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# **Research Article**

Green Synthesized Silver Nanoparticles Grafted Zinc Ferrite (Ag/Znfe2o4): An Effective Nanocatalyst Fabricated For the Efficient Degradation of Malachite Green (MG) and Methylene Blue (MB)

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## Abstract

Green silver nanoparticles (AgNPs) grafted on zinc ferrite (Zn-Fe2O4) prepared by co-precipitation and calcination are used as a novel nanocatalyst designed for the degradation of malachite green (MG) and methylene blue (MB). FTIR, XRD, SEM, EDX-VSM, and TEM analysis were used to characterize the synthesized compound. In the case of malachite green (MG), 98 % degradation is reported just in one minute with the assistance of 1 ml of 10% H2O2. In the absence of H2O2, however, degradation takes a long time. This compound can also catalyze the degradation of methylene blue (MB), however it is not especially successful. Since hydrogen peroxide is so significant in catalysis, the catalytic reaction must follow the fenton type mechanism by generating reactive oxygen species (ROS). Furthermore, the grafted silver nanoparticles serve as a sink

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for electrons that pass from the valence to the conduction bands, reducing the possibility of electron-hole recombination. Based on various spectrophotometric findings it is possible to infer that the catalytic degradation follows 1st order kinetics.

## Introduction

The phenomenon of catalysis has an extensive range of impending applications in different arena such as waste water purification, environmental remediation etc. Spinel structured metal ferrites and their diverse nanocomposite acts as a vibrant photocatalyst in the degradation of several toxic organic dyes present in the industrial effluents which makes aquatic environment unhygienic. Discharge of these colored materials in the water bodies may cause eutrophication, oxidation, hydrolysis and other detrimental chemical reactions [1-4]. Because of its toxic nature, high COD content, and biological deterioration, sewage from material factories is causing a serious environmental catastrophe. [5,6]. Different methods like chemical treatment, adsorption and biodegradation are presently accessible in the treatment of these waste materials [7,8].

The present research workforce are paying attention in the novelty of photocatalytic material which can utilize solar radiation which composed of 40% visible light in the degradation of organic dyes. Currently ferrite based nanocomposite gained more interest as heterogeneous photocatalyst in this area due to its remarkable microstructure, morphology, magnetic property and narrow band gap [9]. Spinel zinc ferrite has a very narrow band gap and it's composite with TiO<sub>2</sub> and Al<sub>2</sub>O<sub>2</sub> can successfully degrade methyl red and thymol blue photocatalytically [10]. Spinel metal ferrites (MFe<sub>2</sub>O<sub>4</sub>, M is a bivalent cation such as Mn<sup>+2</sup>, Zn<sup>+2</sup>) coupled with g-C<sub>3</sub>N<sub>4</sub> has widely used in the photodegradation of various organic dyes [11]. Green synthesized ZnO nanorod composite with NiFe2O4 has remarkable superparamagnetic property can effectively degrade methylene blue under UV and solar radiation and the catalyst can easily separate magnetically [12]. Hydrothermally prepared magnetically separable ZnFe<sub>2</sub>O<sub>4</sub> – graphene composite has amazing degradation potential of methylene blue in prescence of H2O2 [13,14]. The surface of multi walled carbon nanotube (MWCNT) decorated with ZnFe<sub>2</sub>O<sub>4</sub> has up to 99% methylene blue photodegradation ability [15]. Magnetically recoverable Mn-Fe<sub>2</sub>O<sub>4</sub>/g-C<sub>2</sub>N<sub>4</sub>/TiO<sub>2</sub> nanocomposite has been effectively utilized in the photodegradation of methyl orange under solar irradiation. Silver decorated Zinc/ calcium ferrite was found to exhibit exceptional photodegradation capacity of rohadimine B [16]. Silver doped CuFe<sub>2</sub>O<sub>4</sub>/ TiO<sub>2</sub> nancomposite effectively degrade different azo dye photocatalytically [17]. Grafting of noble metal like Ag, Au, and Pt on the exterior of nanomaterial has the benefit of enhancement of electron hole partition by acting as electron trap and also extends the light absorption in the visible range by the mechanism of surface Plasmon resonance [18-20].

