



**Original Article**

# Synthesis, Characterization, and Photocatalytic Application of Nickel Ferrite ( $\text{NiFe}_2\text{O}_4$ )

**Dr. Sujata S. Modhave<sup>1</sup>, Dr. Deepak Nighot<sup>2</sup>**

<sup>1,2</sup> AISSMS College of Engineering, Pune 411 001 (Affiliated to SPPU), India

## Abstract

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**Correspondence Address:**  
Dr. Sujata S. Modhave  
AISSMS College of Engineering,  
Pune 411 001 (Affiliated to  
SPPU), India  
Email:  
[modhavesujata0@gmail.com](mailto:modhavesujata0@gmail.com)

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Nickel ferrite ( $\text{NiFe}_2\text{O}_4$ ) nanoparticles were synthesized using the oxalate precursor route from  $\text{FeSO}_4$  and  $\text{NiSO}_4$ . The precursor was characterized by elemental analysis, infrared spectroscopy, and thermogravimetric analysis (TGA). Calcination was carried out at 600 °C for 4 hours under atmospheric conditions to yield the desired oxide. X-ray diffraction (XRD) confirmed the formation of a single-phase cubic spinel structure, with an average crystallite size of 46.04 nm as calculated by Scherrer's equation. The photocatalytic degradation of spent wash under visible light demonstrated enhanced catalytic performance of the  $\text{NiFe}_2\text{O}_4$  catalyst, attributable to its n-type semiconductor behaviour. The effects of catalyst dosage and spent wash concentration were systematically examined. Maximum degradation was observed in alkaline medium.

**Keywords:** Nickel ferrite ( $\text{NiFe}_2\text{O}_4$ ); Oxalate co-precipitation method; Nanoparticles; Spinel ferrite; X-ray diffraction (XRD); thermogravimetric analysis (TGA); UV-Visible spectroscopy; Band gap energy; Photocatalysis; Spent wash degradation; Visible-light activity.

## Introduction

Ferrites, or ferromagnetic oxides, are ceramic materials consisting of iron-containing compounds. Typically, dark brown or grey in colour, ferrites are hard, brittle, and electrically non-conductive. Their magnetic properties differ significantly from those of metallic ferromagnets, primarily due to low electrical conductivity, which minimizes eddy current and ohmic losses. Nickel and zinc ferrites have been widely explored owing to their magnetic, optical, and catalytic properties. Despite extensive research, optimization of synthesis parameters remains incomplete. Among available preparation methods, the co-precipitation technique offers advantages of homogeneity, fine particle size, and reduced need for repeated grinding and calcination steps. Its simplicity and scalability further support its suitability for industrial applications.

The present work focuses on the synthesis of  $\text{NiFe}_2\text{O}_4$  nanoparticles via the oxalate co-precipitation method, their physicochemical characterization, and their application in the photocatalytic degradation of spent wash.

## Experimental

### Synthesis of Nickel Ferrite ( $\text{NiFe}_2\text{O}_4$ ) Nanoparticles

A mixture of 14 g  $\text{NiSO}_4$  and the required stoichiometric amount of  $\text{FeSO}_4$  was dissolved in a minimum quantity of distilled water. To this solution, 8.8 g of sodium oxalate was added dropwise over 2 hours under continuous stirring and heating. Upon formation of a precipitate, heating and stirring were discontinued, and the mixture was cooled to room temperature. A small quantity of acetone was added, followed by stirring for 1 hour to facilitate nanoparticle formation.

The precipitate was filtered through Whatman No. 41 filter paper and dried under an infrared lamp. The dried material was ignited for 2 hours, ground for 1 hour, and calcined at 600 °C for 2 hours in a muffle furnace. After cooling, it was ground again for 1 hour. The procedure was repeated twice to ensure complete formation of nickel ferrite nanoparticles.

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## Results and Discussion

Study of ferrite was done by using different characterization techniques such as IR Spectroscopy, Thermo gravimetric analysis, X-ray powder diffraction, UV-Visible spectroscopy.

