



## Development of visible-light-active Fe<sub>2</sub>O<sub>3</sub>-hBN photocatalysts for sustainable degradation of emerging pollutants under simulated sunlight conditions

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### ABSTRACT

In this work, Fe<sub>2</sub>O<sub>3</sub>-hBN composites with different Fe<sub>2</sub>O<sub>3</sub> loadings (0.2–2 wt%) were synthesized via a dry impregnation approach and evaluated as sustainable photocatalysts for water purification. The materials were comprehensively characterized using XRD, SEM, TEM, XPS, FTIR, UV-Vis, PL, and N<sub>2</sub> adsorption-desorption analyses to elucidate their structural, optical, and surface properties. Under simulated solar irradiation, the 0.5 wt% Fe<sub>2</sub>O<sub>3</sub>-hBN composite showed the most efficient photocatalytic activity among the tested materials. Approximately 80% removal of methylene blue and more than 70% degradation of ibuprofen were achieved within 120 min, highlighting the superior performance of this composition. This improvement was attributed to enhanced visible-light absorption and a reduced band gap (2.7 eV). Radical scavenging experiments identified photogenerated holes (h<sup>+</sup>) and superoxide radicals (O<sub>3</sub><sup>-</sup>) as the main reactive species. The composite exhibited excellent stability and reusability across multiple cycles. These findings highlight Fe<sub>2</sub>O<sub>3</sub>-hBN as an efficient and eco-friendly photocatalyst for sustainable water remediation applications.

### 1. Introduction

Environmental pollution by organic contaminants, including synthetic dyes and pharmaceutical compounds, has become a major concern due to their persistence, toxicity, and continuous release into aquatic environments. These pollutants, commonly originating from textile, pharmaceutical, and personal care industries, pose significant ecological and human health risks even at low concentrations (Chakraborty et al., 2023; Khan et al., 2024). Consequently, the development of efficient and sustainable treatment technologies for the removal of such hazardous contaminants from water remains a pressing challenge.

Among advanced water treatment strategies, photocatalytic degradation has emerged as a promising and environmentally friendly

approach, as it enables the transformation of organic pollutants into less harmful species by utilizing semiconductor materials and light energy (Mohammed et al., 2023; Navidpour et al., 2024). Solar-driven photocatalysis is particularly attractive because sunlight is abundant, renewable, and cost-free. However, conventional photocatalysts such as TiO<sub>2</sub> and ZnO are limited by their wide band gaps (3.20 and 3.37 eV, respectively), rapid electron-hole recombination, and activation predominantly under ultraviolet irradiation, which accounts for only a small fraction of the solar spectrum. These limitations have motivated extensive research into visible-light-responsive photocatalysts through strategies such as heterojunction construction, surface modification, and composite material design (Ahmadpour et al., 2020).

In this context, two-dimensional (2D) hexagonal boron nitride (hBN) has emerged as a promising support for photocatalytic systems. hBN

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possesses excellent chemical and thermal stability, a layered structure, and a high specific surface area (Cassabois et al., 2016; Sharma et al., 2025). Although its wide band gap (~5.5 eV) prevents intrinsic visible-light photoactivity, hBN can act as an effective support or co-catalyst, facilitating charge migration and suppressing charge recombination when coupled with visible-light-active semiconductors (Gao et al., 2020).

Iron oxide (Fe<sub>2</sub>O<sub>3</sub>), particularly hematite (α-Fe<sub>2</sub>O<sub>3</sub>), is a promising visible-light-active photocatalyst owing to its narrow band gap (~2.1 eV), earth abundance, low toxicity, and chemical stability (Kay et al., 2006; Kumar et al., 2022; Arif et al., 2024). Nevertheless, the photocatalytic performance of Fe<sub>2</sub>O<sub>3</sub> is often hindered by poor charge carrier separation and particle aggregation, which significantly reduce its efficiency (Sivula et al., 2011). Coupling Fe<sub>2</sub>O<sub>3</sub> with a suitable support material represents an effective strategy to overcome these drawbacks by improving dispersion and interfacial charge transfer (Ren and Innocenzi, 2021).

Combining Fe<sub>2</sub>O<sub>3</sub> with hBN to form heterostructured composites offers the potential to integrate the visible-light absorption capability of Fe<sub>2</sub>O<sub>3</sub> with the excellent stability and charge-mediation properties of hBN. Despite this promise, Fe<sub>2</sub>O<sub>3</sub>-hBN systems remain insufficiently explored, particularly at ultra-low Fe<sub>2</sub>O<sub>3</sub> loadings where high dispersion and efficient interfacial charge separation are expected to play a critical role. Moreover, systematic investigations addressing the degradation of chemically distinct pollutants using such composites under solar irradiation are still limited.

In our previous work, we demonstrated the potential of 2D hBN as an effective support for visible-light-active photocatalysts, established by Co<sub>3</sub>O<sub>4</sub>-decorated hBN composites showing enhanced degradation of methylene blue (MB) and ibuprofen (IBP) under visible-light irradiation due to improved interfacial charge separation (Bouziani et al., 2025). Building on these findings, the present study focuses on Fe<sub>2</sub>O<sub>3</sub>-modified hBN composites as an alternative, iron-based photocatalytic system. MB, a model cationic dye, and IBP, a widely detected pharmaceutical contaminant with different physicochemical properties and degradation pathways, were selected to evaluate the versatility of the photocatalyst.

In this work, Fe<sub>2</sub>O<sub>3</sub>-hBN composites were synthesized via a facile dry impregnation approach and systematically characterized in terms of structural, optical, and electronic properties. Their photocatalytic performance toward the solar-driven degradation of MB and IBP was investigated, and the effects of Fe<sub>2</sub>O<sub>3</sub> loading were analyzed to identify the optimal composition. In addition, radical scavenger experiments were conducted to elucidate the dominant reactive species and gain insight into the photocatalytic mechanism. This study provides a deeper understanding of Fe<sub>2</sub>O<sub>3</sub>-hBN heterojunctions and highlights their potential for visible-light-driven removal of hazardous organic pollutants from water.

Recent studies have explored hBN-supported metal oxides such as Co<sub>3</sub>O<sub>4</sub>-hBN and Fe<sub>2</sub>O<sub>3</sub>-hBN, demonstrating that the incorporation of transition-metal oxides onto hBN can enhance visible-light activity through improved charge separation and interfacial electron transport (Bouziani et al., 2025; Sheng et al., 2018). However, most reported systems rely on relatively high oxide loadings, which often lead to nanoparticle agglomeration and reduced photocatalytic efficiency (Ashraf et al., 2024). Moreover, the photocatalytic behavior of Fe<sub>2</sub>O<sub>3</sub>-hBN composites, particularly at ultra-low Fe<sub>2</sub>O<sub>3</sub> contents (<1 wt %), has not been systematically investigated. Compared to these reports, the present work introduces a low-loading Fe<sub>2</sub>O<sub>3</sub>-hBN composite that achieves efficient degradation of both MB and IBP under simulated sunlight. These gaps and challenges in existing literature highlight the innovation and significance of the Fe<sub>2</sub>O<sub>3</sub>-hBN system developed here.

## 2. Materials and methods

Iron(III) nitrate nonahydrate (Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O) and hexagonal boron nitride (hBN, 99.9 % metal basis) were obtained from Aldrich and used

as received. Ibuprofen (IBP, ≥98 %) and methylene blue (MB, 97 %) were employed as representative organic contaminants in the photocatalytic experiments and were also sourced from Aldrich.

### 2.1. Materials synthesis

The Fe<sub>2</sub>O<sub>3</sub>-hBN composite was prepared using a dry impregnation method adapted from previously reported procedures for metal-oxide deposition on solid supports, with minor modifications to control the Fe<sub>2</sub>O<sub>3</sub> loading and dispersion on hBN (Bartholomew and Farrauto, 2010). A series of Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O precursor solutions was prepared to achieve Fe<sub>2</sub>O<sub>3</sub> loadings of 0.2, 0.5, 1.0, and 2.0 wt %. These solutions were gradually added to 1 g hBN in small volumes. After each addition, the partially wet samples were kept in the oven at 100 °C for 15 min. This stepwise impregnation and drying process was repeated until the entire precursor solution had been added. After the final addition, the obtained materials were kept at 100 °C overnight, then calcinated at 500 °C for 3 h to obtain Fe<sub>2</sub>O<sub>3</sub>-hBN composite. Fe<sub>2</sub>O<sub>3</sub> was obtained by dehydrating Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O at 120 °C for 2 h, followed by calcination at 500 °C for 3 h.

