text{WO}_3@ \text{g-C}_3\text{N}_4$	Visible light	50 ppm	0.5	90	95%	[31]
$\text{Fe}_2\text{O}_3/\text{g-C}_3\text{N}_4$	Sunlight	10 ppm	0.1	45	95%	Present work

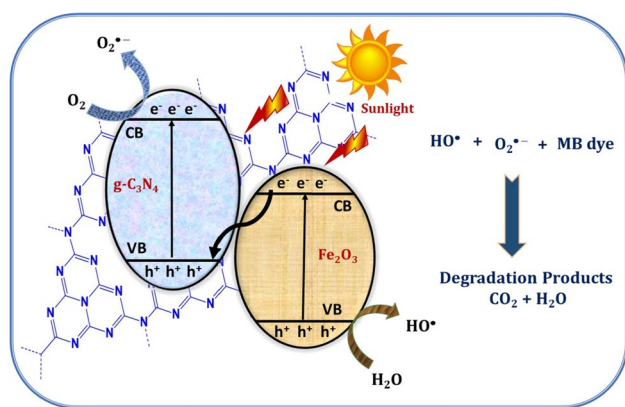


Fig. 8 Possible mechanism of photodegradation of MB dye in the presence of H-IGCN ($\text{Fe}_2\text{O}_3/\text{GCN}$) nanocomposite

6 Conclusion

In conclusion, a very effective $\text{Fe}_2\text{O}_3/\text{GCN}$ composite that was made utilizing several synthesis techniques has been a fascinating way to examine the impact of synthesis technique on various properties of a single nanocomposite. The phase structure, morphology, and optical properties of $\text{Fe}_2\text{O}_3/\text{GCN}$ composites have been thoroughly studied. The photocatalytic activity of as-synthesized $\text{Fe}_2\text{O}_3/\text{GCN}$ for MB dye degradation was high. The increased photocatalytic activity of a nanorods-like $\text{Fe}_2\text{O}_3/\text{GCN}$ composite was attributed to morphology, the synergistic effects of Fe_2O_3 loading on the surface of GCN, and separation of photoexcited electron-hole carriers.

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Author contribution CH: methodology, conceptualization. DS: validation, methodology. VK: resources, data curation, writing—review and editing. NK: investigation, validation. AW: formal analysis, data curation, validation. SB: visualization, formal analysis. SM: data curation, software, writing—review and editing. SB: writing—original draft, writing—review and editing. HP: supervision, resources.

Declarations

Competing interests The authors declare no competing interests.

Statement of industrial relevance The successful synthesis of the $\text{Fe}_2\text{O}_3/\text{GCN}$ nanocomposite with excellent adsorption and photocatalytic properties holds significant industrial relevance in the field of wastewater treatment and environmental remediation. The high degradation efficiency and stability of the composite photocatalyst, along with its ability to effectively degrade organic dyes under sunlight irradiation, make it a promising candidate for the development of advanced photocatalytic systems for industrial applications.

Statement of novelty The novel bifunctional Z-scheme heterojunction synthesized through hydrothermal and sonochemical methods represents a significant advancement in the field of photocatalysis. The distinct nanorod-like shape of the $\text{Fe}_2\text{O}_3/\text{GCN}$ nanocomposite, along with its characterization using various analytical techniques, highlights the meticulous research and comprehensive understanding of the material's properties. The formation of a Z-scheme heterojunction system and the identification of $\cdot\text{OH}$ and $\text{O}_2^{\cdot-}$ as the principal active species through trapping studies demonstrate a novel approach to enhancing photocatalytic activity. The achievement of 95% degradation of methylene blue dye within a short duration further emphasizes the novelty and practical applicability of this nanocomposite.

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