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Ag-Fe₂O₃ nanohybrids for photocatalytic degradation and antibacterial activity

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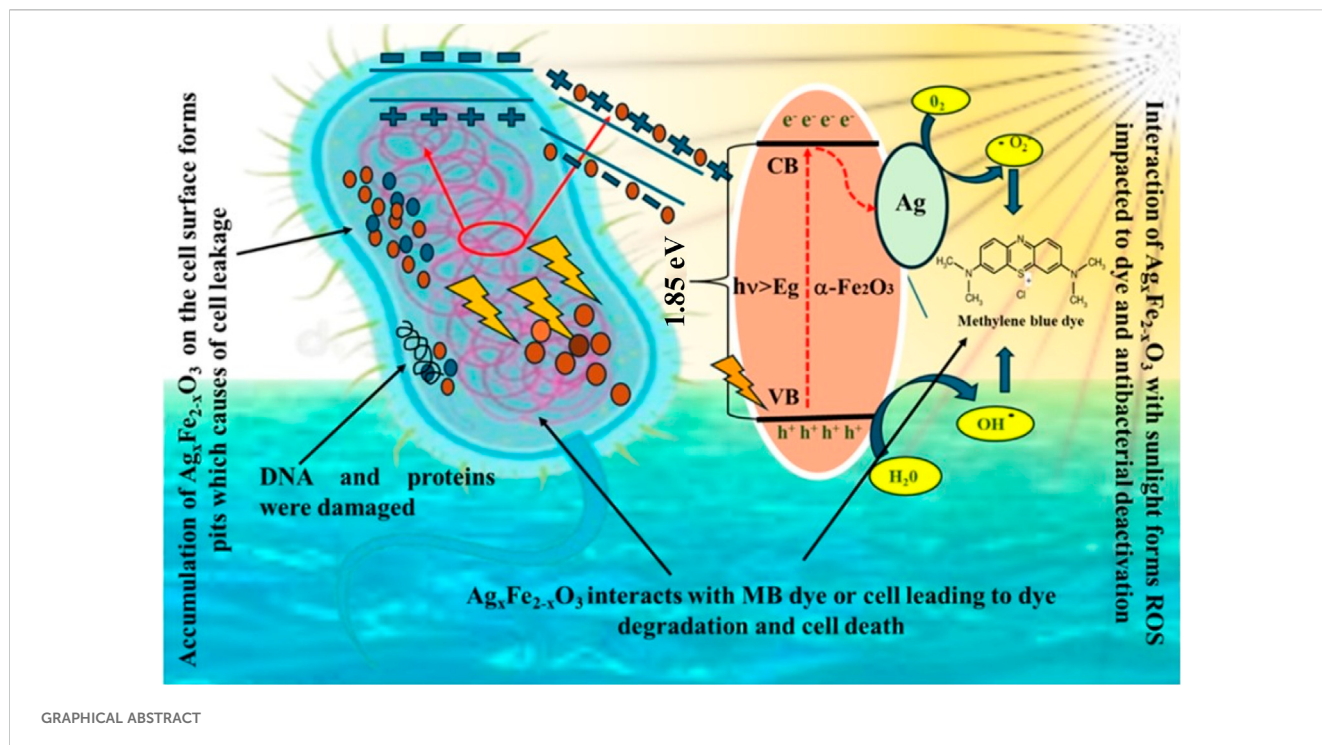
This study presents the synthesis and characterization of Ag_xFe_{2-x}O₃ nanoparticles as highly efficient photocatalysts and excellent antimicrobial agents. Ag_xFe_{2-x}O₃ nanoparticle synthesis was carried out through the sol-gel auto-combustion method. It was extensively characterized using X-ray diffraction (XRD), which confirmed the formation of crystalline α-Fe₂O₃ nanoparticles with a crystallite size of 22.23 nm. Ag-doped Fe₂O₃ has an average crystallite size of 23.08 nm. Fourier transform infrared (FTIR) spectroscopy provided insights about metal oxide bond formation and constant force for octahedral and tetrahedral sites, which suggest the location of the cation and anion. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) revealed rhombohedral Ag-doped Fe₂O₃ nanomaterials with a uniform particle size around 20–50 nm, which matches the XRD results. UV-vis spectroscopy shows absorption shifting toward visible light for the Ag_xFe_{2-x}O₃ compared to pristine Fe₂O₃, indicating the utilization of the maximum light spectrum for efficient light harvesting in the visible region. Ag_xFe_{2-x}O₃ exhibited excellent stability and reusability for visible-light-driven photocatalytic dye degradation with a rate constant of 1.27 × 10⁻² min⁻¹, which is two-fold more than that of the pristine Fe₂O₃ (0.51 × 10⁻² min⁻¹). Ag_xFe_{2-x}O₃ was also found to be more efficient in inhibiting the growth of *Proteus mirabilis* with a 16 mm zone of inhibition.

KEYWORDS

silver, photocatalysis, methylene blue, antimicrobial, plasmon, dyes

1 Introduction

The discharge of organic dyes from industrial processes into water bodies is a significant environmental concern due to their harmful effects on ecosystems and human health (Tkaczyk et al., 2020; Al-Tohamy et al., 2022; Belkhedkar et al., 2016; Mehra et al., 2021). These dyes, such as methylene blue, rhodamine, eosin yellow, crystal violet, etc., have been extensively used in textile, paper, leather, and cosmetic industries, and such dyes remain persistent and create potential toxicity (Tkaczyk et al., 2020; Al-Tohamy et al., 2022). Conventional wastewater treatment methods, including physical, biological, and chemical treatment, often fall short of effectively removing these pollutants (Piaskowski et al., 2018; Donkadokula et al., 2020).



E.K. Lipp et al. and Cabral et al. separately reported that microbes are also transmitted to humans and animals through contaminated water sources, causing diseases like cholera in humans and disruption in aquatic systems (Lipp et al., 2002; Cabral, 2010). Researchers have turned to photocatalytic degradation as a promising approach for addressing this issue. E. Cabiscol et al. reported that the method involves the utilization of semiconductor nanomaterials to generate reactive oxygen species (ROS) under light, which breaks down the dye molecules into harmless by-products (Cabiscol et al., 2000; Patra et al., 2015). ROS damage microbial cell membranes, proteins, and DNA, leading to the inactivation of pathogens. Hence, the photocatalytic process enables efficient water disinfection and reduces microbial load and organic dyes, which helps protect human and aquatic life.

There are various reported organic and inorganic semiconductor photocatalysts, such as pentacene quinone (PQ) (Pandit et al., 2014), graphitic carbon nitride ($\text{g-C}_4\text{N}_4$), zinc oxide (ZnO) (Pandit et al., 2014), tin oxide (SnO_2), tungsten oxide (WO_3), iron oxide (Fe_2O_3) (Jawale et al., 2021), and titanium dioxide (TiO_2) (Konstantinou and Albanis, 2004). Among these semiconductors, Fe_2O_3 has garnered significant attention due to its abundance, low cost, non-toxicity, and excellent chemical and thermal stability. With a bandgap of 1.8–2.0 eV (Konstantinou and Albanis, 2004; Uttam Pandit et al., 2024), Fe_2O_3 can absorb approximately 40% of the solar spectrum, making it a promising candidate for photocatalytic and antimicrobial applications (Dhlamini et al., 2017). However, its practical use is often limited by challenges such as poor electron conductivity, rapid electron-hole recombination, and slow oxygen evolution kinetics (Uttam Pandit et al., 2024). To overcome these limitations, researchers have explored functionalized strategies, such

as doping, heterostructure formation, and plasmonic enhancement to improve the photocatalytic and antimicrobial performance of Fe_2O_3 (Shinde et al., 2011; Mishra and Chun, 2015). These functionalization strategies not only enhance photocatalytic activity under visible light but also confer desirable characteristic such as stability, cost effectiveness, and non-toxicity.