### 2.2. Characterization

The structural, optical, and textural properties of pristine h-BN and Fe<sub>2</sub>O<sub>3</sub>-hBN composites were investigated using complementary characterization techniques. Crystalline features were analyzed by powder X-ray diffraction. Phase assignment was performed by matching the experimental diffraction patterns with reference files from the International Centre for Diffraction Data database. The reflections were indexed to hexagonal boron nitride (h-BN; PDF 34–0421) and hematite (α-Fe<sub>2</sub>O<sub>3</sub>; PDF 33–0664). Fe<sub>2</sub>O<sub>3</sub>, hBN, and 0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN composite (the most performing photocatalyst) crystallite size (d) was determined from the Scherrer formula (Eq.1) (Shiraiishi and Inagaki, 2003)

$$d = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

where K, λ, β, θ represent shape-related constants, X-ray wavelength (0.15406 nm), full width at half maximum (FWHM), and Bragg angle, respectively.

The optical absorption behavior and charge-carrier recombination characteristics were examined by UV–Vis diffuse reflectance spectroscopy and photoluminescence spectroscopy, respectively. The bandgap energy (E<sub>g</sub>) was obtained from the Kubelka-Munk function, as depicted in Eq. (2) (Jaramillo-Páez et al., 2017):

$$A(h\nu - E_g)^{\frac{n}{2}} = ah\nu \quad (2)$$

Specific surface area and porosity were determined from nitrogen adsorption-desorption measurements following sample outgassing. Morphological features and particle dispersion were evaluated using scanning and transmission electron microscopy. Surface functional groups and bonding environments were identified by Fourier-transform infrared spectroscopy. Additional experimental details, instrumental parameters, and extended characterization data are provided in the Supporting Information.

### 2.3. Photocatalytic tests

Photocatalytic degradation experiments were conducted using hBN and Fe<sub>2</sub>O<sub>3</sub>-hBN compounds with varying Fe<sub>2</sub>O<sub>3</sub> loadings under simulated solar irradiation. Photocatalytic tests were conducted in a batch reactor employing a 250 mL Pyrex reactor, externally shielded with aluminum foil. For each run, 100 mL of an aqueous solution of MB or IBP at an initial concentration of 10 ppm was used, with a photocatalyst dosage of 1 g/L. Irradiation was provided by an Osram Ultra-Vitalux lamp (300 W), which emits a solar-like spectrum, placed 10 cm above

the solution surface. The reactor was equipped with a circulating water jacket to maintain a constant temperature throughout the reaction. Prior to light exposure, the suspension was magnetically stirred in the dark for 20 min to establish adsorption-desorption equilibrium. Photocatalytic reactions were then carried out for 120 min. At predetermined time intervals, 1.5 mL aliquots were withdrawn, filtered through a 0.45  $\mu\text{m}$  regenerated cellulose (RC) membrane, and analyzed. MB concentration was quantified by monitoring the absorbance at  $\lambda_{\text{max}} = 664 \text{ nm}$  using UV-Vis spectroscopy, whereas IBF concentration was determined by high-performance liquid chromatography (HPLC).

HPLC analyses were performed on a Shimadzu LC-20AD system equipped with dual pumps, an autosampler (SIL-20AC), a column oven (CTO-20A), a photodiode array detector (SPD-M20A), and a refractive index detector (RID-10A). Chromatography analysis was done via a C18 reversed-phase column ( $4.6 \times 150 \text{ mm}$ ,  $5 \mu\text{m}$ ) with a mobile phase consisting of acidified water (pH 2, adjusted with  $\text{H}_3\text{PO}_4$ ) and methanol in a 1:4 (v/v) ratio at a flow rate of 1.0 mL/min. The column temperature was kept at  $50^\circ\text{C}$ , the injection volume was  $20 \mu\text{L}$ , and IBF detection was carried out at  $223 \text{ nm}$  using the PDA detector. Data acquisition and processing were carried out with Shimadzu LabSolutions software. To ensure direct comparability of photocatalytic performance between  $\text{Fe}_2\text{O}_3$  loadings and to focus on the structure-activity relationship, all degradation experiments were carried out under fixed conditions (natural pH, identical catalyst dosage, and identical initial pollutant concentrations). Photolysis experiments under similar irradiation conditions were performed, resulting in negligible degradation of both MB and IBP, confirming the stability of these organic species under simulated solar light illumination.

All photocatalytic degradation experiments were carried out in triplicate under identical conditions, and the results are presented as

mean  $\pm$  standard deviation.

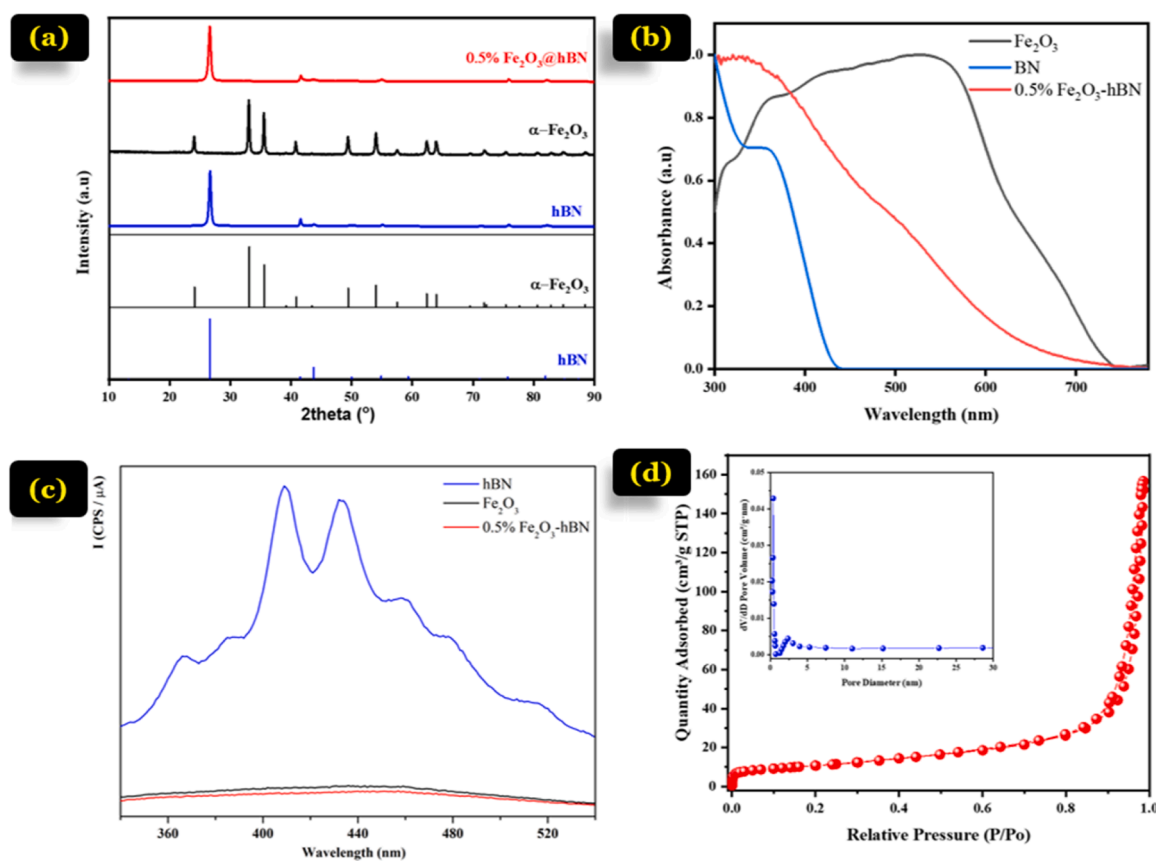
## 2.4. Identification of active species

The dominant reactive species involved in the photocatalytic degradation were examined through scavenger-assisted experiments. Specific quenchers were introduced into the reaction system to selectively suppress individual charge carriers or reactive oxygen species. Potassium iodide (0.3 mM), chloroform (2.4 mM), and isopropyl alcohol (0.5 mM) were employed to inhibit photogenerated holes ( $h^*$ ), superoxide radicals ( $\bullet\text{O}_2^-$ ), and hydroxyl radicals ( $\bullet\text{OH}$ ), respectively. All experiments were conducted under identical photocatalytic conditions using methylene blue as the target pollutant and the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite as the catalyst, with the only modification being the addition of the corresponding scavenger.