For the characterization of precursor of metal analysis is done and concentration of metal (Fe, Ni) was found by Volumetric analysis. Observed concentration of Fe and Ni is well match with calculated value. the infrared spectra of Precursor i.e.  $\text{NiFe}_2\text{O}_4(\text{C}_2\text{O}_4)\cdot 5\text{H}_2\text{O}$ . It is Clearly seen that the carboxyl ate band change is due to ligand and metal bonding, the possible band assignment of fundamental

### Degradation of Spent Wash using Nickel ferrite:

#### 1. pH= 2:

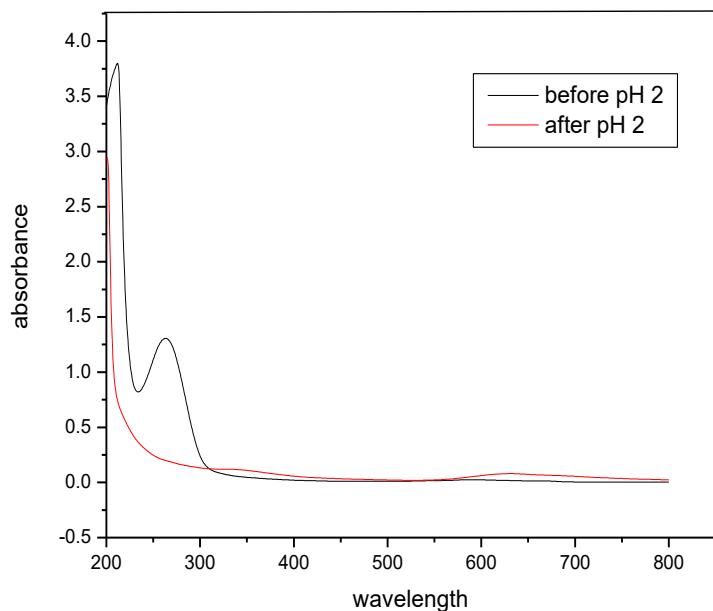


Fig. 1 % Degradation against amount of catalyst 25 mg is 48.73 %

#### 2. pH= 4:

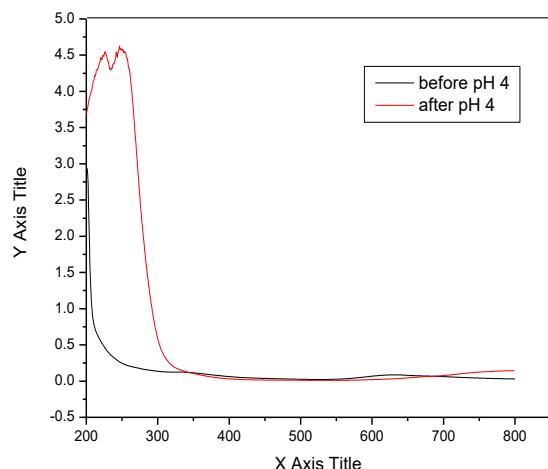


Fig. 2 Degradation is not done

frequency for oxalate group OH AND M-O etc., are given, lattice water absorbs at around  $3359.8\text{ cm}^{-1}$  Although this precursor absorbs clearly near  $3318\text{ cm}^{-1}$  which is due to OH stretching vibration. The band at around  $1618\text{ cm}^{-1}$  correspond to  $\nu_{\text{sys}}$  ( $\text{C}=\text{O}$ ) and band at  $1361\text{ cm}^{-1}$  Corresponding to  $\nu_{\text{sys}}$  ( $\text{C}-\text{O}$ ), Suggest that oxalate group Bentley linked to metal ions. The polymeric octahedral Structure have been assigned to this precursor. the band at  $492\text{ cm}^{-1}$  assigned for M-O BAND. The metal (M-O) Band at  $492\text{ cm}^{-1}$  indicates a six-co-ordinate environmental of metal ions. Figure 2 represent the TGA.



3. pH=8:

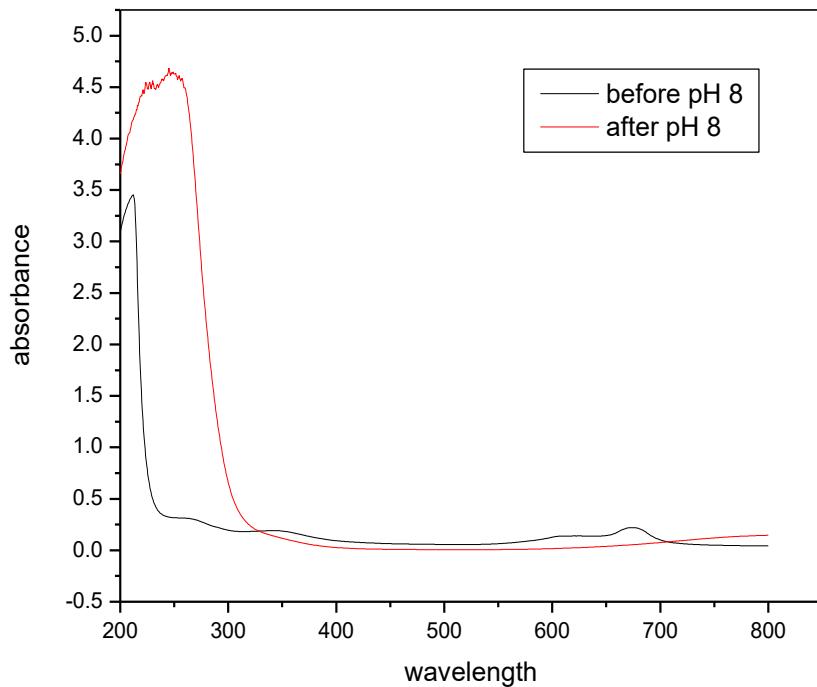


Fig. 3 % Degradation against amount of catalyst 25 mg is 15.34 %

4. pH=10:

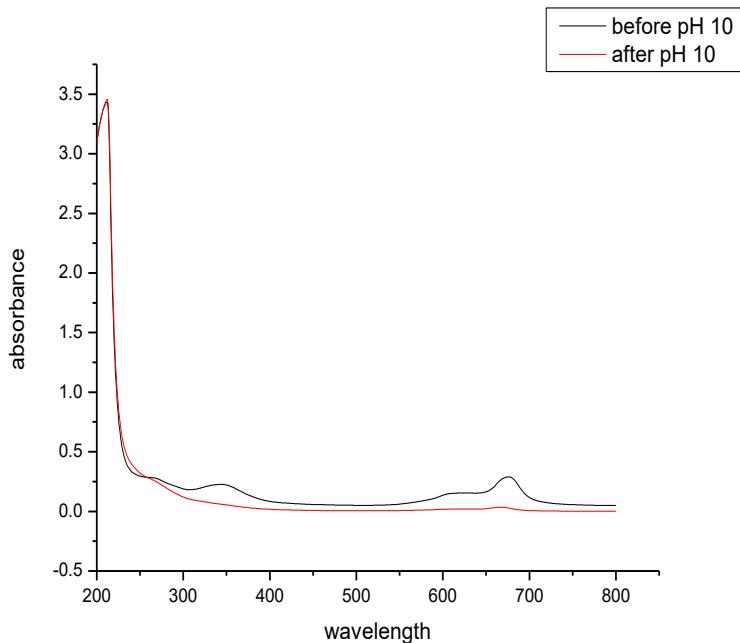
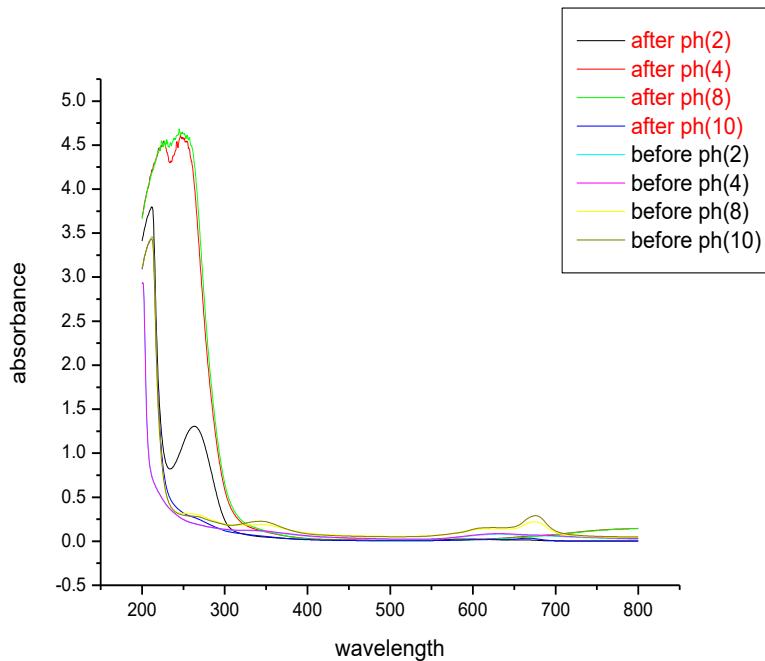