Doping of Fe_2O_3 with transition metals such as Cu (Gao et al., 2012; Sun et al., 2021), Ti (Shams et al., 2019; Reveendran and Khadar, 2018), and Zn (Nikolic et al., 2013) has been shown to enhance its optical absorbance, electrical conductivity, and charge separation efficiency. Plasmonic loading is an effective strategy to further improve the photocatalytic performance of Fe_2O_3 under visible light. Various plasmonic-based photocatalysts include gold (Au), silver (Ag), palladium (Pd), and chromium (Cr). Au nanoparticles possess excellent plasmonic properties, but their abundance is low, and they are more expensive than Ag. Ag nanoparticles offer several advantages, such as cost-effectiveness and superior plasmonic efficiency in the visible light range. Ag-doped Fe_2O_3 nanomaterials have demonstrated impressive photocatalytic activity under visible light irradiation, particularly for the degradation of organic dyes like methylene blue (MB) (Bhowmik et al., 2020; Wang et al., 2018; Hitam and Jalil, 2020; Khalaji, 2022). V. K. Sharma et al. independently reported that Ag shows a broad spectrum of antimicrobial activity, which disrupts cell membranes, inhibits enzymatic functions, and interferes with DNA replication, leading to cell death (Araujo et al., 2021; Sharma et al., 2009). When Ag is doped with Fe_2O_3 , it enhances the charge transfer mechanism and reduces the electron activity to reduce microbial infections. The incorporation of Ag nanoparticles expands the absorption range of Fe_2O_3 into the visible region and promotes the effective generation and separation of electron-hole pairs, thereby enhancing overall photocatalytic and antimicrobial activity.

There are several reported methods for the synthesis of Ag-doped Fe_2O_3 , such as hydrothermal, microwave-assisted synthesis, electrochemical deposition, sonochemical synthesis, and the sol-gel auto-combustion method (Bera et al., 2014; Park et al., 2018). The sol-gel auto-combustion method gained popularity for synthesizing Ag-doped Fe_2O_3 -based nanomaterial due to its simplicity, cost-effectiveness, and ability to produce materials with controlled size, shape, and porosity. Additionally, this method allows the incorporation of doping and plasmonic materials, which enables the fabrication of advanced photocatalysts with enhanced Fe_2O_3 properties. For instance, Ag-doped Fe_2O_3 nanostructures have demonstrated remarkable applications in antibacterial, gas sensing, energy storage, water purification, biomedical, solar cell, and corrosion protection.

The present study focuses on the synthesis and characterization of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ ($X = 0.0000, 0.0025, 0.0075, \text{ and } 0.0125$) nanostructures using the sol-gel auto-combustion method, which shows excellent photocatalytic and antimicrobial activity. The photocatalytic degradation of methylene blue (MB) under visible light irradiation revealed that the Ag-doped 0.0125 sample exhibits the highest degradation up to 75% in 1 h, with a rate constant $1.270 \times 10^{-2} \text{ min}^{-1}$, which is nearly twice that of pristine Fe_2O_3 ($0.515 \times 10^{-2} \text{ min}^{-1}$). The antimicrobial activity of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ nanoparticles was evaluated against various bacteria and fungi strains, where $X = 0.0125$ demonstrates a significant inhibition zone of 16 mm for *Proteus mirabilis*, 14.8 mm for *E.coli*, 13.6 mm for *Candida*, 13.6 mm for *Bacillus subtilis*, and 13.8 mm for *Aspergillus niger* and *Staphylococcus aureus*. The $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ nanoparticles exhibit superior antibacterial efficiency against both Gram-positive and Gram-negative bacteria compared to fungal strains.

This work presents key advancements beyond existing Ag/ Fe_2O_3 systems as the optimal Ag concentration (Ag 0.0125 in $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$), maximizing both activities. It also provides a deeper mechanistic insight into the synergy of Ag SPR-enhanced visible light absorption, efficient charge separation at the Ag/ Fe_2O_3 interface, and ROS generation for exceptional combined performance (>95% MB degradation in 60 min plus significant antimicrobial efficacy) under optimized conditions, surpassing systems focused on single applications. We believe that combining this system with our other earlier reported photocatalyst materials will surely enhance photocatalytic activities (Pandit et al., 2025; Kumar Singh et al., 2024; Daphal et al., 2025; Pandit V. U. et al., 2022; Pandit V. U. et al., 2022; Pandit, 2022; Grouli et al., 2023; VRU Pandit, 2022; Jawale et al., 2022; Nevase et al., 2015).

2 Experimental methods

2.1 Chemicals used in the synthesis method

$\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ were ($X: 0.0000, 0.0025, 0.0075, \text{ and } 0.0125$) synthesized using the sol-gel auto-combustion method. The starting materials used in the synthesis were of pure and analytical grade, and no additional purification was performed. Ferric nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) was obtained from Himedia Laboratory LLC, USA (98.0%). Silver nitrate AgNO_3 was purchased from Loba Chemie PVT, Ltd. (99.9%). Citric acid ($\text{C}_6\text{H}_8\text{O}_7$) was purchased from Research Lab Fine Chem

Industries, India (99.0%). An ammonia solution of 25% extra pure NH_4OH was obtained from Loba Chemicals, PVT. Double-distilled water served as the solvent for the synthesis process.

2.2 Synthesis process

The Supplementary Figure S1 illustrates the detailed synthesis of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, which was carried out through a previously reported method (Uttam Pandit et al., 2024). The calculated amounts of ferric nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), silver nitrate (AgNO_3), citric acid ($\text{C}_6\text{H}_8\text{O}_7$), and ethylene glycol (CH_2OH)₂ were dissolved in deionized water. The pH of the resulting solution was adjusted to 9 by gradually adding ammonia solution. Once the pH reached 9, the solution was heated at a temperature of 80 °C–90 °C with continuous stirring. As a result, the solution transformed into a dried gel, which then underwent self-propagation to form a fluffy powder. The synthesized material was subsequently subjected to calcination at 550 °C. The yield of the product was around 89%. After calcination, the material underwent characterization using advanced instrumental techniques to analyze its properties. The calcinated material was employed for photocatalytic and antimicrobial applications.

2.3 Material characterizations

The synthesized samples were examined using various advanced analytical techniques. The crystallite size, X-ray density, and dislocation density were analyzed using a powder X-ray diffractometer (Rigaku Ultima IV) with a Cu-K α radiation source ($\lambda = 1.54 \text{ \AA}$), scanning in the 2θ range of 20°–80° at a rate of 5°/min. The functional group and bonding characteristics were identified through Fourier transform infrared (FTIR) spectroscopy using a Bruker Platinum ATR spectrometer. The optical properties, including the absorbance of the dye solution, were studied using a UV-visible spectrophotometer (Shimadzu UV-3600 and Shimadzu UV-800). The surface morphology was examined through scanning electron microscopy (SEM) using a Hitachi S3400N, and transmission electron microscopy (TEM) was performed using a JEOL JEM 2100 Plus instrument. Elemental composition was determined using energy-dispersion X-ray analysis (EDAX) integrated with the SEM.