## 3. Results and discussion

### 3.1. Characterization

The crystallinity and phase composition of pure  $\text{Fe}_2\text{O}_3$ , hBN, and the  $\text{Fe}_2\text{O}_3$ -hBN composites were investigated by X-ray diffraction (XRD). XRD patterns are shown in Fig. 1a. The XRD pattern of hBN displays characteristic peaks at approximately  $26.6^\circ$ ,  $41.5^\circ$ ,  $43.6^\circ$ ,  $50.2^\circ$ , and  $55.1^\circ$ , corresponding to the (002), (100), (101), (102), and (004) crystallographic planes of hexagonal boron nitride (hBN), respectively (Zhang et al., 2017). Furthermore, the XRD pattern of  $\text{Fe}_2\text{O}_3$  reveals peaks at around  $24.1^\circ$ ,  $33.2^\circ$ ,  $35.6^\circ$ ,  $40.9^\circ$ ,  $49.5^\circ$ ,  $54.0^\circ$ , and  $62.5^\circ$ , which are associated to the (012), (104), (110), (113), (024), (116), and (214) planes of hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) (Liu et al., 2020). These peaks align



**Fig. 1.** (a) XRD patterns of  $\alpha\text{-Fe}_2\text{O}_3$ , hBN, and 0.5 %  $\alpha\text{-Fe}_2\text{O}_3$ -hBN. Reference data for hBN (JCPDS no. 34-0421) and  $\alpha\text{-Fe}_2\text{O}_3$  (JCPDS no. 33-0664) are included for comparison. (b) UV-vis spectra of  $\alpha\text{-Fe}_2\text{O}_3$ , hBN and 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN. (c) PL spectra of hBN,  $\text{Fe}_2\text{O}_3$  and 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN. (d) Nitrogen adsorption-desorption isotherm of 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite. Inset: pore size of 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN.

with the reference data for  $\alpha$ - $\text{Fe}_2\text{O}_3$  and confirm that hematite is the dominant phase present. Moreover, the XRD pattern of 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite showed only hBN diffraction patterns, because of the low content of  $\text{Fe}_2\text{O}_3$  (below the typical detection limit of XRD) and high dispersion, as confirmed by the TEM-EDS and FESEM results (Figs. 2 and 3). The average crystallite sizes were calculated using the Scherrer equation, based on the most intense diffraction peaks: the (002) peak for hBN and the (104) peak for  $\text{Fe}_2\text{O}_3$ , and are listed in Table 1. It is noteworthy to mention that the crystallite size remained similar following the addition of  $\text{Fe}_2\text{O}_3$ , indicating effective dispersion of iron oxide on hBN.

The UV-Vis absorbance spectra of  $\text{Fe}_2\text{O}_3$ , hBN, and the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite (Fig. 1b) reveal distinct differences in light absorption properties. Pristine  $\text{Fe}_2\text{O}_3$  exhibits strong absorption across a wide range, extending into the visible region up to 650 nm, attributed to charge transfer transitions between O 2p orbitals and Fe 3d states, consistent with its relatively small band gap ( $\sim 2.1$  eV) (Boudjemaa et al., 2009). In contrast, hBN shows almost no absorption beyond 400 nm, reflecting its wide band gap and UV-limited activity (Chou et al., 2022). The 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite displays an absorption profile similar to  $\text{Fe}_2\text{O}_3$ , with a slight red shift, indicating successful incorporation of  $\text{Fe}_2\text{O}_3$  onto the hBN matrix. While these results suggest that  $\text{Fe}_2\text{O}_3$  is the primary contributor to visible light harvesting, the presence of hBN may enhance the separation of photogenerated charges in the composite, potentially improving photocatalytic performance (Shenoy et al., 2021). The bandgap energies of hBN,  $\text{Fe}_2\text{O}_3$ , and 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN were estimated from the Kubelka-Munk function, assuming an indirect Eg of hBN is around 5.4 eV (Cassabois et al., 2016; Watanabe et al., 2004; Tarrío and Schnatterly, 1989), while  $\text{Fe}_2\text{O}_3$  displays an Eg  $\approx$  of 2.1 eV (Young et al., 2013; Ahn et al., 2022). Photoluminescence (PL) spectroscopy was performed to probe the optical properties and defect-related emissions in bare hBN,  $\text{Fe}_2\text{O}_3$ , and 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN

composites. 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN showed a suppression of PL intensity in comparison to hBN and  $\text{Fe}_2\text{O}_3$ , as shown in Fig. 1c. This is consistent with previous reports on PL quenching in metal-oxide/h-BN composites (Zhou et al., 2022). The reduced emission is attributed to charge transfer or non-radiative recombination facilitated by Fe species, which may act as electron traps or introduce surface states that deactivate the emissive centers in hBN (Mani et al., 2020). Although Fe species were not detected in the XPS spectrum or XRD pattern, which is reasonably attributed to the low  $\text{Fe}_2\text{O}_3$  loading (0.5 wt %), the photoluminescence (PL) spectra of the  $\text{Fe}_2\text{O}_3$ -hBN composite exhibit features consistent with those reported for  $\alpha$ - $\text{Fe}_2\text{O}_3$ -based systems, particularly in terms of absorption-emission behavior. Compared to pristine hBN, the  $\text{Fe}_2\text{O}_3$ -hBN composite shows a noticeable PL quenching effect, indicating a more efficient separation of photogenerated charge carriers.

The suppression of PL intensity suggests a reduction in radiative electron-hole recombination, which can be attributed to the formation of an interfacial charge-transfer pathway between  $\text{Fe}_2\text{O}_3$  and hBN. In this heterojunction system, photogenerated electrons in  $\text{Fe}_2\text{O}_3$  are transferred toward the hBN support, while holes remain in the valence band of  $\text{Fe}_2\text{O}_3$ , thereby inhibiting recombination processes. This interpretation is consistent with the enhanced photocatalytic performance observed for the  $\text{Fe}_2\text{O}_3$ -hBN composite under visible-light irradiation. A more detailed discussion of the band alignment and charge-transfer mechanism is provided in the subsequent Mechanism section.

The nitrogen adsorption-desorption isotherms of  $\text{Fe}_2\text{O}_3$ , hBN, and the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite show distinct differences in surface area and porosity. hBN has the highest adsorption capacity and surface area, displaying a Type IV isotherm with an H3 hysteresis loop characteristic of mesoporous structures and mainly micropores ( $< 2$  nm) (Fig.S2).  $\text{Fe}_2\text{O}_3$  exhibits lower adsorption, a broader pore size distribution centered around 20- 40 nm, and a less uniform mesoporous network due to particle agglomeration (Fig.S3). The 0.5 %  $\text{Fe}_2\text{O}_3$ /hBN composite