Taking into thoughtfulness of all these aspects, the present study aims at the synthesis of novel green synthesized silver nanoparticle grafted on the surface of spinel structured zinc ferrite to get the

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collective effect of improvement of electron hole partition and absorption of visible light radiation due to surface plasmon resonance by silver nanoparticle. In this attempt, we detail the fabrication, characterization and catalytic activity of green synthesized silver nanoparticle grafted Zinc ferrite. In absence of direct visible light catalytic performance was evaluated based on the degradation of Malachite Green (MG) and Methylene Blue (MB). FTIR, XRD, VSM, TEM, HR-TEM, SEM, and EDX were used to characterize the nanocatalyst's structural and physicochemical properties.

#### **Experimental**

#### Materials

Iron (III) nitrate nanohydrate  $[Fe(NO_3)_3.9H_2O]$ , zinc nitrate hexahydrate  $[Zn(NO_3)_3.9H_2O]$ , silver nitrate  $(AgNO_3)$  and sodium hydroxide (NaOH) are procured from Merck. Methylene blue as well as malachite green is arranged from Sigma-Aldrich. Required chemicals were used without being purified in any way.

#### **Preparation of Zinc ferrite**

Co-precipitation was used to produce zinc ferrite  $(ZnFe_2O_4)$  nanoparticles, which were then calcined in a muffle furnace [21,22]. Analytical grade 4M sodium hydroxide (NaOH) solution was gradually added to salt solutions of 5 mM ferric nitrate [Fe(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O] and 2.5 mM zinc nitrate [Zn(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O] during the synthetic procedures. The pH of the mixture was revised up to 12 by introducing NaOH solution drop wise. After washing with distilled water and ethanol, the precipitate was dried in a low-temperature oven. The extracted precipitates were eventually calcined in a muffle furnace for 5 hours at 500°C.

#### Green synthesis of Silver Nanoparticles

For the biosynthesis of silver nanoparticles 4mM silver nitrate solutions was prepared. To 100 ml of this solution 20 ml of water extract of flowers of *Brassica oleracea italica* (Broccoli) was added and kept in dark for 24 hrs. Formation of dark brown colour (Figure 1) indicates the formation of silver nanoparticles, which is confirmed by UV visible study.



Figure 1: Silver nanoparticles formation from the flower extract of Brassica oleracea italica (Broccoli).

#### **Grafting of Silver Nanoparticle on Zinc Ferrite**

1 g of the as synthesized zinc ferrite is dispersed in the 100 ml of previously synthesized silver nanoparticle by green method. The binary mixture is stirred for 6 hours in a magnetic stirrer before being sonicated for 1 hour. Centrifugation is being used to separate the silver grafted zinc ferrite nanoparticles that have already been formed.

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#### Study of the catalytic performance

The ability of silver nanoparticles grafted zinc ferrite catalyst to degrade Malachite Green (MG) and Methylene Blue (MB) in the absence of direct sunlight and in the presence and absence of hydrogen peroxide was investigated according to published literature. [23,24]. The extent of degradation is monitored spectrophotometrically (Analytical Technologies TS-2080) and degradation percentage is calculated with the help of the equation. 1.

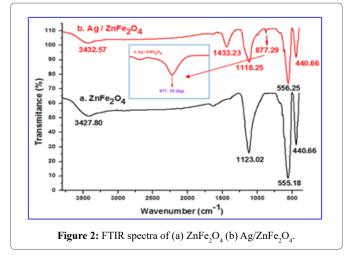
Degradation (%) = 
$$\left(\frac{A_0 - A}{A_0}\right) \times 100$$
 (1)

Where  $A_0$  and A are the absorbance's of the dye before and after the degradation.

## **Results and Discussion**

#### FTIR studies4

As revealed in Figure. 2(a), the Fourier transform infrared (FTIR) spectrum verified the formation of the as-fabricated zinc ferrite spinel structure. The intrinsic stretching vibration of iron-oxygen and zinc-oxygen bonds is assigned to the two major bands at 440.66 cm<sup>-1</sup> and 556.18 cm<sup>-1</sup> respectively in the tetrahedral and octahedral sites of the spinel ferrite [25,26]. The same stretching vibration of Metal - Oxygen is observed when zinc ferrite is grafted with nano silver particle (fig.1b). The band at 1, 123.02 cm<sup>-1</sup> (figure.2a) and 1118.25 cm<sup>-1</sup> (figure.2b) ascribed to a stretching vibration of phenolic group of flavonoide type of compounds present in the plant extract used as stabilizing agent. The band at 1,462.1 cm<sup>-1</sup> corresponds to the vibration mode of O-H band. At 3, 427.80 cm<sup>-1</sup> (figure.2a) and 3432.57 cm<sup>-1</sup> (figure.2b), the wide band corresponds to the O-H stretch vibration of the water molecule of moisture rapt in the compound. An additional small band at 877.27 cm<sup>-1</sup> in (figure 2) reflect the incorporation of silver nano particle in the zinc ferrite.