2.4 Photocatalytic activity study

The photocatalytic activity of synthesized $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ ($X: 0.0000, 0.0025, 0.0075, \text{ and } 0.0125$) was studied through monitoring the degradation of MB dye as a function of time. In the experiment, 10 mg of the catalyst was dispersed in the reaction flask containing 100 mL of 10 ppm MB dye solution. To establish adsorption-desorption equilibrium between the dye and the catalyst, the mixture was stirred initially in the dark for 15 min. After equilibrium was achieved, the sample was exposed to sunlight

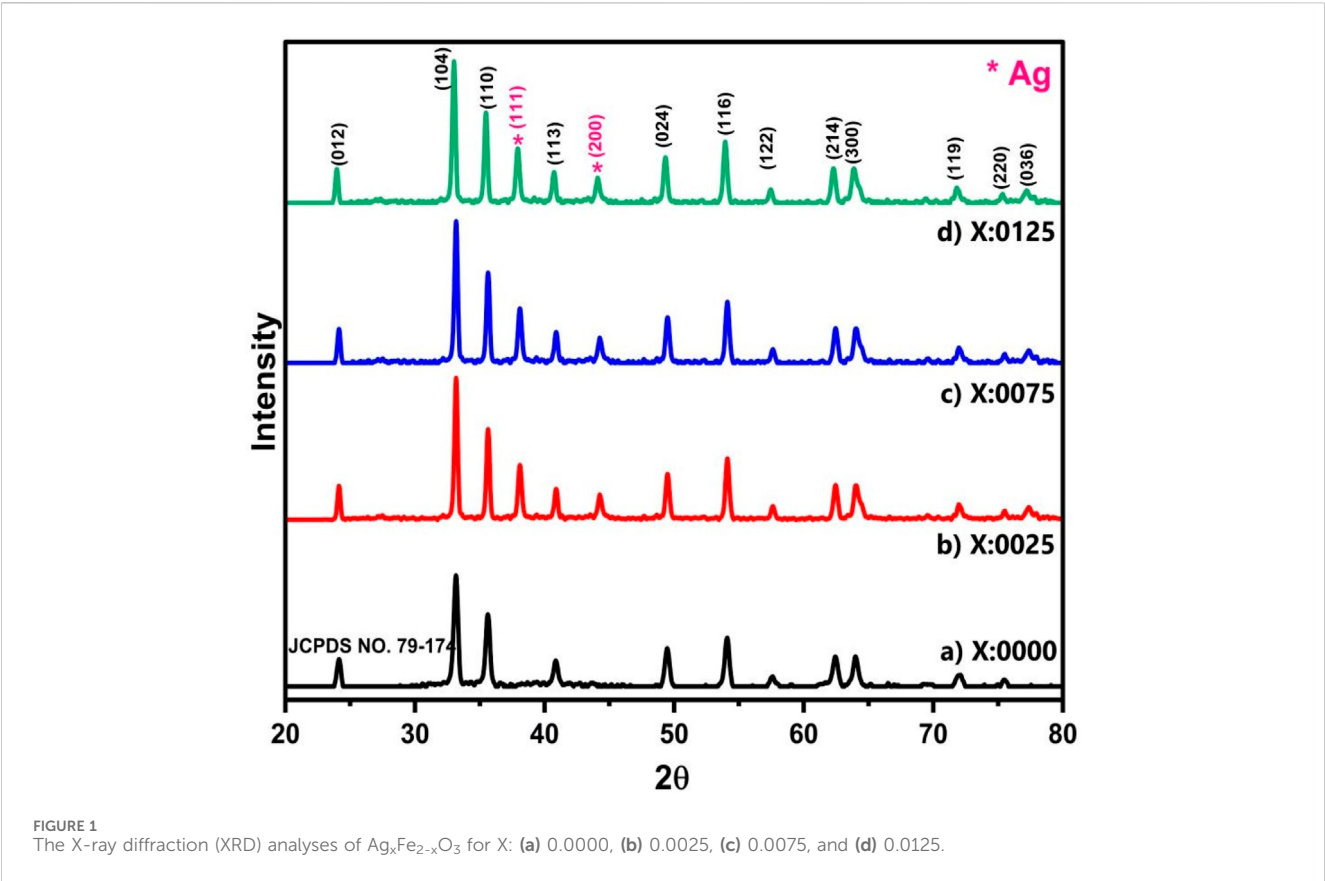


TABLE 1 The crystallographic parameters: lattice parameters, crystalline size, X-ray density, and dislocation density of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where X: 0.0000, 0.0025, 0.0075, and 0.0125.

Sample	Crystallite size	Lattice parameter value	X-ray density	Dislocation density
	(nm)	Å	Gm/c ³	Line/nm ²
0.0000	23.22	8.9321	3.276	0.00186
0.0025	23.99	8.9694	3.349	0.00174
0.0075	22.99	8.9347	3.297	0.00189
0.0125	22.26	8.9321	3.300	0.00202

while being continuously stirred with a magnetic stirrer to maintain homogeneity. After a fixed time interval, 2 mL of the sample was withdrawn and centrifuged for about 15 min at 6,000 rpm to separate the catalyst particles. The supernatant liquid was analyzed using a UV-3,600 Shimadzu UV-visible spectrophotometer by monitoring the change in concentration. The absorbance value was recorded at 526 nm and was used to determine the percentage of dye degradation.

The percentage of MB degradation was calculated using the following equation:

$$\% \text{ MB degradation} = \frac{C_0 - C_t}{C_0} \times 100$$

where C_0 is the initial concentration of MB, and C_t is the concentration at irradiation time t .

2.5 Antimicrobial activity study

The agar well diffusion method was employed to determine the antimicrobial activity of the synthesized $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ (X: 0.0000, 0.0025, 0.0075, 0.0125) nanoparticles. Strains viz., *Candida albicans* (NCIM 3100), *E.coli* (NCIM2065), *A. niger* (ATCC504), *S. aureus* (NCIM2178), *B. subtilis* (NCIM2063), and *P. mirabilis* (NCIM2388) were used to assess the antibacterial activity. Here, 100 μL of inoculum (10^6 CFU/mL) was swabbed onto Muller-Hinton agar (Himedia) plates using a sterilized cotton bud. Five wells were bored in each plate with a sterile cork borer. Stock solutions of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ and streptomycin (control) were prepared at a concentration of 1 mg/mL, and DMSO was used as a negative control. Aliquots (80–90 μL) of these samples were added to each well. The plates were incubated at 37 C for 24–48 h. The minimum inhibition