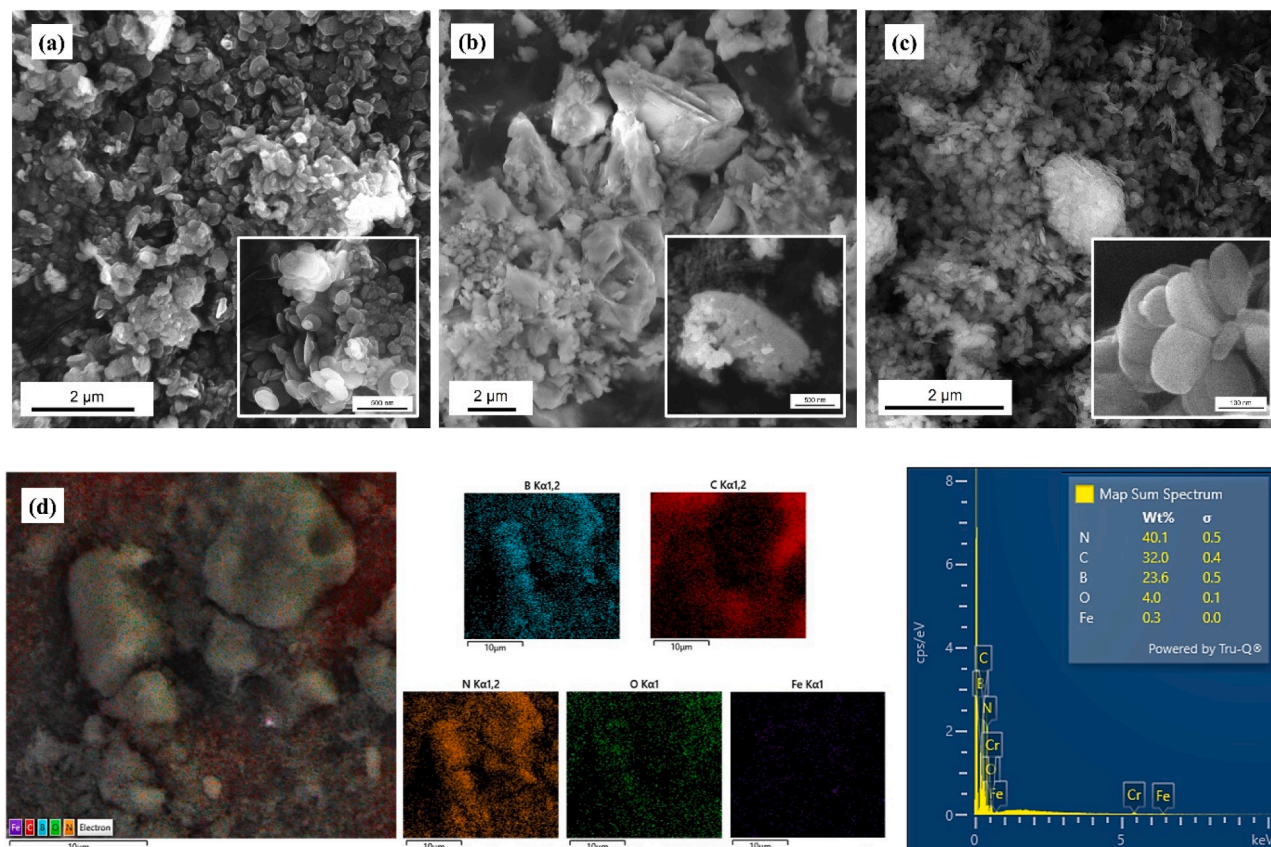


Fig. 2. (a) FESEM images of hBN, (b)  $\text{Fe}_2\text{O}_3$ , (c) 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN, (d) elemental mapping and EDS spectrum of 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN.

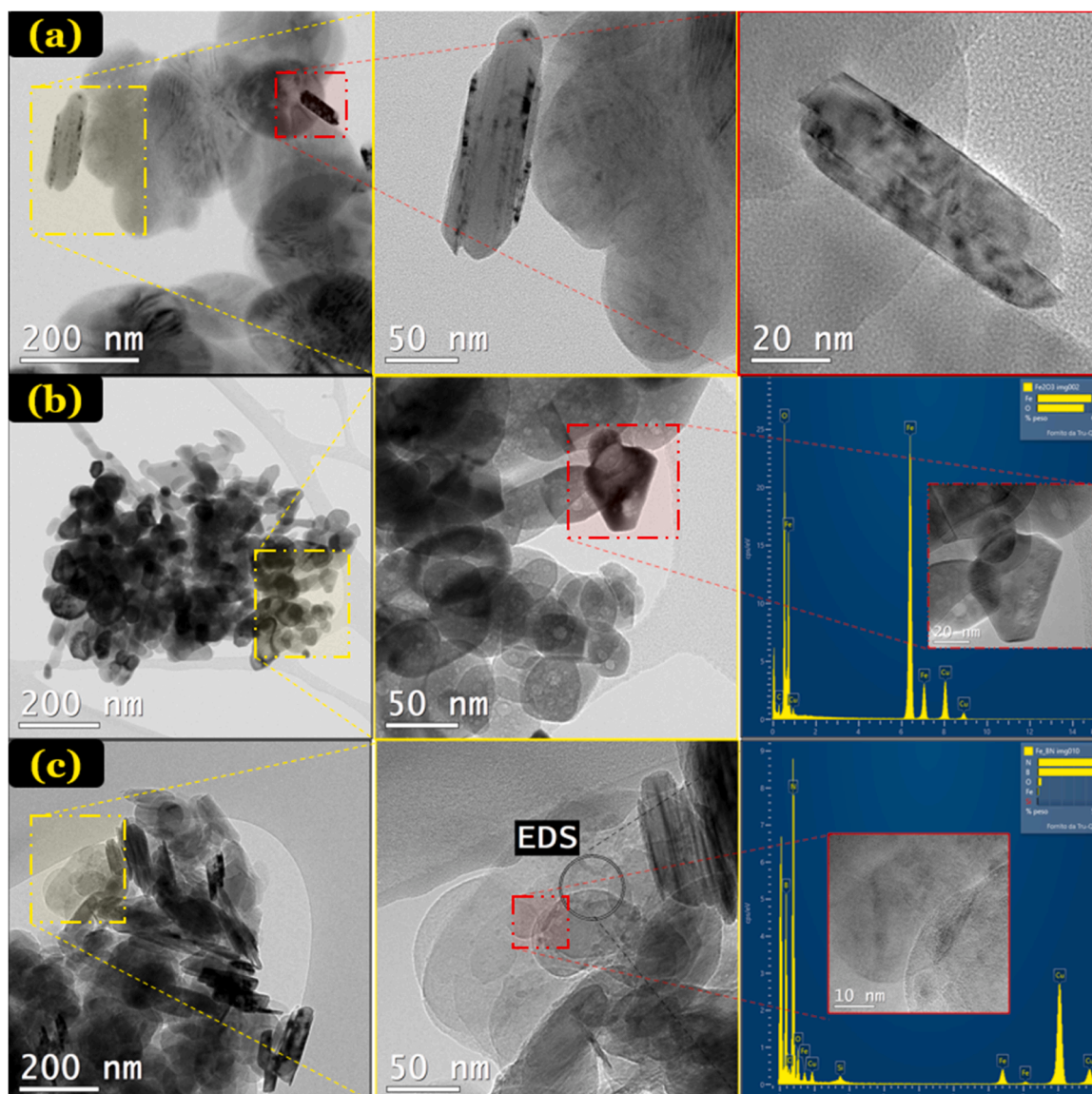


Fig. 3. (a) TEM images of hBN, and TEM images and EDS spectrum of (b)  $\text{Fe}_2\text{O}_3$ , and (c) 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN.

**Table 1**  
Physico-chemical properties of hBN,  $\text{Fe}_2\text{O}_3$ , and 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN.

Samples	Crystallite size D (nm)	$S_{\text{BET}}$ ( $\text{m}^2/\text{g}$ )	Pore volume ( $\text{cm}^3/\text{g}$ )	Pore size (nm)	$E_g$ (eV)
hBN	28.0	32.0	0.21	25.0	5.3
$\text{Fe}_2\text{O}_3$	39.3	15.2	0.10	22.1	2.1
0.5 % $\text{Fe}_2\text{O}_3$ - hBN	30.0	38.0	0.24	22.0	2.7

maintains the mesoporous nature of hBN but with slightly reduced surface area ( $38.0 \text{ m}^2/\text{g}$ ) and increased pore volume ( $0.24 \text{ cm}^3/\text{g}$ ), indicating well-dispersed  $\text{Fe}_2\text{O}_3$  that introduces additional meso-porosity without blocking pores (Fig.1d). Pure  $\text{Fe}_2\text{O}_3$  has a BET surface area of  $15.2 \text{ m}^2/\text{g}$ , pore volume  $0.10 \text{ cm}^3/\text{g}$ , and larger pores ( $\sim 22.1 \text{ nm}$ ), while pure hBN shows  $32.0 \text{ m}^2/\text{g}$  surface area,  $0.21 \text{ cm}^3/\text{g}$  pore volume, and pores ( $\sim 25 \text{ nm}$ ). Overall,  $\text{Fe}_2\text{O}_3$  addition increases surface area but enhances pore volume and retains the mesoporous structure, expectedly beneficial for catalytic applications.

