

Antibacterial Activity of NiWO₄/NiFe₂O₄ Magnetic Binary Nanocomposite with Visible-Light-Driven Photocatalytic Degradation of Methylene Blue

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Received: 14th June, 2026; Revised: 20th July 2026; Accepted: 20th August, 2026; Available Online: 05th September 2026

ABSTRACT

A magnetically separable NiWO₄/NiFe₂O₄ binary nanocomposite was synthesized and characterized using FTIR, XRD, UV-DRS, VSM, SEM-EDX, and TEM. The nanocomposite exhibited a concentration-dependent antibacterial activity against both the Gram-negative *Escherichia coli* and Gram-positive *Corynebacterium*. At 100 µL, inhibition zones of 14 mm and 15 mm were observed against *E. coli* and *Corynebacterium*, respectively, compared with 26 mm for the standard antibiotic Amoxicillin-Clavulanate. The antibacterial activity is attributed to ROS-induced oxidative damage to bacterial cellular components. The nanocomposite also demonstrated excellent visible-light photocatalytic activity, achieving complete degradation of methylene blue within 45 min, with good recyclability for potential wastewater treatment. The crystallite size and optical band gap were 17.8 nm and 2.48 eV, respectively. The combination of antibacterial activity, visible-light photocatalysis, magnetic separability and recyclability highlights NiWO₄/NiFe₂O₄ as a promising multifunctional nanocomposite for antibacterial and environmental applications.

Keywords: NiWO₄/NiFe₂O₄, binary catalyst, Antibacterial Activity, photocatalytic dye degradation, Methylene blue dye.

How to cite this article: Supriya .G, Swapna.D, Sruthi.V and Paul Douglas.S, Antibacterial Activity of NiWO₄/NiFe₂O₄ Magnetic Binary Nanocomposite with Visible-Light-Driven Photocatalytic Degradation of Methylene Blue. *Int J Drug Deliv Technol.* 2026;16(7): 1829-1835. DOI: 10.25258/ijddt.16.7.196

Source of support: Nil.

Conflict of interest: None

1. INTRODUCTION

Dyes used in the textile, plastic, paint, and pharmaceutical industries are highly stable, hardly biodegradable and toxic organic pollutants. Population growth and industrialization increase the release of various pollutants into natural water bodies. Thus, removing these pollutants from industrial effluent is essential. Wastewater treatment is very important for the balance of nature, the environment, and human health. If wastewater is not treated properly, it causes severe pollution, which has hazardous effects on human health, biodiversity, and aquatic life.. Conventional treatment methods tend to have drawbacks such as incomplete degradation, secondary pollution, and high operational costs. Advanced oxidation processes (AOP's), especially semiconductor photocatalysis, have emerged as promising alternatives because they facilitate the oxidative degradation of resistant pollutants under light irradiation. electrons excite from the valence band to the conduction band with exposure to light with which the energy greater than or equal to the semiconductor bandgap, generating electron-hole pairs. Conduction band electrons convert dissolved oxygen into superoxide radicals (O₂•⁻) whereas Valence-

band holes react with water or hydroxyl groups to generate highly reactive hydroxyl radicals(•OH).By attacking dye molecules these reactive oxygen species, produce intermediates that may be further mineralized. NiWO₄ exhibits favorable visible-light absorption, chemical stability^{1,2}, catalytic activity, low cost³⁻⁵,and corrosion resistance⁶⁻⁸, whereas magnetic NiFe₂O₄ is a stable and magnetically separable material⁹⁻¹³. However, rapid electron-hole recombination limits the efficiency of individual photocatalysts¹⁴⁻¹⁷. Constructing a heterojunction can enhance charge separation and photocatalytic activity. Therefore, a binary NiWO₄/NiFe₂O₄ nanocomposite was developed for the visible-light-driven degradation of Methylene Blue in wastewater.