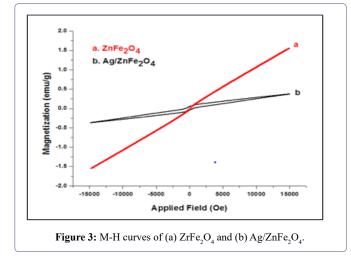
#### VSM studies

The M-H hysteresis curve analyses the magnetic properties of zinc ferrite and it's composite with grafted silver nano particles (figure 3). The magnetic parameters are tabulated in the Table 1.

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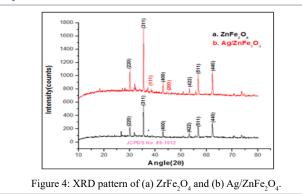
Sample		Magnetic Parameters		
	Ms (emu/g)	Hc (Oe)	Mr (emu/g)	
ZnFe2O4	1.5555	99.528	24.167	
Ag/Zn- Fe2O4	0.37020	610.87	48.745	

Compared with zinc ferrite (1.555 emu / g), the saturation magnetization value of the composite is significantly decreased by one fourth (0.3702 emu / g). This demonstrates the doping of zinc ferrite with the silver nano particle. However, the extraordinary rise in composite coercivity (610.87 Oe) compared with zinc ferrite (99.528 Oe) suggests an improvement in the composite's ferromagnetic character.



## **XRD** studies

The powder XRD pattern of ZnFe<sub>2</sub>O<sub>4</sub> and Ag/ZnFe<sub>2</sub>O<sub>4</sub> are presented in the figure 4 (a, b). In figure. 4 (a), the uppermost peak at angle  $2\theta = 35.3$  corresponds to the plane (311) for the ZnFe<sub>2</sub>O<sub>4</sub> sample precisely corresponding with the JCPDS card No. 89-1012. Other prominent peaks of zinc ferrite and their corresponding planes are well established. High crystalline nature of the compound is suggested by sharp peaks in the XDR pattern of the sample. The grafting of silver nanoparticles on the surface of zinc ferrite can be ascertained by the appearance of two low intensity peak at 38.4 and 44.6 corresponds to the plane of (111) and (200) of silver [27]. The mean crystallite size (Dc) of the ZnFe<sub>2</sub>O<sub>4</sub> and Ag/ZnFe<sub>2</sub>O<sub>4</sub> was expected to be 36.76 nm and 40.77 nm respectively using the Debye–Scherrer equation [28]. The increase in the crystallite size indicates the grafting of silver nanoparticle on the surface of zinc ferrite.



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### SEM and EDX studies

The SEM image of  $ZnFe_2O_4$  (Figure 5A, 5B) reveals that the Zn-Fe<sub>2</sub>O<sub>4</sub> has a fine-grained structure with consolidated morphology. The presence of silver in composite in dispersed form is confirmed from the SEM picture of Ag/ ZnFe<sub>2</sub>O<sub>4</sub> (Figure. 5C, 5D). The EDX spectrum of ZnFe2O4 (Figure 6A) revealed the existence of only the elements Zn, Fe, and O, with no particulates. However the EDS spectrum of Ag/ZnFe<sub>2</sub>O<sub>4</sub> (Figure 6B) confirmed the grafting of Silver in the surface of zinc ferrite.