The FTIR spectra of  $\alpha\text{-Fe}_2\text{O}_3$ , hBN, and the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite are given in Fig.S4. The bare hBN spectrum shows two prominent

peaks in the range of  $1300 - 800 \text{ cm}^{-1}$ , corresponding to the B-N stretching and B-N-B bonding modes, typical of hBN (Zhang et al., 2020). In the case of  $\alpha\text{-Fe}_2\text{O}_3$ , characteristic peaks at around  $550 \text{ cm}^{-1}$  are observed, which are attributed to the Fe-O stretching vibrations in the hematite structure (Battisha et al., 2006). For the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite, the infrared spectrum was dominated by the characteristic vibrational modes of h-BN, with bands observed at approximately  $1400 \text{ cm}^{-1}$  corresponding to B-N stretching and near  $800 \text{ cm}^{-1}$  assigned to B-N bending vibrations. No distinct absorption features attributable to  $\text{Fe}_2\text{O}_3$  were detected. This observation is reasonably attributed to the extremely low iron oxide loading, which likely falls below the sensitivity limit of FTIR spectroscopy, resulting in signals too weak to be resolved against the intense h-BN framework bands (Nakamoto, 2008).

The general morphology of pristine hBN and  $\text{Fe}_2\text{O}_3$  materials, as well as that of the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite, was investigated by field emission SEM (FESEM). hBN (Fig.2a) exhibits a layered structure, typical of the hexagonal boron nitride material (Kar et al., 2021), whereas  $\text{Fe}_2\text{O}_3$  (Fig.2b) appears as made up of aggregated (nano)particles, with a lot of irregularity in both shape and size for the aggregates. In the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite (Fig.2c),  $\text{Fe}_2\text{O}_3$  nanoparticles are expected to be well-dispersed across the BN matrix, but this

morphological feature is hardly visible in the FESEM images, even at high magnification (see the inset to Fig. 2c). This might, in any case, suggest that the composite synthesis successfully prevented  $\text{Fe}_2\text{O}_3$  agglomeration and will be further confirmed by TEM (Fig. 3). The elemental mapping and the EDS spectrum (Fig. 2d) indicate the targeted loading is achieved and the presence of all the expected elements (Fe, B, N, O, and C) in the composite, confirming the successful addition of  $\text{Fe}_2\text{O}_3$  into the hBN matrix.

Electron microscopy imaging studies at the nanoscale through TEM (and EDS analyses thereof) reveal the successful incorporation of  $\text{Fe}_2\text{O}_3$  nanoparticles into the BN matrix. TEM images of BN (Fig. 3a) show well-organized, layered sheets with a general hexagonal shape, whereas the  $\text{Fe}_2\text{O}_3$  sample (Fig. 3b) exhibits typical nanoparticle aggregation, with particle sizes ranging in the nanoscale, from 10 to 50 nm. The EDS spectrum confirms its elemental composition made of Fe and O, as expected. In the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite (Fig. 3c),  $\text{Fe}_2\text{O}_3$  nanoparticles are well dispersed across and on top of the BN sheets, indicating the successful synthesis that prevented aggregation among the  $\text{Fe}_2\text{O}_3$  nanoparticles themselves. Moreover, the EDS spectrum supports the presence of the expected elements (B, N, Fe, and O) at the nanoscale, indicating the highly uniform nature of the material.

XPS was used to investigate the surface chemical composition of the hBN and 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite. In both samples (Fig. 4a and Fig. S5), the deconvolution of B 1 s exhibited a primary peak at  $\sim 190.5$  eV corresponding to B-N bonding, confirming the presence of hBN. A secondary peak at  $\sim 192.0$  eV was observed in both samples and is attributed to oxidized boron (B-O), with slightly higher intensity in the  $\text{Fe}_2\text{O}_3$ -hBN, suggesting enhanced surface oxidation or possible interaction with Fe species (Tay et al., 2014; Liu et al., 2022). Similarly, the

deconvolution of the N 1 s spectrum displayed a peak at  $\sim 398.5$ – $399.5$  eV in both samples, attributed to  $\text{sp}^2$ -hybridized nitrogen in the BN lattice, while a minor shoulder at  $\sim 401$  eV was more pronounced in the composite may result from Fe-induced surface states or N–O bonding (Tay et al., 2014). The C 1 s spectrum of bare h-BN revealed a single peak at  $\sim 284.5$  eV due to adventitious carbon (Barr and Seal, 1995; Han et al., 2019), whereas the composite displayed additional features at  $\sim 286.0$  and  $\sim 288.5$  eV, associated with C–O and  $\text{CO}_3^{2-}$  species, likely originating from surface hydroxylation or interaction with  $\text{Fe}_2\text{O}_3$  (Sheng et al., 2018; Han et al., 2019). Deconvolution of the O 1 s spectrum in  $\text{Fe}_2\text{O}_3$ -hBN (Fig. 4d) revealed a broad peak centered at  $\sim 530.1$  eV with a shoulder at  $\sim 531.8$  eV, attributed to lattice oxygen and surface hydroxyls, respectively (Ashraf et al., 2024). Despite the absence of a detectable Fe 2p signal consistent with low  $\text{Fe}_2\text{O}_3$  loading (0.5 wt %) and surface sensitivity limitations, the methodologies described in Biesinger et al. provide clear evidence that Fe signals often become attenuated or overlapped when Fe is encapsulated or present at low concentrations (Biesinger et al., 2011). The deconvolved spectra of B, N, and O core-levels still capture subtle chemical interactions, supporting the idea that  $\text{Fe}_2\text{O}_3$  incorporation modifies surface chemistry via mild oxidation and potential Fe-O-B or Fe-N interfacial interactions.

### 3.2. Photocatalytic experiments

#### 3.2.1. MB and Ibuprofen degradation

The photocatalytic degradation of methylene blue (MB) was evaluated using pristine  $\text{Fe}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ -hBN composites with different  $\text{Fe}_2\text{O}_3$  loadings (0, 0.2, 0.5, 1, and 2 wt %) under simulated solar light irradiation. A preliminary screening of all  $\text{Fe}_2\text{O}_3$  loadings (0.2–2 wt %)

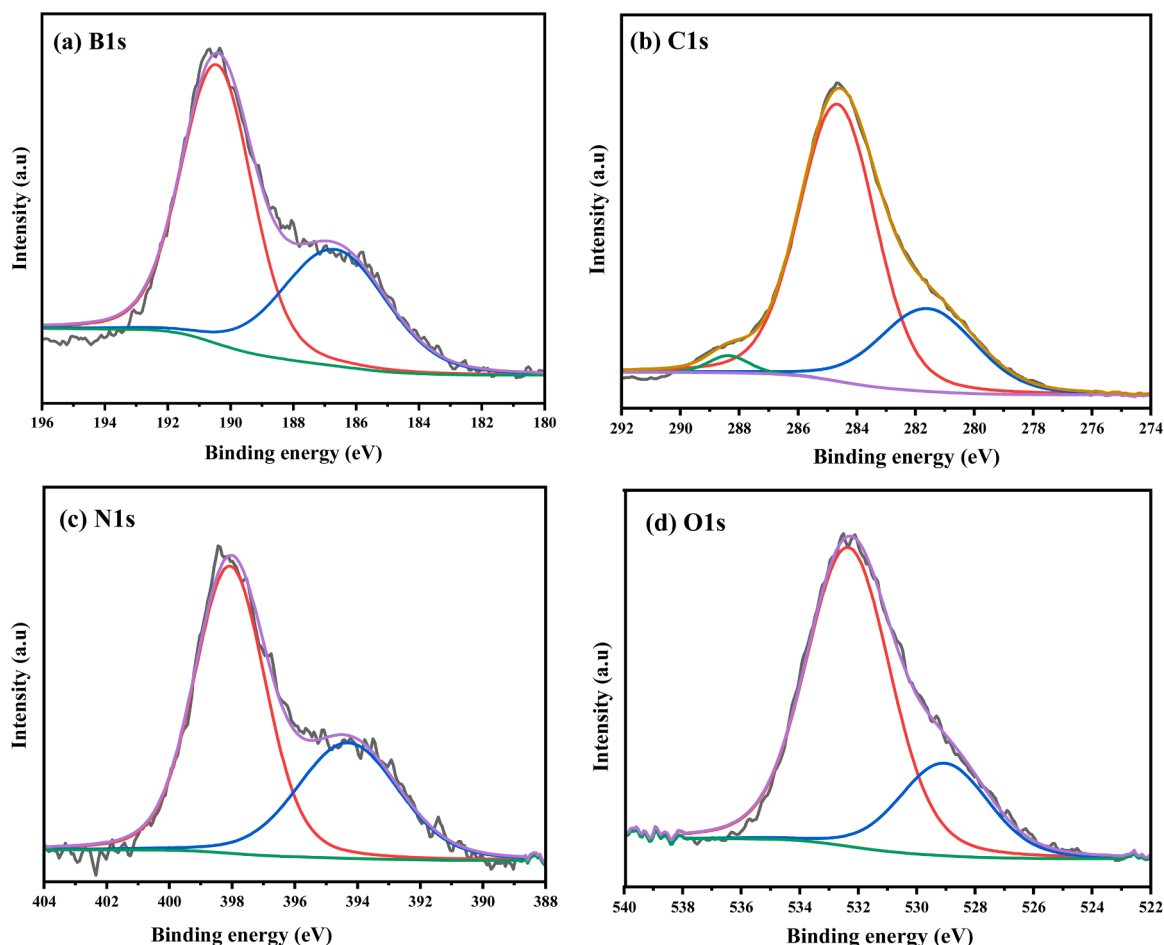


Fig. 4. High-resolution spectra of (a) B 1 s, (b), C 1 s, (c) N 1 s, and (d) O 1 s for 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite.