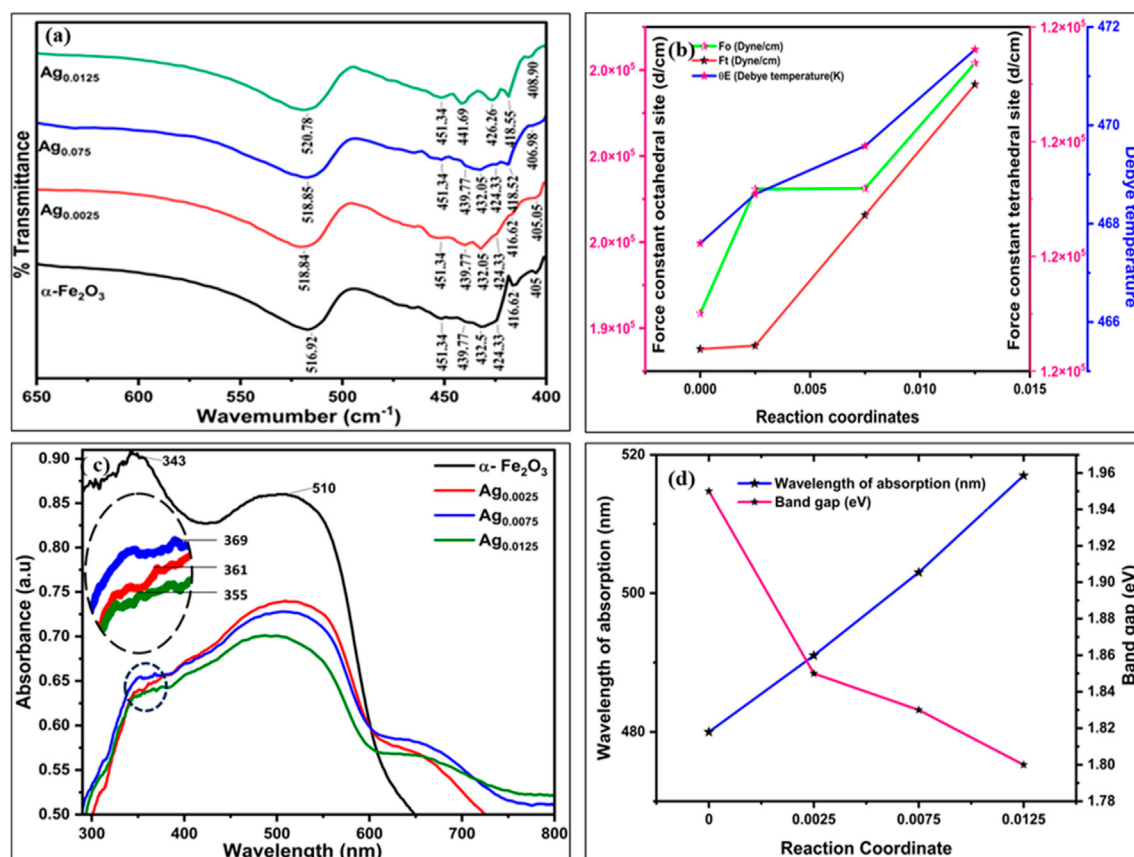


FIGURE 2

(a) FTIR spectra of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where $x = 0, 0.0025, 0.0075, 0.0125$ and (b) variation in the force constant at the octahedral sites (F_o), tetrahedral sites (F_t), and Debye temperature θ_E . (c) UV-visible spectra of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where $x = 0, 0.0025, 0.0075, 0.0125$ (inset of (c) is the zoom image of the UV-visible spectrum of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where $x = 0, 0.0025, 0.0075, 0.0125$) and (d) wavelength of absorption and band gap vs. Ag concentrations.

TABLE 2 FTIR spectral data variation in the force constant at the octahedral sites (F_o), tetrahedral sites (F_t), and Debye temperature θ_E for $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where $x = 0, 0.0025, 0.0075, 0.0125$.

Sample	$V_2 \text{ cm}^{-1}$	$V_2 \text{ cm}^{-1}$	F_o (dyne/cm)	F_t (dyne/cm)	Debye temperature (K)
0.0000	516.92	405.00	194,171.88	119,192.71	467.60
0.0025	518.84	405.05	195,616.98	119,222.14	468.60
0.0075	518.85	405.98	195,624.52	120,361.00	469.58
0.0125	520.78	408.90	197,082.58	121,499.32	471.54

concentration (MIC) of the $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ was observed by the broth dilution technique in the concentration range of 1,000 $\mu\text{g/mL}$, 500 $\mu\text{g/mL}$, 125 $\mu\text{g/mL}$, 62.5 $\mu\text{g/mL}$, and 31.25 $\mu\text{g/mL}$, respectively.

3 Results and discussions

3.1 X-ray diffraction analysis

The XRD analysis (Figure 1) was carried out with Cu-K α radiation ($\lambda = 1.54182 \text{ \AA}$) in the 2θ range of 20° – 80° , which provides detailed insights into the structural properties of the materials. XRD revealed the distinct peaks corresponding

crystallographic planes, (012), (104), (110), (113), (024), (116), (122), (214), (300), (119), and (220), which confirmed the rhombohedral crystal structure of $\alpha\text{-Fe}_2\text{O}_3$ (JCPDS NO. 79-174) (Zhao et al., 2021). Additionally, the presence of (111) and (200) peaks confirms the doping of Ag in the Fe_2O_3 lattice, which aligns with the standard reference (JCPDS NO. 89-3722) (Karunakaran et al., 2013). The XRD pattern for $\alpha\text{-Fe}_2\text{O}_3$ is similar to that of Ag-doped Fe_2O_3 (0.0025, 0.0075, 0.0125), except for the appearance of Ag-related peaks. No peaks correspond to other phases, indicating the formation of a highly pure rhombohedral Ag-doped Fe_2O_3 .

The changes in lattice parameters, crystallite size, X-ray density, and dislocation density are documented in Table 1. Krejčíková et al. reported that crystallite size was calculated with the Debye–Scherrer formula

TABLE 3 (a) UV-visible spectra of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where X: 0, 0.0025, 0.0075, 0.0125 and (b) wavelength of absorption and band gap with respect to Ag concentrations.

Sample	Wavelength of absorbance (nm)	Band gap (eV)
0.0000	480	1.85
0.0025	491	1.75
0.0075	503	1.71
0.0125	517	1.70

(Equation 1). Initially, it increases with 0.0025 Ag doping (23.22 nm–22.99 nm), due to the stabilization of Ag^+ ions in the Fe_2O_3 lattice, which reduces defects and promotes crystal growth (Krejčíková et al., 2012).

$$\text{DE} = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

$$a = d\sqrt{h^2 + k^2 + l^2} \quad (2)$$

$$d_x = \frac{8M}{Na^3} \quad (3)$$

$$\rho_D = \frac{1}{D^2} \quad (4)$$

However, at the 0.0075 Ag and 0.0125 Ag concentrations, the crystallite size decreases, as the introduction of Ag^+ ions, which introduces strain and defects into the lattice, inhibits further crystal growth and leads to a smaller crystallite size, which is documented in Supplementary Figure S2a. The lattice parameters were calculated with Equation 2. They

increased from an initially low Ag doping of the material (8.9321 Å to 8.9694 Å) because the substitution of Fe^{3+} ions (ionic radius ~0.64 Å) by larger Ag^+ ions (ionic radius ~1.15 Å) causes lattice expansion. However, at higher Ag (0.0075, 0.0125) concentrations, the lattice parameters decrease slightly (8.9347 Å to 8.9321 Å), which may be attributed to the saturation of Ag^+ ions in the lattice or the formation of Ag clusters on the surface, reducing the overall strain on the $\alpha\text{-Fe}_2\text{O}_3$ lattice, as recorded in Supplementary Figure S2b.

The X-ray density was calculated with Equation 3. It increases with Ag (0.0025) doping due to the higher atomic weight of Ag (107.87 g/mol) compared to Fe (55.85 g/mol), which increases the overall density of the material. However, at higher Ag concentrations (0.0075, 0.0125), the X-ray density alters slightly, which may be due to the change in lattice packing efficiency or the formation of an Ag cluster on the surface documented in Supplementary Figure S2c. Equation 4 helps calculate dislocation density. It decreases initially with low Ag (0.0025) concentration, as Ag^+ ions occupy interstitial sites or substitute Fe^{3+} ions, reducing lattice strain and defects. However, at higher Ag (0.0075, 0.0125) concentrations, the dislocation density increases (Supplementary Figure S2d), likely due to the introduction of additional strain and defects caused by the higher concentration of Ag^+ ions, which disrupts the lattice structure.

3.2 FTIR and UV–visible analysis of Ag-doped Fe_2O_3 samples

Figure 2a reveals the FTIR spectra of Ag-doped Fe_2O_3 samples (X: 0.0025, 0.0075, 0.0125) and gives significant insights into structural modification induced by the incorporation of Ag ions in the lattice.