2.EXPERIMENTAL

Without additional purification, analytical-grade nickel nitrate, sodium tungstate, citric acid, polyethylene glycol, ferric citrate, ethylene glycol, ammonia, and double-distilled water were utilised.

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2.1 SYNTHESIS OF NiWO₄

Ni(NO₃)₂·6H₂O (29 g) and Na₂WO₄·2H₂O (32.9 g) were dissolved separately in 100mL of PEG solution (2 g/100 mL) and vigorously stirred. After standing for 24 hours, the precipitate was filtered, and dried for 5 hours at 110°C, and then calcined for six hours at 400°C.

2.2 SYNTHESIS OF NiFe₂O₄.

Nickel ferrite is synthesised using the sol-gel method, for this ferric citrate (11.9 g), citric acid (11.5 g), and nickel nitrate (5.8 g) were combined. After adding 20 mL of ethylene glycol and using ammonia to bring the pH to neutral, the liquid was heated and stirred until it dry. The product was calcined at 500°C for five hours.

2.3 SYNTHESIS OF BINARY NANOCOMPOSITE(NiWO₄/NiFe₂O₄)

The NiWO₄/NiFe₂O₄ nanocomposite sample is prepared by combining in a 0.8:0.2 weight ratio of (NiWO₄:NiFe₂O₄). The powders are thoroughly ground using a mortar and pestle to ensure uniform mixing.

3. RESULTS AND DISCUSSION

3.1 XRD SPECTRAL STUDIES

The X-ray diffraction pattern Fig.1 shows characteristic NiWO₄ reflections at 19.28°, 23.97°, 24.92°, 30.69°, 36.45°, 41.61°, 48.34°, and 54.59°, assigned to the (100), (011), (110), (111), (022), (102), (022), and (202) planes, respectively¹⁸. NiFe₂O₄ reflections occur at 30.69°, 35.61°, 36.45°, 41.61°, and 54.59°, corresponding to the (220), (311), (222), (400), and (422) planes^{19,20}. The average

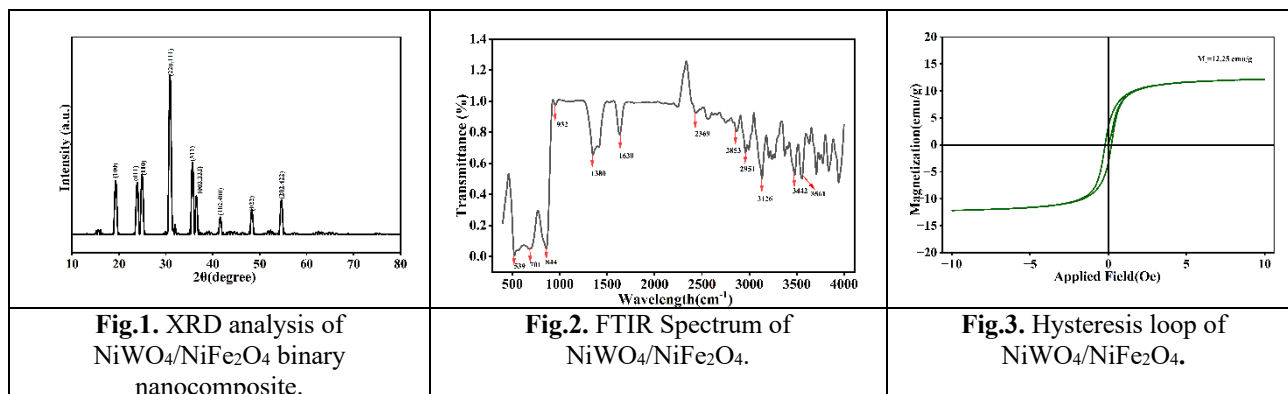
crystallite size of the NiWO₄/NiFe₂O₄ nanocomposite was calculated using the Debye-Scherrer equation, $D=0.9\lambda/(\beta\cos\theta)$, from the XRD data .where β represents the FWHM. The calculated average crystallite size was 17.8 nm.