revealed a clear performance trend. The composite containing 0.2 wt %  $\text{Fe}_2\text{O}_3$  exhibited insufficient surface coverage and weak photocatalytic activity, whereas the 0.5 wt %  $\text{Fe}_2\text{O}_3$ -hBN sample achieved optimal dispersion and the highest degradation efficiency. In contrast, higher  $\text{Fe}_2\text{O}_3$  loadings (1–2 wt %) resulted in reduced photocatalytic performance, which can be attributed to partial nanoparticle agglomeration and light-shielding effects. Based on these results, detailed structural, optical, and mechanistic characterizations in this work were focused on the optimal 0.5 wt %  $\text{Fe}_2\text{O}_3$ -hBN composite.

As shown in Fig. 5, all  $\text{Fe}_2\text{O}_3$ -loaded hBN composites exhibited enhanced photocatalytic activity compared to pristine  $\text{Fe}_2\text{O}_3$  and bare hBN, indicating a clear synergistic interaction between  $\text{Fe}_2\text{O}_3$  and the hBN support. Among the tested samples, the 0.5 wt %  $\text{Fe}_2\text{O}_3$ -hBN demonstrated the highest photocatalytic efficiency, reaching approximately 80 % MB degradation within 120 min of simulated solar light irradiation.

The pseudo-first-order kinetic analysis (Fig. 5b) further supports these results, with the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN sample exhibiting the highest apparent rate constant, confirming the fastest degradation kinetics. This higher performance can be attributed to an optimum steadiness among number of active  $\text{Fe}_2\text{O}_3$  sites and high surface area of hBN, which maximizes interfacial contact and promotes efficient charge separation while suppressing electron-hole recombination.

When the  $\text{Fe}_2\text{O}_3$  loading was increased beyond this optimal value, a gradual decrease in photocatalytic efficiency was observed. This decline is attributed to two main factors: (i) increased particle-particle contact and local aggregation at higher  $\text{Fe}_2\text{O}_3$  contents, which reduces the effective  $\text{Fe}_2\text{O}_3$ -hBN interfacial area and enhances charge recombination, and (ii) partial surface coverage and light-shielding effects, which limit light penetration and reduce the accessibility of active sites. Conversely, at very low  $\text{Fe}_2\text{O}_3$  loading (0.2 %), the number of available active sites is insufficient to fully exploit the synergistic interaction, resulting in lower degradation efficiency.

A similar trend was observed for IBP photodegradation under simulated solar light irradiation. As demonstrated in Fig. 5c and Fig. 5d, the 0.5 %  $\text{Fe}_2\text{O}_3$ -hBN composite exhibited the highest performance, completing more than 70 % IBP removal, whereas pristine  $\text{Fe}_2\text{O}_3$  and hBN showed significantly lower efficiencies. These results further confirm the synergistic interaction between  $\text{Fe}_2\text{O}_3$  and hBN across pollutants with different chemical structures.

Control experiments performed under identical irradiation conditions in the absence of a catalyst resulted in only negligible photolysis for both MB and IBP, confirming that direct sunlight-driven degradation is minimal and that the observed removal originates predominantly from the photocatalytic activity of the  $\text{Fe}_2\text{O}_3$ -hBN composites.

To contextualize the photocatalytic activity of the optimized 0.5 wt

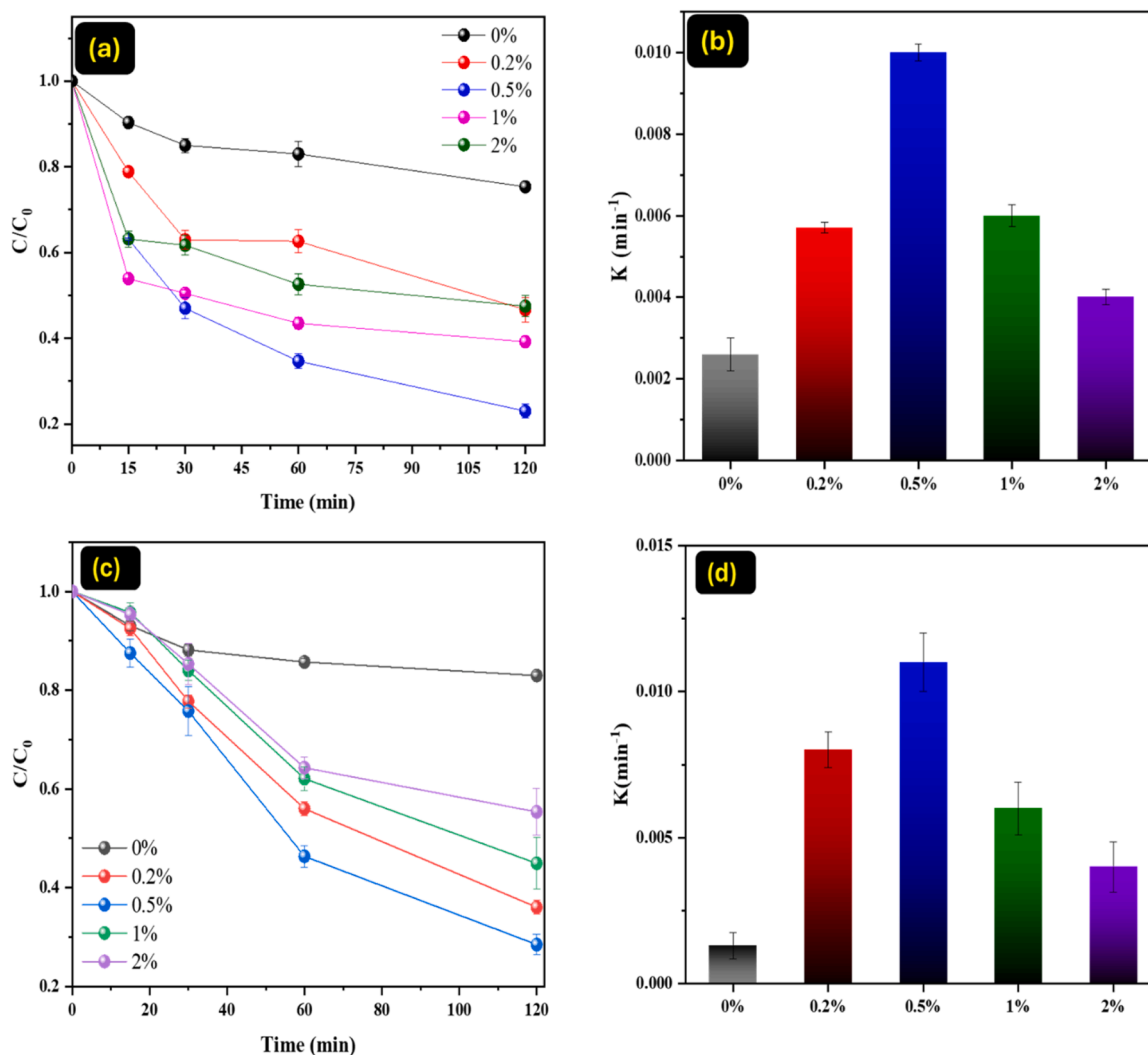


Fig. 5. Photocatalytic performance of the synthesized materials under simulated solar irradiation: (a) methylene blue degradation profiles for h-BN and  $\text{Fe}_2\text{O}_3$ -hBN composites; (b) corresponding apparent rate constants ( $k$ ,  $\text{min}^{-1}$ ); (c) ibuprofen degradation profiles in the presence of h-BN and  $\text{Fe}_2\text{O}_3$ -hBN composites; and (d) corresponding apparent rate constants ( $k$ ,  $\text{min}^{-1}$ ).