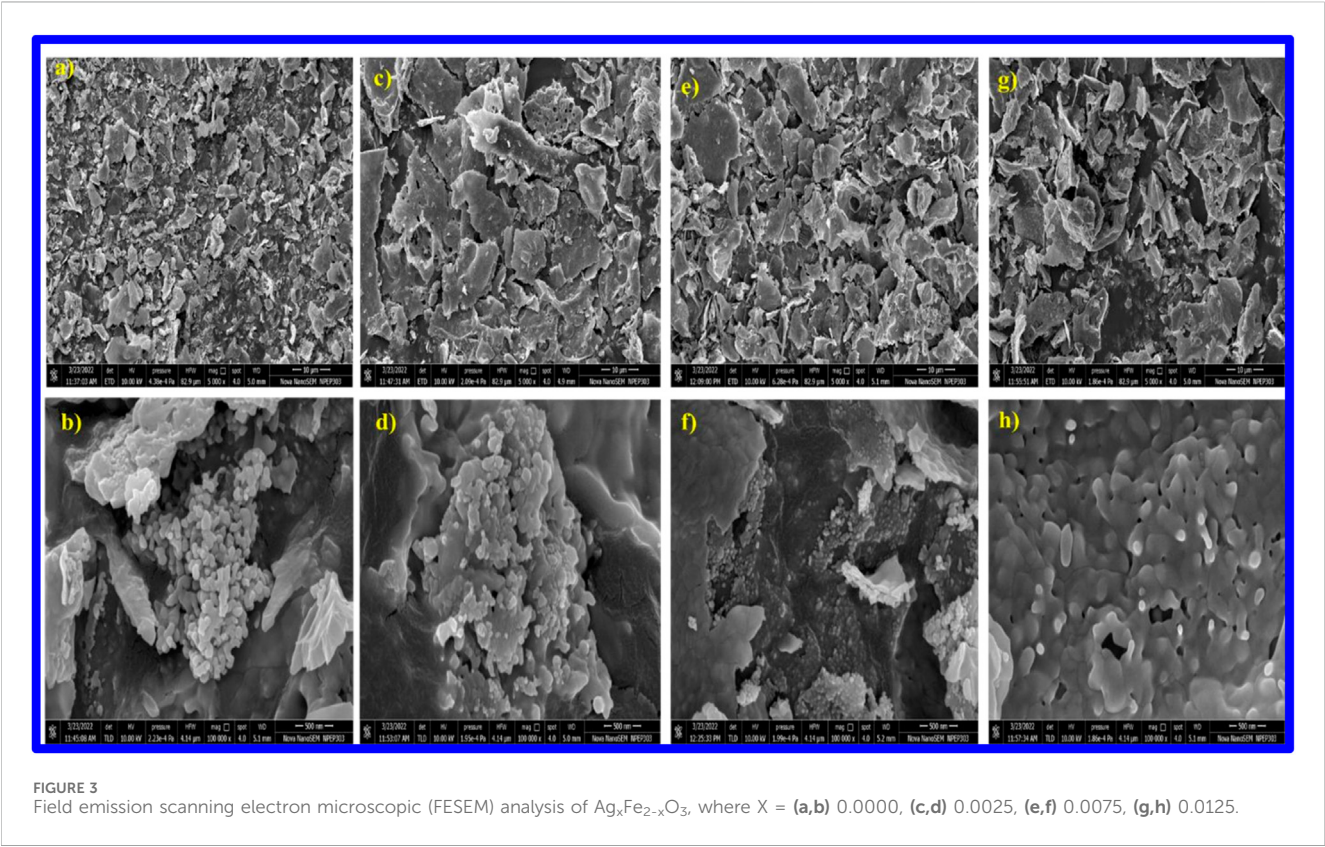


TABLE 4 Elemental analysis with weight for $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where X: 0.0125%.

Sr. NO	Element	Weight %
1	O	27.5
2	Ag	3.5
3	Fe	69.0

The vibrational bonds corresponding to the octahedral site and tetrahedral site were calculated using Equations 5–8 and are documented in Table 2. A slight (194,171.88–197,082.58) change was observed in the octahedral bond (ν_2) with an increase in Ag concentration ($X = 0$ to $X = 0.0125$). It suggests a change in the bond length and local symmetry around the octahedral sites, which may be due to the substitution of Fe^{3+} by Ag ions. Additionally, the force constant for tetrahedral sites (ν_2) increases (119,192.71–121,499.32) with increasing Ag concentrations ($X = 0$ to $X = 0.0125$).

This indicates stronger cation-oxygen bonding with higher Ag concentrations, which impacts reduced bond length and enhances electrostatic interaction at tetrahedral sites. This is further supported by the rise in the Debye temperature (θ_E), which was calculated by Equation 7. As concentration increases from $X = 0$ to $X = 0.0125$, the Debye temperature increases from 462.60 K to 471.54 K, which may increase lattice vibrations and improve thermal stability. Figure 2b findings suggest that the incorporation of Ag ions into the Fe_2O_3 lattice not only modifies the local structural environment but also influences the vibrational and thermal properties of the materials, which could have implications for catalytic behavior.

$$F_t = 4\pi^2 c^2 \nu_1^2 \mu \quad (5)$$

$$F_o = 4\pi^2 c^2 \nu_2^2 \mu \quad (6)$$

$$\theta_E = \frac{h c \nu_{av}}{K_B} \quad (7)$$

The UV-visible absorption spectra of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ (X : 0, 0.0025, 0.0075, 0.0125) are presented in Figure 2c. A systematic bathochromic shift in the absorption edge was observed with increasing Ag doping concentration. Undoped $\alpha\text{-Fe}_2\text{O}_3$ exhibits an absorption edge at 480 nm, corresponding to a band gap of 1.85 eV. Upon Ag incorporation, the absorption edge progressively red-shifted to 491 nm ($X = 0.0025$), 503 nm ($X = 0.0075$), and 517 nm ($X = 0.0125$), indicating band gap narrowing. Synchronously, a distinct absorption emerged in the UV region, centered near 369 nm for $X = 0.0025$. This peak shows a hypsochromic shift to 361 nm ($X = 0.0075$) and 355 nm ($X = 0.0125$) with higher Ag loading (inset of Figure 2c is the zoom image of the UV-visible spectrum of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where X : 0, 0.0025, 0.0075, 0.0125). Khurana et al. reported the same trend and attributed it to electronic interactions between the Ag species and the Fe_2O_3 matrix, likely arising from localized surface plasmon resonance (LSPR) of metallic Ag nanoparticles (Khurana and Jaggi, 2021).

Ag loading on Fe_2O_3 nanoparticles can influence the shifting of the band gap from 1.85 eV to 1.70 eV. This may depend on various factors such as quantum confinement effects and charge transfer between Ag and Fe_2O_3 , providing insights into the electronic structure and the potential for light absorption. Additionally, Ag doping in $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ exhibits a surface plasmonic resonance (SPR) effect due to the presence of Ag nanoparticles on the surface. SPR shows the collective oscillation of conducting electrons in Ag and Fe_2O_3 nanoparticles when exposed to light, leading to strong absorption in the visible region. This is analyzed in Figure 2c through the shifting of peaks in the 350–500 nm region. It is concluded that $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ shows extended absorbance in the visible region and captures more photons, which impacts high SPR and leads to increased photocatalytic and antimicrobial activity.