3.2 FT-IR STUDIES

Fig. 2 presents the FTIR spectra. The bands at **844 and 932 cm⁻¹** indicate W–O and W=O stretching vibrations, whereas **701cm⁻¹** indicates metal–oxygen bonds.²¹ The bands which are at **1380 and 2369 cm⁻¹** corresponds to C–O and C=O groups from atmospheric CO₂. and the bands at **1630 and 3442 cm⁻¹** represent O–H bending and stretching of adsorbed water^{22,23}. The spinel structure is confirmed by the peak²⁴ at 539 cm⁻¹, and metal–oxygen bands are seen in the 4000–400 cm⁻¹ range. The **3100–3600 cm⁻¹** band corresponds to O–H stretching, while **2951 and 2853 cm⁻¹** indicate C–H stretching of residual organic species.²⁵

3.3 MAGNETIC PROPERTIES

Saturation magnetization (Ms) is the maximum magnetization of a material under an external magnetic field. VSM analysis gave a saturation magnetization of 12.25 emu/g, as shown in Fig.3. Bulk nickel ferrite generally shows Ms values between 50-55 emu/g.²⁶ Several factors influence the saturation magnetization of nickel ferrite, such as Doping elements ,Crystallinity, and Particle size. Nanoparticles tend to have lower Ms values due to Increased surface effects, and reduced crystallinity.



3.4 SEM AND EDX STUDIES

SEM images shown in Fig.4.display a rough, polycrystalline, aggregated morphology with interparticle

gaps that could provide accessible reaction sites. EDX identified Ni, W, Fe, and O at 17.74, 40.35, 23.71, and 18.20 mass%, respectively; No additional elemental peaks were observed, indicating good compositional purity.

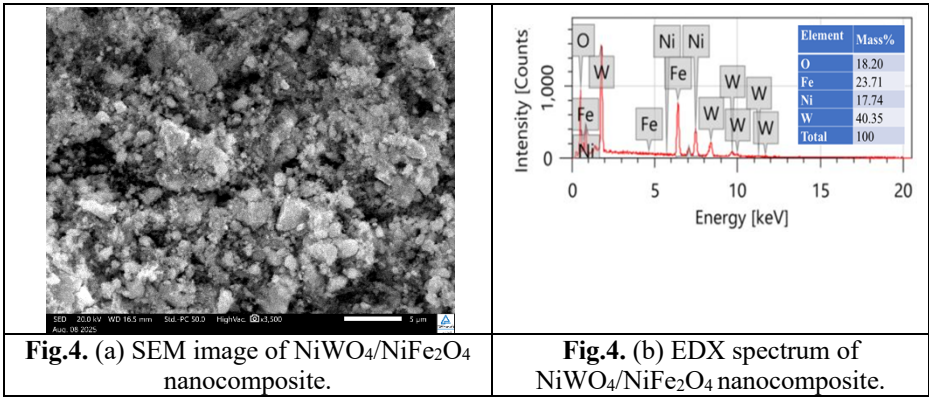


Fig.4. (a) SEM image of NiWO₄/NiFe₂O₄ nanocomposite.

Fig.4. (b) EDX spectrum of NiWO₄/NiFe₂O₄ nanocomposite.

3.5 TEM STUDIES

The structure and morphology of the synthesised NiWO₄/NiFe₂O₄ nanocomposite were investigated in more detail using TEM. TEM and selected area diffraction

patterns of the NiWO₄/ NiFe₂O₄ sample are displayed in Fig. 5a, 5b and 5c. The average particle size is 20.7 nm. The crystalline size determined from XRD data was nearly equivalent with the calculated average particle size , which was determined to be 20.7 nm using ImageJ software.