% Fe<sub>2</sub>O<sub>3</sub>-hBN composite, a comparison with representative Fe<sub>2</sub>O<sub>3</sub> and hBN-based photocatalysts reported in the literature is provided in Table 2. The Table shows that the Fe<sub>2</sub>O<sub>3</sub>-hBN composite exhibits competitive degradation performance under simulated solar-light irradiation compared to many reported Fe<sub>2</sub>O<sub>3</sub>-based heterojunctions, confirming its potential as an efficient photocatalyst for environmental purification.

### 3.2.2. Photocatalyst stability

To effectively exhibit the structural stability of the 0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN photocatalyst, the structural characteristics of the spent photocatalyst, after one photocatalytic cycle, were examined using XRD, with the results illustrated in Fig. 6a. The diffraction profile of the spent sample shows no obvious changes relative to that of the pristine composite, indicating that the crystalline framework remains intact during the photocatalytic process.

### 3.3. Role of reactive species in the degradation process

The role of reactive oxygen species in the MB photocatalytic degradation using the most promising photocatalyst (0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN) was investigated through scavenger experiments. Fig. 6b illustrates the effect of introducing selective radical scavengers on the photocatalytic degradation of methylene blue. For comparison, a reference experiment conducted in the absence of any scavenger was also included and showed an 80 % MB removal. The significant decrease in degradation efficiency upon the addition of CHCl<sub>3</sub>, a superoxide radical (O<sub>2</sub><sup>-•</sup>) scavenger, indicates that O<sub>2</sub><sup>-•</sup> plays a pivotal role in the photodegradation process. A similar trend was observed by the addition of IPA as a hydroxyl radical (OH) scavenger, though to a lesser extent, suggesting that •OH radicals contribute to the degradation mechanism as well. Moreover, the addition of KI, a hole (h<sup>+</sup>) scavenger, resulted in a marked decline in photocatalytic performance, underscoring the importance of photogenerated holes in the reaction. These findings suggest that the MB degradation in the presence of Fe<sub>2</sub>O<sub>3</sub>-hBN involves a synergistic effect of O<sub>2</sub><sup>-•</sup>, •OH radicals, and h<sup>+</sup>, with superoxide radicals and photogenerated holes being the dominant active species.

### 3.4. Photocatalytic reaction pathway using 0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN

Based on the scavenger-assisted tests, the photocatalytic behavior of the 0.5 wt % Fe<sub>2</sub>O<sub>3</sub>-hBN composite is mainly ruled by superoxide radicals (O<sub>2</sub><sup>-•</sup>) and photogenerated holes (h<sup>+</sup>), which play the dominant roles in the degradation process. The moderate decrease in photodegradation efficiency observed in the presence of isopropanol (IPA) indicates that hydroxyl radicals (•OH) also participate in the reaction, although to a lesser extent.

The relatively moderate photoluminescence (PL) quenching

observed for the 0.5 wt % Fe<sub>2</sub>O<sub>3</sub>-hBN composite is consistent with previous reports on low-loading metal oxide/2D-material heterojunctions, where enhanced photocatalytic activity arises mainly from interfacial charge transfer rather than pronounced changes in PL intensity (Sheng et al., 2018; Ashraf et al., 2024; Han et al., 2019). In such systems, the limited semiconductor content often results in modest PL sensitivity, while efficient charge migration across the heterointerface still effectively suppresses charge recombination and enhances photocatalytic performance.

Based on these results, a plausible photocatalytic degradation mechanism for MB is proposed in Fig. 7. To correlate the observed photocatalytic behavior with the electronic structure of the Fe<sub>2</sub>O<sub>3</sub>-hBN composite, the conduction band (CB) and valence band (VB) positions were estimated using the electronegativity method (Butler, 1977; Yang et al., 2016). Using the experimentally determined band gap of the composite (E<sub>g</sub> = 2.7 eV) and the absolute electronegativity of Fe<sub>2</sub>O<sub>3</sub> (X = 5.88 eV) (Yang et al., 2016), the CB and VB positions were calculated to be +0.03 eV and +2.73 eV versus the normal hydrogen electrode (NHE), respectively. These values are in good agreement with reported band-edge positions of α-Fe<sub>2</sub>O<sub>3</sub> (CB ≈ +0.3 eV, VB ≈ +2.4 eV).

In contrast, hBN exhibits a wide band gap of approximately 5.3 eV, with a much more negative conduction band (≈ -1.30 eV) and a deep valence band (≈ +4.00 to +4.20 eV) (Harikrishnan et al., 2024; Pakdel et al., 2014). This staggered band alignment facilitates efficient interfacial charge separation, whereby photogenerated electrons in Fe<sub>2</sub>O<sub>3</sub> are transferred to the more negative CB of hBN, enabling the reduction of dissolved oxygen to superoxide radicals (O<sub>2</sub><sup>-•</sup>), while the photogenerated holes remaining in the VB of Fe<sub>2</sub>O<sub>3</sub> participate in oxidation reactions leading to MB and IBP degradation. This charge-transfer pathway is fully consistent with the scavenger experiment results and explains the enhanced photocatalytic activity of the Fe<sub>2</sub>O<sub>3</sub>-hBN composite under visible-light irradiation (Fig. 7b).

Specifically, upon light irradiation, Fe<sub>2</sub>O<sub>3</sub> absorbs photons and generates electron-hole pairs via excitation of electrons from the VB to the CB (Eq. (3)). The photogenerated electrons are subsequently transferred to hBN and react with dissolved oxygen to form O<sub>2</sub><sup>-•</sup> radicals (Eq. (4)). Simultaneously, the photogenerated holes can oxidize water molecules to produce •OH radicals (Eq. (5)) or react with hydroxide ions (OH<sup>-</sup>) to generate additional •OH species (Eq. (6)). These reactive oxygen species (O<sub>2</sub><sup>-•</sup> and •OH), together with the direct oxidative action of h<sup>+</sup>, synergistically attack and degrade MB molecules into smaller intermediate products and ultimately mineralized species (Eq. (7)).

In summary, the photocatalytic activity of 0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN is principally governed by the generation of O<sub>2</sub><sup>-•</sup>, with photogenerated h<sup>+</sup> and •OH acting as secondary contributors to the MB degradation.



**Table 2**

Photocatalytic performances of the best material fabricated in the present manuscript and other data reported in the scientific literature.

Photocatalyst	Target pollutant	Light source	Key conditions	Photocatalytic performance	Reference
Fe <sub>2</sub> O <sub>3</sub> -hBN (0.5 wt %)	MB (10 mg l <sup>-1</sup> ), IBP (10 mg l <sup>-1</sup> )	Simulated sunlight (300 W)	100 mg catalyst, 120 min	MB ≈ 80 %; IBP ≈ 70 %	This work
α-Fe <sub>2</sub> O <sub>3</sub> nanoparticles	MB (10 mg l <sup>-1</sup> )	Visible light	50 mg, 120 min	~63 % MB degradation	(Sheng et al., 2018)
Fe <sub>2</sub> O <sub>3</sub> /hBN nanocomposite	RhB (10 mg l <sup>-1</sup> )	Visible light	30 mg, 120 min	~72 % RhB degradation	(Sheng et al., 2018)
Fe <sub>2</sub> O <sub>3</sub> /graphene oxide	MB (20 mg l <sup>-1</sup> )	Visible light	20 mg, 90 min	~80 % MB degradation	(Ashraf et al., 2024)
α-Fe <sub>2</sub> O <sub>3</sub> /g-C <sub>3</sub> N <sub>4</sub> heterostructure	Methyl orange (MO)	Visible light	Varied (see ref.)	Enhanced degradation vs. pristine components	(Khurram et al., 2022)
Z-scheme Fe <sub>2</sub> O <sub>3</sub> /g-C <sub>3</sub> N <sub>4</sub> composite	Organic dyes	Visible light	Varied (see ref.)	Improved photocatalytic activity via Z-scheme charge transfer	(Zhang et al., 2026)
α-Fe <sub>2</sub> O <sub>3</sub> /TiO <sub>2</sub>	MB (10 mg l <sup>-1</sup> )	Visible light	30 mg, 120 min	~85 % MB degradation	(Cao et al., 2020)
Fe <sub>2</sub> O <sub>3</sub> /CQDs	IBP (15 mg l <sup>-1</sup> )	Visible light	30 mg, 120 min	~76 % IBP degradation	(Zhang et al., 2020)
Pani-FeCuCeO <sub>2</sub>	MB (20 mg l <sup>-1</sup> )	Visible light	25 mg, 120 min	~79.2 % MB degradation	(Madi et al., 2025)
Fe <sub>2</sub> O <sub>3</sub> /MoS <sub>2</sub>	AB113 (20 mg l <sup>-1</sup> )	Visible light	30 mg, 45 min	~20 % AB113 degradation	(Bagheri and Chaibakhsh, 2021)
Fe <sub>2</sub> TiO <sub>5</sub> /rGO(10 %)	MB (10 mg l <sup>-1</sup> )	Visible light	50 mg 150 min	~55 % MB degradation	(Guediri et al., 2020)