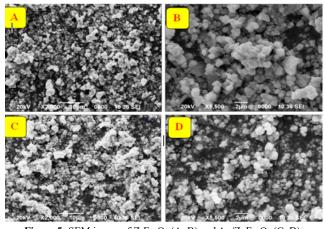


Figure 5: SEM image of  $ZrFe_2O_4$  (A, B) and  $Ag/ZnFe_2O_4$  (C, D)

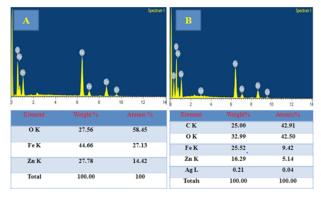


Figure 6: EDX pattern of (A) ZrFe<sub>2</sub>O<sub>4</sub> and (B) Ag/ZnFe<sub>2</sub>O<sub>4</sub>.

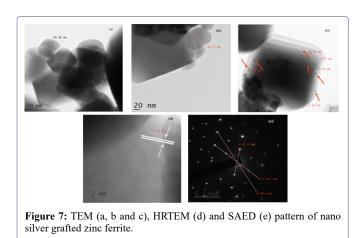
## TEM, HRTERM and SAED Studies

Transmission electron microscopy image of the as synthesized silver nano grafted zinc ferrite particles are shown in figures 7 (a, b). The cubic nature of the particles is clearly visible, with an average size of 67 nm, which varies from the average size estimated using the Debye Scherer equation from XRD observation. This disparity arises from the fact that XRD studies only take into consideration thickness in the lattice plane. With a mean size of 5.168 nm, the implanted silver nanoparticles on the surface of zinc ferrite are clearly identifiable (figure 7c). According to the HR-TEM analysis, the [220] plane corresponds to the lattice fringe length of 0.270 nm (Fig. 7d). Selected area electron diffraction (SAED) study indicates the crystalline nature of the prepared compound (figure 7e).

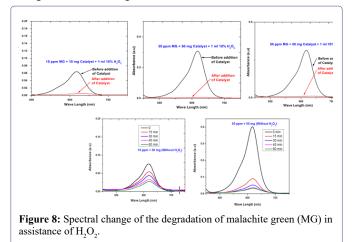
## Catalytic performance of Ag/ZnFe,O<sub>4</sub> nanocatalyst

The ability of newly synthesized silver nanoparticle grafted zinc ferrite to degrade malachite green (MG) and methylene blue (MB) at

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room temperature in the presence and absence of hydrogen peroxide was investigated. The spectrophotometric analysis makes it possible to calculate the percentage of catalytic degradation of both dyes over a given time frame. It was reported that by using 10 mg, 30 mg and 50 mg of the catalyst in the presence of 1 ml of 10%  $H_2O_2$  instantaneous degradation of the dye was observed in 10 ppm, 30 ppm, and 50 ppm concentrations of the dye. However, in the absence of  $H_2O_2$ , the degradation takes a considerable time interval, implying that  $H_2O_2$  performs a significant role in the process. In the presence of 1 ml 10 %  $H_2O_2$  this catalyst resulted in a maximum of 98 % degradation of malachite green. Fig.8 depicts the spectral changes of malachite green during instantaneous degradation under various conditions.



The degradation of methylene blue was also monitored using the same catalyst under the same conditions; however, 0nly 77 % degradation is achieved in 100 min time interval (figure 9). Thus, in comparison to the previous observation, the catalyst's efficiency in degrading methylene bule is inadequate.

#### Study of the degradation kinetics

Considering the effectiveness of the synthesized nanocatalyst in the degradation of MG over MB, the rate of the degradation of MG is only monitored and it was found to follow first order kinetics. The rate equation of degradation can be written as;  $\ln (C_0/C) = kt$ , where  $C_0$  and C are the initial and concentration of MG after time 't' with rate constant 'k' of the reaction. Concentration can be used in place of absorbance because they are both directly proportional according

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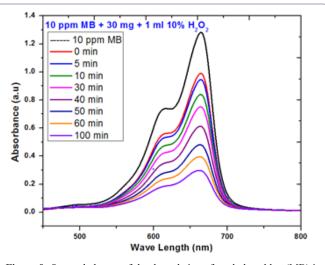
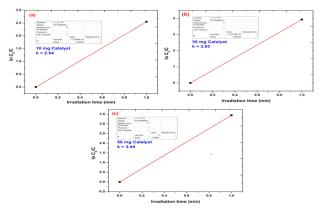
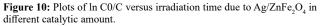


Figure 9: Spectral change of the degradation of methylene blue (MB) in assistance of  $\rm H_2O_2.$ 

to Beer-Lambert's law. Plots of  $\ln C_0/C$  against time (t) at 617 nm for different catalytic amounts showed a linear nature, as shown in the figure 10a, 10b and 10c.