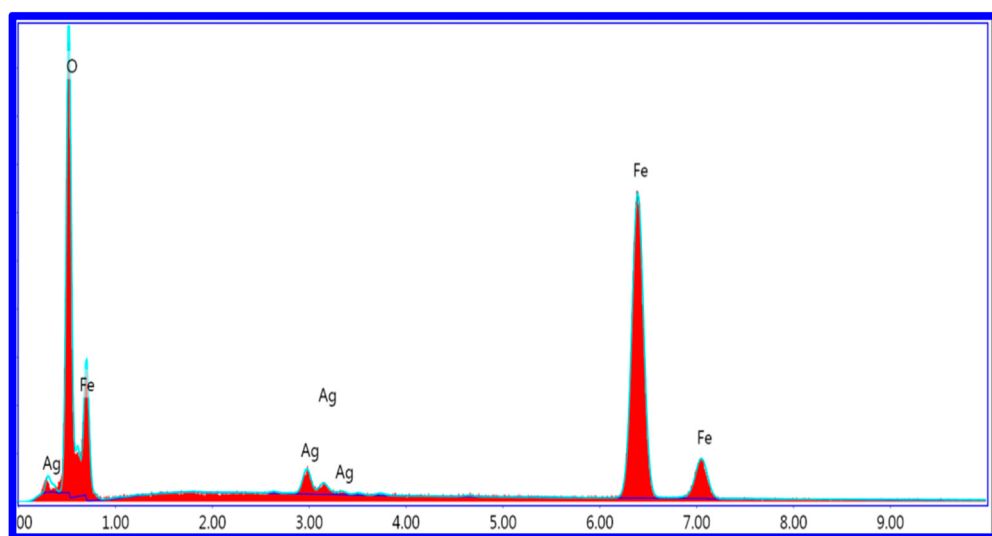


FIGURE 4
Elemental analysis for $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where X: 0.0125 with respect to Ag concentrations.

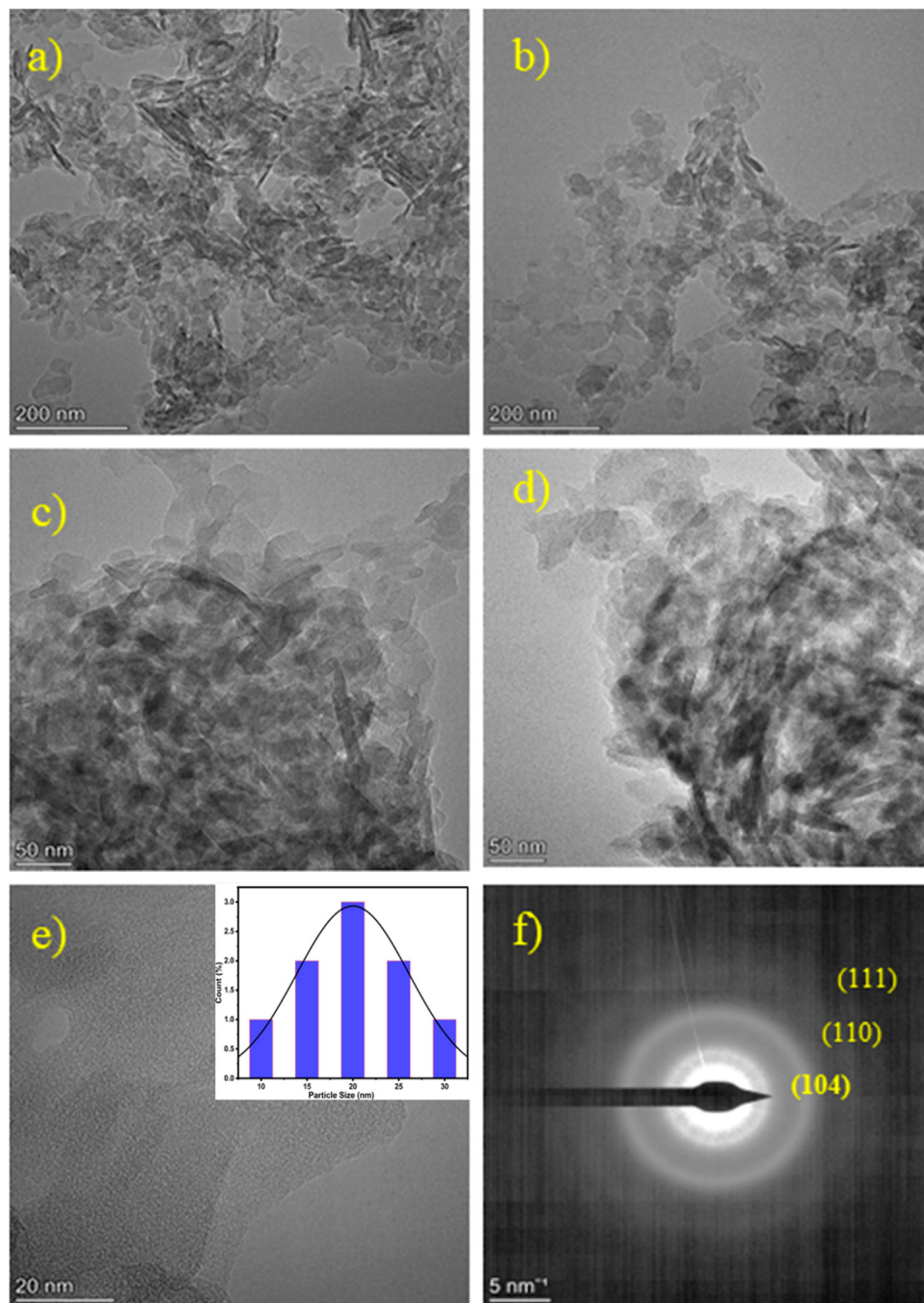


FIGURE 5
(a–e) Transmission Electron Microscopic (TEM) analysis of 0.0125 Ag_xFe_{2-x}O₃. Inset of (e) is particle size distribution (histogram) and (f) SAED pattern of 0.0125 Ag_xFe_{2-x}O₃.

The band gap values of the corresponding materials were calculated using Equation 8 and documented as shown in Figure 2d.

$$E_g = \frac{1239}{\lambda} \quad (8)$$

The calculated values of band gaps 1.85 eV, 1.75 eV, 1.71 eV, and 1.70 eV are reported in Table 3 (Supplementary Figure S3).

This reduction in band gap may be attributed to the incorporation of Ag ions into the Fe₂O₃ lattice, which introduces localized electronic states near the conduction and valence bands. The interaction occurs due to sp-d exchange and d-d transitions. It modifies the electronic structure effectively, which extends the light absorption in the visible region.