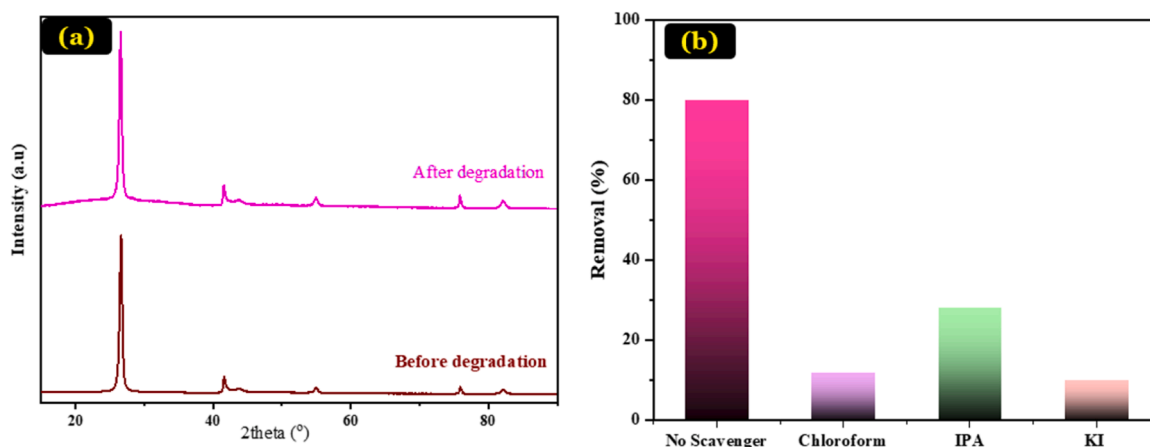


Fig. 6. a) XRD patterns of 0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN photocatalyst before and after photocatalytic reaction. b). Photocatalytic degradation of MB using a 0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN composite under simulated sunlight in the presence of scavengers: KI (0.3 mM), IPA (0.5 mM), and CHCl<sub>3</sub> (2.4 mM).

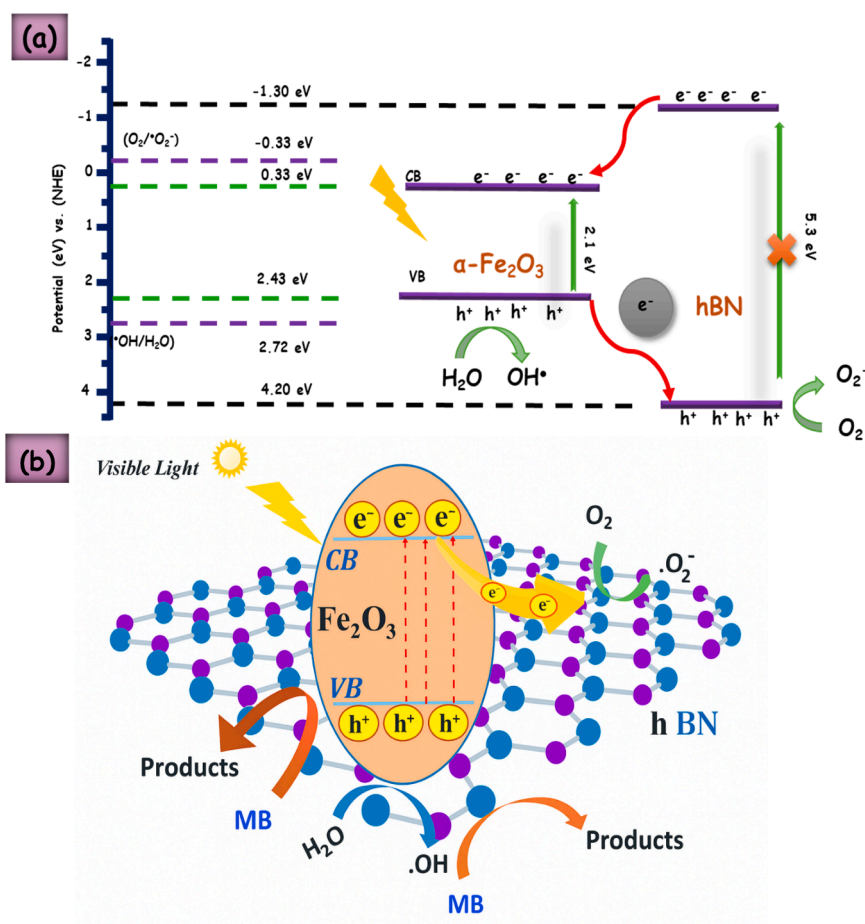
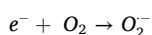
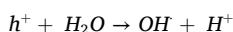


Fig. 7. (a) Schematic illustration of the band structure alignment of the Fe<sub>2</sub>O<sub>3</sub>-hBN composite; (b) proposed photocatalytic reaction pathway for methylene blue degradation over the 0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN photocatalyst.



(4) 4. Conclusion



(5)



(6)



(7)

In this work, Fe<sub>2</sub>O<sub>3</sub>-hBN composites with varying Fe<sub>2</sub>O<sub>3</sub> loadings were successfully synthesized via a dry impregnation method. Comprehensive characterization confirmed the effective incorporation and well dispersion of Fe<sub>2</sub>O<sub>3</sub> nanoparticles on the hBN matrix, resulting in modified surface chemistry and enhanced optical properties. Photocatalytic tests demonstrated that the 0.5 % Fe<sub>2</sub>O<sub>3</sub>-hBN composite

exhibited the highest efficiency for degrading both MB and IBP under simulated sunlight irradiation, attributed to an optimal balance between active site availability and the surface area of the support (hBN). Scavenger-assisted experiments indicate that superoxide radicals and photogenerated holes make the primary contributions to the degradation pathway, whereas hydroxyl radicals participate to a lesser extent. Overall, the Fe<sub>2</sub>O<sub>3</sub>-hBN composite represents a promising photocatalyst for simulated sunlight-driven environmental remediation, combining the advantageous properties of both Fe<sub>2</sub>O<sub>3</sub> and hBN to overcome their limitations. Despite the promising photocatalytic performance demonstrated under controlled laboratory conditions, several challenges must be addressed for practical application. The presence of competing ions, natural organic matter, and complex matrices in real wastewater may influence degradation efficiency and requires further investigation. In addition, efficient catalyst separation and recovery strategies, as well as extended multi-cycle reusability studies, are essential to evaluate long-term stability and economic feasibility. Future work will therefore focus on testing the Fe<sub>2</sub>O<sub>3</sub>-hBN composite in real wastewater systems and developing practical recovery approaches to facilitate its potential application in environmental remediation.

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### CRediT authorship contribution statement

**Mariam Bouziani:** Writing – original draft, Methodology, Investigation, Conceptualization. **Asmae Bouziani:** Writing – original draft, Investigation, Conceptualization. **Abdelghani Hsini:** Writing – review & editing, Investigation. **Claudia L. Bianchi:** Writing – review & editing, Resources. **Ermelinda Falletta:** Writing – review & editing, Resources, Investigation. **Giuseppina Cerrato:** Writing – review & editing, Resources, Investigation. **Alessia Giordana:** Writing – review & editing, Resources, Investigation. **Gökhan Çelik:** Writing – review & editing, Resources, Conceptualization. **Robert Hausler:** Supervision, Methodology.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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### Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.hazadv.2026.101064](https://doi.org/10.1016/j.hazadv.2026.101064).

### Data availability

Data will be made available on request.

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