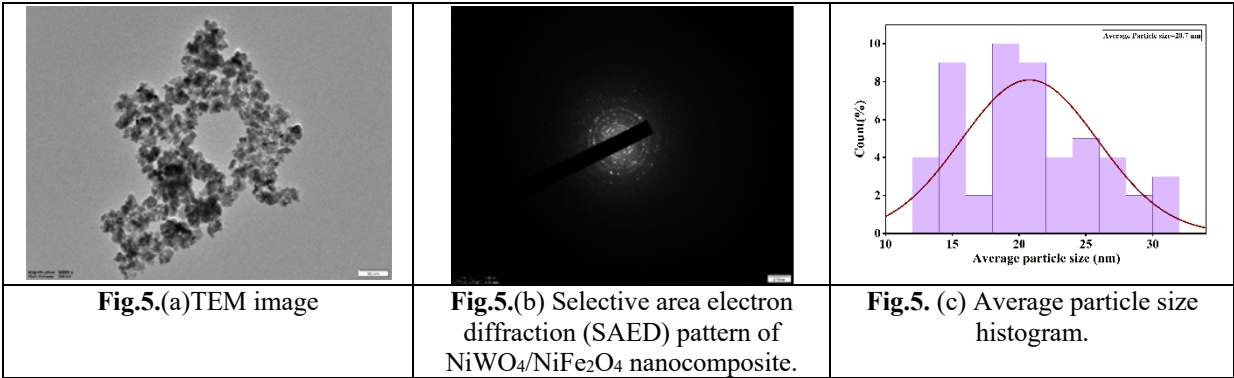


Fig.5.(a)TEM image

Fig.5.(b) Selective area electron diffraction (SAED) pattern of NiWO₄/NiFe₂O₄ nanocomposite.

Fig.5. (c) Average particle size histogram.

3.6 UV-DRS STUDIES

The **optical band gap energy** of the NiWO₄/ NiFe₂O₄ nanocomposite was determined by the **Tauc plot method**. A plot of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) was used to

analyze the bandgap, as illustrated in **Fig.6** the bandgap value was determined by extrapolating the plot's **linear section** to intersect with energy axis. From this linear extrapolation, the estimated **optical bandgap** of NiWO₄/ NiFe₂O₄ was **2.48 eV**.

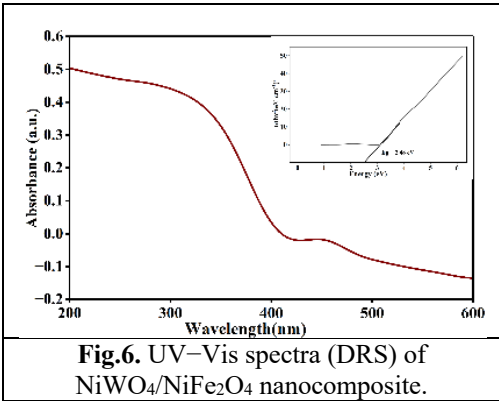


Fig.6. UV-Vis spectra (DRS) of NiWO₄/NiFe₂O₄ nanocomposite.

4. PHOTOCATALYTIC STUDIES

Methylene Blue was utilised as an experimental pollutant to assess the photocatalytic activity of the synthesized catalyst. The dye solution was magnetically stirred in the

dark for 30 min to reach equilibrium before exposure to sunlight.

4.1 INFLUENCE OF IRRADIATION TIME

Irradiation time plays a **key role** in determining the **photocatalytic degradation efficiency** of methylene blue dye. The study involved NiWO₄, NiFe₂O₄, and a **binary NiWO₄/NiFe₂O₄ nanocatalyst**. All other variables were kept **constant** to isolate the effect of irradiation time. Fig.7a shows the correlation between **irradiation time** and **degradation efficiency (C/C₀)**.

4.2 DYE CONCENTRATION RANGE

Experiments were conducted at dye concentrations of 5 to 100 ppm with a constant catalyst loading of 500mg. The highest degradation efficiency was observed at 10 ppm. Initially, degradation increased with dye concentration due

to greater interaction with the catalyst's active sites. Beyond 10 ppm, efficiency decreased because excessive dye accumulation reduces the availability of active sites. Thus, 10 ppm was identified as the optimum concentration as shown in Fig.7b.