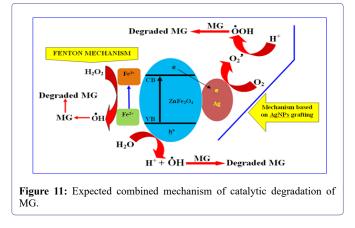
The rate constant (k) for malachite green degradation in the existence of hydrogen peroxide was found to be 2.54 min<sup>-1</sup>, 3.93 min<sup>-1</sup>, and 3.44 min<sup>-1</sup> for 10 mg, 30 mg, and 50 mg catalyst, respectively, based on these linear plots. Since this highest rate constant (k) value for 30 mg catalyst signifies a maximum degradation rate, 30 mg may be regarded as the optimum catalytic dose in this degradation process.

#### Expected mechanism of the degradation of MG

In this study, hydrogen peroxide, in addition to  $Ag/ZnFe_2O_4$  plays an important role in speeding up the rate of toxic malachite green degradation. As a consequence, the reaction kinetics of such a degradation process could be supported by fenton type mechanisms. The generation of reactive oxygen species (ROS) such as hydroxyl and perhydroxyl radicals is the primary cause of MG degradation. [29]. Furthermore, the grafted silver nanoparticles serve as a sink for electrons that pass from the valence to the conduction bands, reducing the possibility of electron-hole recombination. The superoxide anion is formed when these electrons combine with the oxygen molecule. The

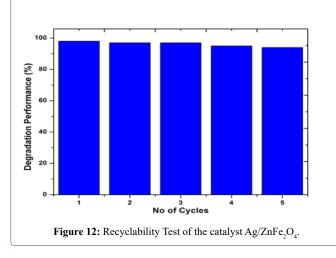
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holes in the valence band interact with the water molecule to produce hydroxyl radicals, which cause catalytic degradation [30]. The combined mechanism is shown in the figure 11.



#### Study of the recyclability of the nanocatalyst

The recyclability of the nanocatalyst was reviewed by centrifuging the catalyst from the reaction mixture. The retrieved catalyst was reused five times to degrade malachite green solution. Figure 12 show that  $Ag/ZnFe_2O_4$  nanocatalyst retains its catalytic competence after five consecutive runs for the purpose of malachite green degradation.



#### Comparative study of MG degradation with other catalyst.

The effectiveness of the  $Ag/ZnFe_2O_4$  catalyst in MG degradation was evaluated by comparing to that of other documented catalysts in the literature. With an elevated percentage of degradation,  $Ag/Zn-Fe_2O_4$  is found to be advanced and novel in this degradation strategy. The comparison is tabulated as under in Table 2.

## Conclusion

By grafting green silver nanoparticles onto the surface of zinc ferrite, a novel Ag/ZnFe<sub>2</sub>O<sub>4</sub> catalyst was successfully fabricated. The TEM analysis shows a significant visual of grafted AgNPs on the surface zinc ferrite, with an average particle size of 5.168 nm. Its exemplary performance as a catalyst is confirmed by the 98 % degradation of malachite green in one minute in the presence of hydrogen peroxide. In the occurrence of hydrogen peroxide, the catalyst was also able to degrade methylene blue. It is logical to conclude that such catalytic

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Catalyst	MG Conc	Amount	Degrada- tion Time	Percentage	Reference	
CoFe2O4/ mpg-C3N4	10 mg/L	80 mg	120 min	93.41 %	[31]	
M n O 2 - MCM	100 mg/L	100 mg	60 min	100 %	[32]	
Chitosan-Ascor- bic Acid@ NiFe2O4	70 mg/L	70 mg	90 min	99.92 %	[33]	
Zno	20 mg/L	20 mg	100 min	85.29 %	[34]	
Ni – dopped Bi2Se3	10-5 mol/L	25 mg	5 min	100 %	[35]	
Ag/ZnFe2O4	10 mg/L	10 mg	1 min	98%	This Work	
Table 2: Comparative study of MG degradation with other catalyst.						

nanomaterials can be used to remove toxic dyes from water resources, thereby mitigating environmental challenges.

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# **Graphical Abstract**



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