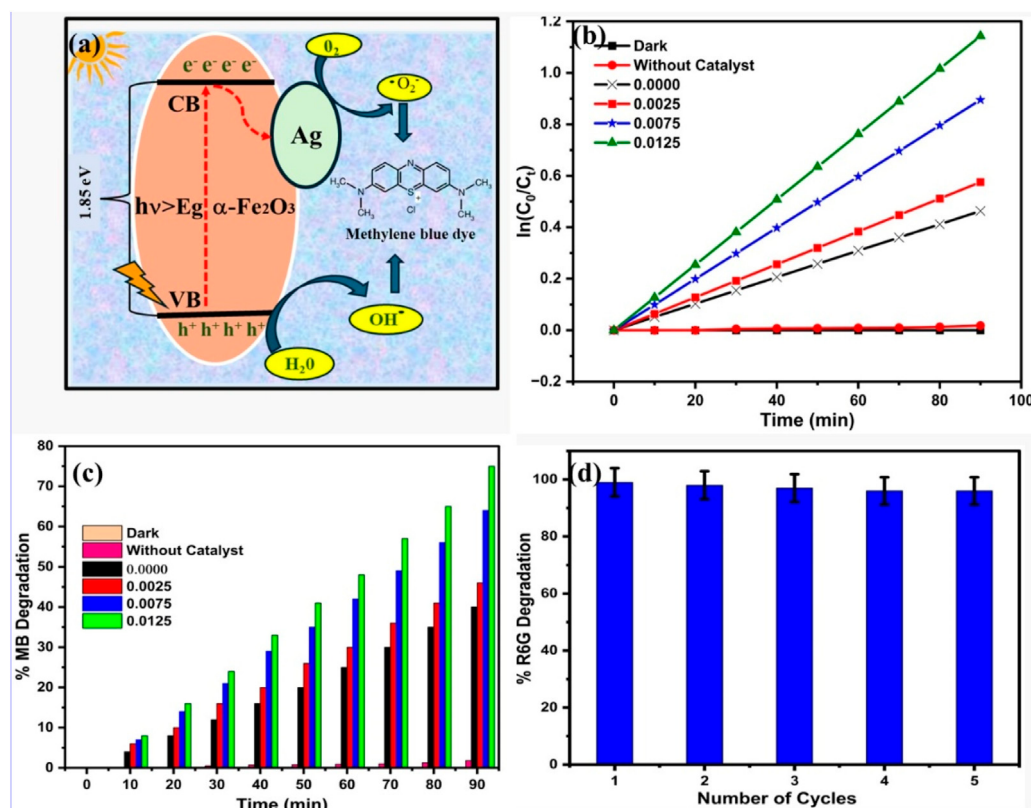


FIGURE 6 (a) Photocatalysis mechanism, (b) kinetic plot of dye degradation, (c) % dye degradation, and (d) recycle study for the dye degradation.

TABLE 5 Photocatalytic degradation of MB dye in the dark, without catalyst, and with $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ catalyst where X: 0, 0.0025, 0.0075, 0.0125.

Material	Rate of reaction K_{app} (min^{-1})	With standard error
Dark	0	0
Without	0.016×10^{-2}	$\pm 0.1039 \times 10^{-4}$
0.0000	0.515×10^{-2}	$\pm 1.5861 \times 10^{-4}$
0.0025	0.639×10^{-2}	$\pm 1.3783 \times 10^{-4}$
0.0075	0.995×10^{-2}	$\pm 3.6582 \times 10^{-4}$
0.0125	1.270×10^{-2}	$\pm 6.746 \times 10^{-4}$
P25 TiO_2	2.33×10^{-2}	$\pm 2.22 \times 10^{-3}$

3.3 Scanning electron microscopy (SEM)

Figures 3a,b present the FESEM sheet-like micrograph of Fe_2O_3 nanoparticles, revealing the formation of fine, asymmetrically shaped particles with dimensions ranging from 50 nm to 70 nm.

Ag nanoparticles were well dispersed in different concentrations from 0.0025, 0.0075, and 0.0125, as shown in Figures 3c–h, respectively, which provide valuable information about the dispersion of Ag nanoparticles on the Fe_2O_3 surface and their distribution within the material. Herein, silver appears

as smaller, separate entities on the surface of the $\alpha\text{-Fe}_2\text{O}_3$ particles (Figures 3a–h). The SEM images a–c–e–g portray irregularly shaped particles or aggregates that make 2–3-micron-sized sheets with spikes on the surface, due to the dispersion of Ag nanoparticles onto the Fe_2O_3 material. Ag nanoparticles were well dispersed and appeared as smaller grooves from the sheet's separate entities on the surface of the Fe_2O_3 particles.

The energy-dispersive X-ray spectroscopy (EDS) analysis confirms the presence of oxygen (O), iron (Fe), and silver (Ag) within the Ag-doped samples. Quantitative elemental composition data for the representative composition $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ ($x = 0.0125$) are summarized in Table 4. The measured weight percentages are 27.5% (O), 69.0% (Fe), and 3.5% (Ag), confirming the successful incorporation of Ag into the Fe_2O_3 matrix. The full EDS spectra are provided in Figure 4.

3.4 Transmittance electron microscopy

Figures 5a–e illustrate TEM images at a concentration of 0.0125 $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$. It can be seen that the morphology of Fe_2O_3 particles is a tiny rod-like morphology of 30–50 nm. The images 5a–d clearly reveal that Ag nanoparticles are highly dispersed on the surface of the Fe_2O_3 . Figure 5e,f is a high-resolution TEM image that shows lattice fringes with a spacing of approximately 0.236 nm. A statistical histogram of the diameter distribution of Ag

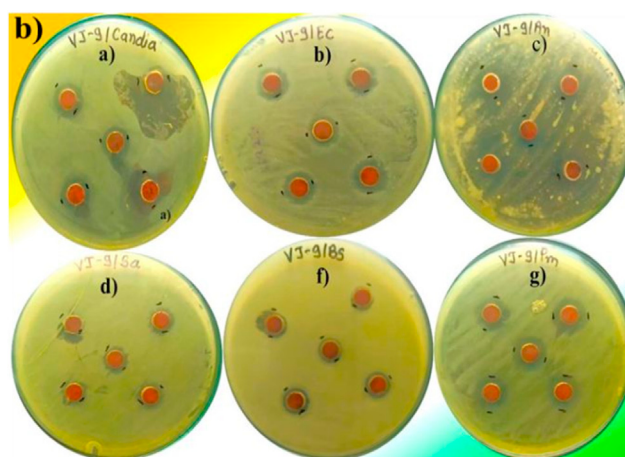
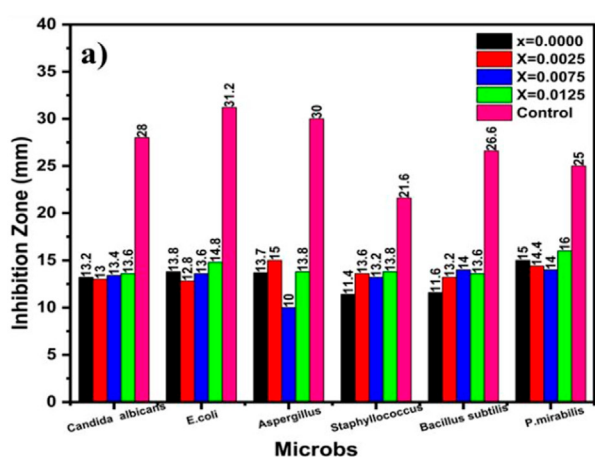


FIGURE 7

(a) The antimicrobial study bar graphs for $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ (X: 0.0000, 0.0025, 0.0075, 0.0125, 0.0125) nanoparticles. Strains viz., *Candida albicans* (NCIM 3100), *E. coli* (NCIM2065), *Aspergillus niger* (ATCC504), *Staphylococcus aureus* (NCIM2178), *Bacillus subtilis* (NCIM2063), and *Proteus mirabilis* (NCIM2388) were used to assess the antibacterial activity. (b) Photographs of test $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, X: 0.000 Petri plates.

NPs in 0.0125 $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ (He et al., 2022) is shown in the inset of Figure 5e.

This spacing corresponds to the Ag (111) plane, indicating the crystal structure of the Ag nanoparticles. Moreover, the size distribution of Ag NPs is narrow, with an average size of 10.63 nm, as depicted in the figure. This indicates that the present method used demonstrates effective control over the size of Ag nanoparticles. Overall, the TEM analysis demonstrates that Ag nanoparticles are well dispersed on the Fe_2O_3 nanorods, with a consistent crystal structure and narrow size distribution (Pandit V. U. et al., 2022).