4.3 EFFECT OF CATALYST DOSE

The impact of catalyst dose on MB degradation was studied at pH 10 and 10 ppm MB concentration, using dosages of 0.2 to 1.0 g/L (Fig.7c). Degradation efficiency increased up to 0.4 g/L due to reactive species generation and increased active surface area²⁷. Above 0.4 g/L, efficiency decreased due to reduced light penetration, particle agglomeration, and turbidity. Thus, 0.4 g/L was identified as the optimum catalyst dosage.

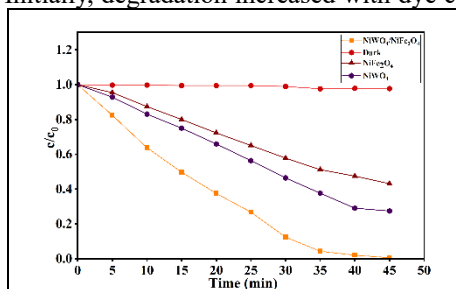


Fig.7a. Effect of irradiation time.

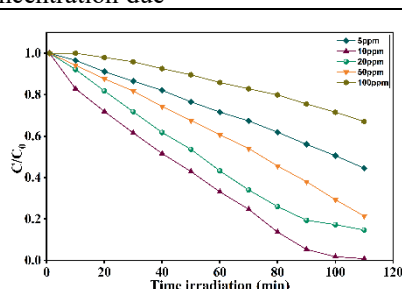


Fig.7b. Effect of Methylene Blue Dye concentration.

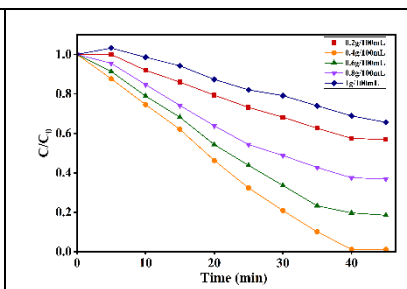


Fig.7c. Effect of catalyst loading on methylene blue degradation

4.4 EFFECT OF pH

Methylene Blue photocatalytic degradation was examined at pH 2, 4, 6, 8, and 10 using the NiWO₄/NiFe₂O₄ photocatalyst in order to determine the effect of solution pH. As shown in Fig.8a, the highest degradation was observed at pH 10. The enhanced activity under basic conditions which lead to increased hydroxyl radical generation and favourable interactions²⁸ between the photocatalyst surface and MB molecules, resulting in faster photocatalytic reactions and improved degradation efficiency²⁹.

4.5 INFLUENCE OF TEMPERATURE

The impact of temperature on the methylene blue degradation was studied from 30 to 45 °C. Fig.8b illustrates the change in methylene blue photodegradation

as a function of temperature within this range. Higher temperatures enhance photodegradation by promoting free-radical formation and reducing electron-hole recombination. whilst low temperatures were found to have low photo degradation effectiveness.

4.6 RECOVERABILITY

The reusability of the photocatalyst was evaluated over three consecutive cycles for Methylene Blue degradation. Before reuse, the catalyst was separated by centrifugation after each cycle and vacuum dried for 2 hours in the dark. As shown in Fig.8c, the photocatalyst maintained nearly consistent degradation efficiency across all cycles, indicating good stability, durability, and recovery potential. The NiWO₄/NiFe₂O₄ photocatalyst therefore demonstrates promising potential for practical applications in MB removal from contaminated water.

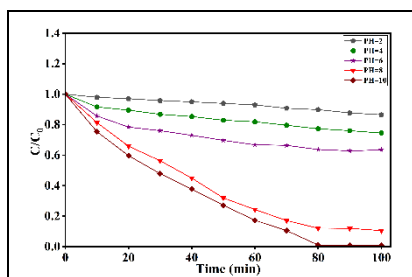


Fig.8a. Effect of pH on the degradation of Methylene Blue.