3.5 Photocatalytic activity for $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$

Figure 6a shows the proposed mechanism for photocatalytic degradation of MB dye using $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$. Figures 6b,c show the kinetic and percentage dye degradation study for 10 ppm MB dye degradation using $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, where X: 0.0000, 0.0025, 0.0075, 0.0125. Figure 6d shows a recycle study of up to five cycles for x: 0.0125 $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$. The photocatalytic degradation mechanism for MB dye using $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ (X: 0, 0.0025, 0.0075, 0.0125), documented in Figure 6a, may be significantly enhanced under visible light irradiation due to the improved charge carrier separation and surface plasmonic resonance effect of Ag. When Fe_2O_3 was exposed to visible light, it generated the electron-hole pair (e^-/h^+), initiating redox reactions. The Fe^{2+} atom in the catalyst reacts with H_2O to generate Fe^{3+} and produces hydroxyl radicals ($\cdot\text{OH}$), which are highly reactive and contribute to breaking down the MB dye molecules. The Ag nanoparticles play a crucial role in increasing the photocatalytic activity of Fe_2O_3 compared to previous reports (Supplementary Table 2). Ag nanoparticles act as electron acceptors, effectively trapping photogenerated electrons from the conduction band of Fe_2O_3 . This electron

transfer prevents recombination with photogenerated holes, thereby prolonging the lifetime of charge carriers. Additionally, the trapped electrons react with oxygen molecules to generate superoxide radical anions ($\text{O}_2^{\cdot-}$), which further contribute to the degradation of MB dye. The plasmonic effect of Ag nanoparticles also increases light absorption, promoting more efficient excitation of charge carriers and accelerating the photocatalytic process.

3.5.1 Reaction kinetics for the degradation of MB

The reaction kinetics data also support the enhanced photocatalytic performance of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$, as shown in Table 5.

The rate constant (K_{app}) increases with increasing Ag concentrations. Pure Fe_2O_3 exhibits a rate constant of $0.515 \times 10^{-2} \text{ min}^{-1}$, while the highest photocatalytic activity was observed for 0.0125 with a rate constant of $1.270 \times 10^{-2} \text{ min}^{-1}$, which is almost 2.5 times higher than pure Fe_2O_3 . We have compared the rate of MB degradation with P25 TiO_2 as a representative catalyst candidate and concluded that the Ag dopant materials are moving towards the TiO_2 results, and there is a scope for the exact optimization of the Ag-doped Fe_2O_3 study.

3.5.2 Recycling study

The reusability potential of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ for MB dye degradation was evaluated up to five consecutive cycles for x = 0.0125, illustrated in Figure 6d, as it shows the highest activity. After each batch of reactions, the catalyst was recovered via centrifugation, washed thoroughly with deionized (DI) water, and dried before reuse. The catalyst shows excellent stability and high photocatalytic degradation efficiency, attributed to the sustained generation of $\cdot\text{OH}$ radicals. The integrity of the catalyst was confirmed through XRD and FTIR analysis conducted after the photocatalytic experiment. The result indicates no significant changes in the crystal structure or functional group, further highlighting the stability and reusability of the material.

3.6 Antimicrobial activity for $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$

The antimicrobial activity of nanoparticles (100 $\mu\text{g/mL}$) and the commercial antibiotic (50 $\mu\text{g/mL}$) has been examined, and significant zones of inhibition (ZOIs) were observed, as shown in Figures 7a,b. The antibacterial activity of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ was tested against *Candida albicans* (NCIM 3100), *E. coli* (NCIM 2065), *Aspergillus niger* (ATCC504), *Staphylococcus aureus* (NCIM 2178), *Bacillus subtilis* (NCIM 2063), and *Proteus mirabilis* (NCIM2388) using the agar well diffusion method. From the ZOI values, $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ compounds are more efficient in inhibiting the growth of *Proteus mirabilis* for X: 0.0125, as a maximum ZOI of 16 mm has been observed (Supplementary Table 1). The zone of inhibition for X: 0.0125 against *C. albicans* was 13.6 mm, *E. coli* was 14.8 mm, *Aspergillus niger* was 13.8 mm, *S. aureus* was 13.8 mm, *B. subtilis* was 13.6 mm, and *Proteus mirabilis* was 16 mm. AgFNPs were found to be more efficient against Gram-positive and Gram-negative strains than against the fungal strains.

These results were compared to commercial antibiotic streptomycin, which have zones of inhibition of 20 mm, 28 mm, 30 mm, 26 mm, 25 mm, and 30.00 mm for *Bacillus subtilis*, *Staphylococcus aureus*, *E. coli*, *Proteus mirabilis*, *Candida albicans*, and *Aspergillus niger*, respectively, illustrated in photographs of the test and control of $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ on petri plates (Supplementary Figure S5). Possible mechanisms for the reduced effects of AgFe_2O_3 against Gram-positive or Gram-negative bacteria may be that, because these AgFe_2O_3 are highly stable in the ambient environment, there is less contribution of metal ion release for antibacterial activity. In contrast, reactive oxygen species may be generated from Fe_2O_3 when exposed to visible light. The created electron-hole pairs can contribute to the generation of reactive oxygen species such as superoxide radical anions (O_2^-), hydroxyl radicals ($\text{OH}^\cdot$), etc. The generated species result in membrane desorption, leading to the death of the bacteria (Supplementary Table 4). Various interactions like electrostatic, dipole-dipole, hydrogen bond, hydrophobic, and van der Waals interactions are responsible for disruption of cellular function and disruption and disorganization of membranes.

4 Conclusion

In conclusion, $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ nanoparticles were synthesized successfully using the simple sol-gel auto-combustion method. An increase in the Ag content resulted in a decrease in the crystallite size of $\alpha\text{-Fe}_2\text{O}_3$ to 22.26 nm. As Ag concentration increased from X = 0.0000 to X = 0.0125, the octahedral bond (V1) slightly changed, indicating alterations in bond length and symmetry due to Fe^{3+} ions being replaced by Ag^+ ions. Additionally, the force constant for tetrahedral sites (V2) increased, which suggests the formation of a ferrite structure. The band gap decreased progressively with increasing Ag content, which is significant for photocatalytic and antimicrobial applications. The FESEM images revealed a sheet-like morphology. TEM analysis of 0.0125% Ag-incorporated Fe_2O_3 revealed a rod-like morphology with lengths ranging from 30 to 50 nm. The incorporation

of plasmonic Ag nanoparticles into $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ showed excellent photocatalytic degradation of MB dyes, achieving a rate constant of $1.97 \times 10^{-2} \text{ min}^{-1}$, nearly double that of Fe_2O_3 ($0.515 \times 10^{-2} \text{ min}^{-1}$). $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ also exhibited greater efficacy against Gram-positive and Gram-negative strains than against fungal strains. At X = 0.0125, $\text{Ag}_x\text{Fe}_{2-x}\text{O}_3$ showed the highest inhibition against *P. mirabilis*, with a maximum zone of inhibition (ZOI) of 16 mm.

Data availability statement

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding authors.

Author contributions

AA: Formal Analysis, Data curation, Methodology, Resources, Investigation, Writing – review and editing. VP: Methodology, Writing – review and editing, Formal Analysis, Data curation, Resources, Investigation. VJ: Data curation, Resources, Formal Analysis, Methodology, Writing – review and editing, Investigation. MG: Writing – review and editing, Methodology, Conceptualization, Investigation, Formal Analysis, Resources, Data curation. DL: Writing – review and editing, Conceptualization, Writing – original draft, Formal Analysis, Visualization, Project administration, Resources.

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Conflict of interest

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Supplementary material

The Supplementary Material for this article can be found online at: <https://www.frontiersin.org/articles/10.3389/fccts.2025.1655324/full#supplementary-material>

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