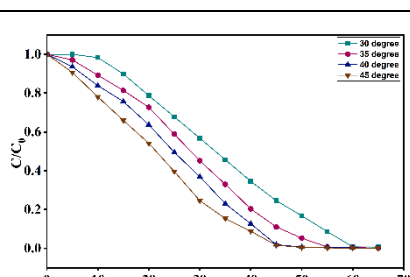


Fig.8b. Effect of Temperature on Methylene Blue degradation.

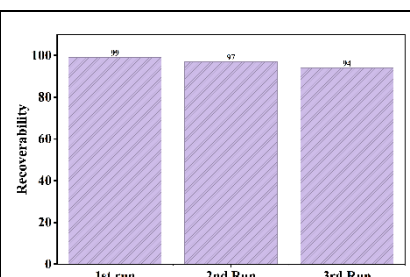


Fig.8c. Recoverability of NiWO₄/NiFe₂O₄ photocatalyst on Methylene Blue degradation.

5. ANTIBACTERIAL STUDIES

The Bacterial strains used were *Escherichia coli* (*E.coli*), Gram-negative, and *Corynebacterium*, Gram-positive. Microbial Technology (IMTECH), Chandigarh, India. Provided all microbial cultures. Cultures were grown overnight at 37°C. Maintained on freshly prepared nutrient agar slants. For long-term storage, preserved at -20°C in 15% glycerol (v/v). The NiWO₄/NiFe₂O₄ nanocomposite exhibited effective antibacterial properties against both Gram-positive *Corynebacterium* and Gram-

negative *E.coli*. The zone of inhibition was measured to assess the catalyst's antibacterial effectiveness compared with the standard antibiotic, Amoxicillin-Clavulanate. The test compound shows dose dependent antibacterial activity, with maximum inhibition zones of 14mm (*E.coli*) and 15mm *Corynebacterium* at 100µL, compared with 26mm for Amoxicillin-clavulanate. NiWO₄/NiFe₂O₄ works against bacteria by causing ROS induced oxidative damage that kills bacterial cells. Table 1 and Figure 9 show that the binary NiWO₄/NiFe₂O₄ nanocomposite exhibited strong antibacterial performance.



Fig.9: Antimicrobial activity of NiWO₄/NiFe₂O₄ nanocomposite.

TEST MICROORGANISM (GRAM -VE)	ANTI MICROBIAL ACTIVITY		TEST MICROORGANISM (GRAM +VE)	ANTI MICROBIAL ACTIVITY	
<i>E. coli</i>	Concentration	Zone of inhibition	<i>Corynebacteria</i>	Concentration	Zone of inhibition
	Control	26mm		Control	26mm
	10 µl	10mm		10 µl	12mm
	50 µl	12mm		50 µl	12mm
	100 µl	14mm		100 µl	15mm

Fig.9 Antimicrobial activity of NiWO₄/NiFe₂O₄ nanocomposite.

Table.1. Antimicrobial Studies of Gram positive and Gram negative stains.

6. CONCLUSION

A NiWO₄/NiFe₂O₄ binary nanocomposite was successfully synthesized, combining the photocatalytic activity of NiWO₄ and with the magnetic properties of NiFe₂O₄. Under optimized conditions of pH 10, 45°C, 10 ppm dye concentration, and 0.4 g/L catalyst, the catalyst achieved complete methylene blue degradation within 45 min under visible light. The improved photocatalytic activity resulted from the combined effect of NiWO₄ and NiFe₂O₄. With a saturation magnetization of 12.25 emu/g, the catalyst can be separated and reused over three cycles. Significant antibacterial activity against *Corynebacterium* and *E.coli* further highlights its potential for photocatalytic and antimicrobial applications.

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