Fig. 4 % Degradation against amount of catalyst 25 mg is 15.34 %



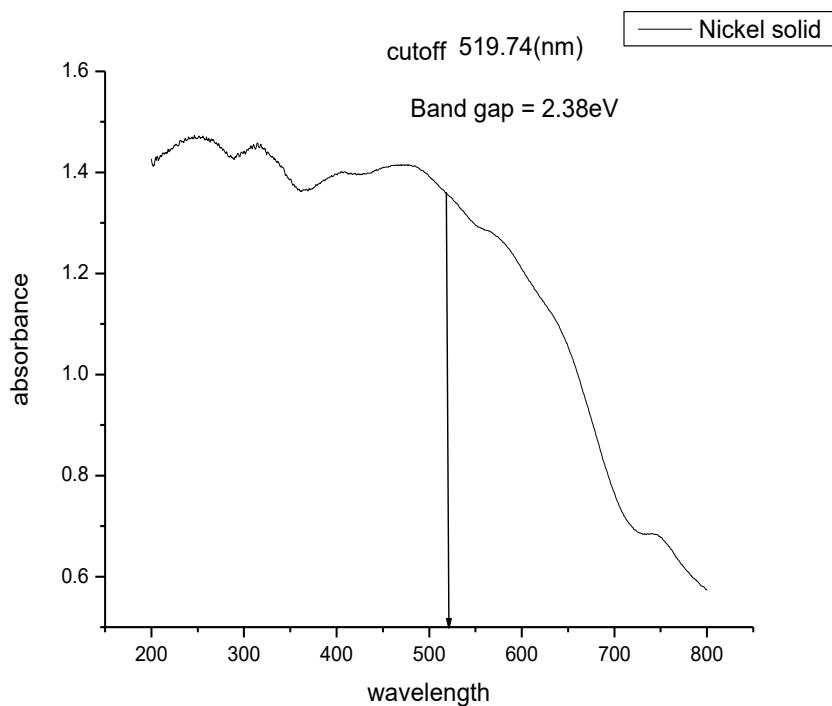
**5. Common Plot:**



**Fig. 5 Degradation of spent wash before & after for 25mg**

**Solid UV-Visible:**

**1. Solid UV for Ni Ferrite:**



**Fig. 8 The Band Gap Observe for Ni Ferrite is 2.38 eV**

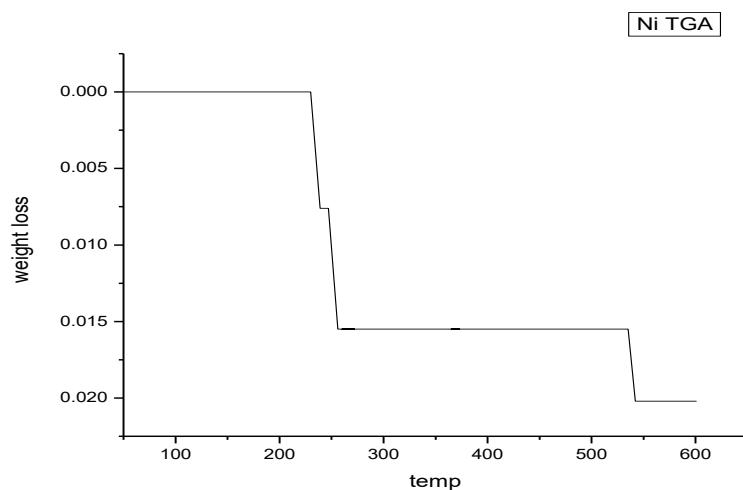


**Band Gap Table**

Compound Name	Band Gap
Nickel Ferrite	V

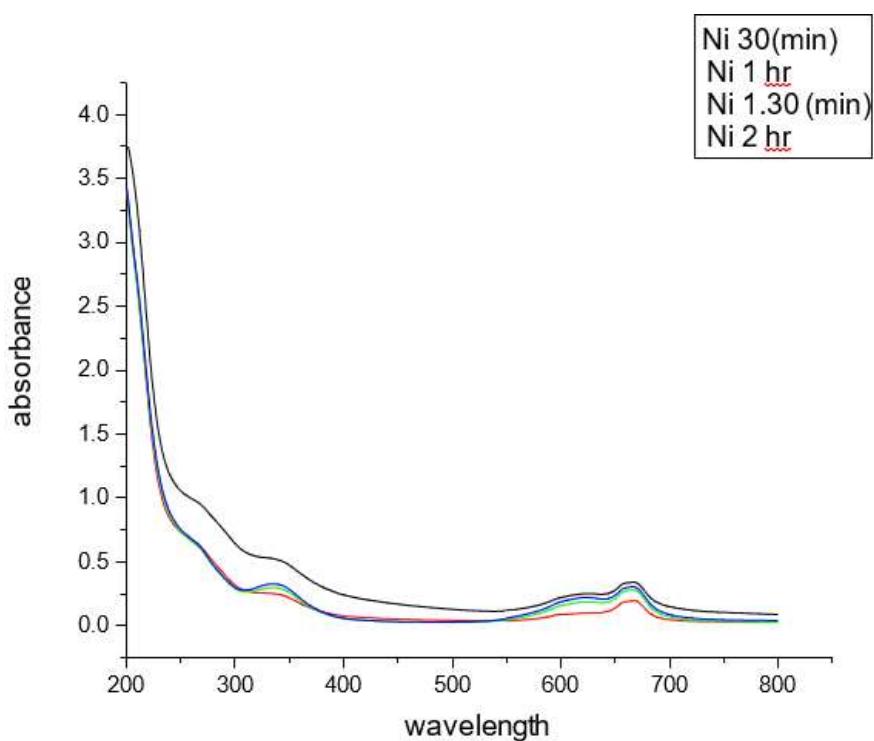
**Thermo Gravimetric Analysis:**

**TGA of Nickel Oxalate:**



**TGA Curve for  $\text{Ni Fe}_2(\text{C}_2\text{O}_4)_3 \cdot 5\text{H}_2\text{O}$**

**Kinetic Study of Nickel Ferrite in Neutral Condition:**

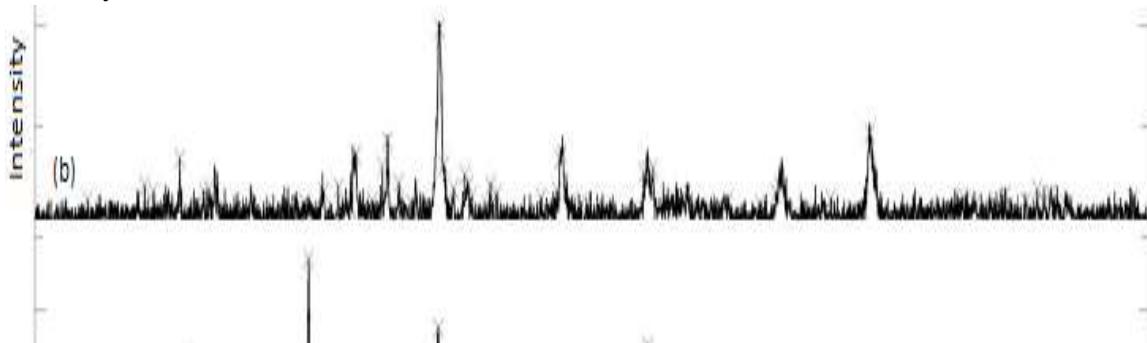


**Kinetic Study of Ni in Neutral Condition**

**Kinetic Study of Ni in Neutral Condition**



**4.4 XRD Analysis:**



**XRD analysis**

**X-ray diffraction data for d-value:**

NiFe <sub>2</sub> O <sub>4</sub>
5.279(21)
5.109(18)
4.662(35)
3.076(20)
2.968(37)
2.786(44)
2.728(23)
2.536(100)
2.517(31)
2.105(34)
2.098(37)
1.886(27)
1.865(30)
1.616(21)
1.487(47)
1.211(15)

**Photocatalytic Activity:**

**Spent wash indicator:**

Nickel ferrite showed catalytic activity in spent wash indicator.



**Spent Wash showed catalytic activity in neutral condition**



## Photo catalytic property:

Photo catalytic experiments were conducted using photo catalyst to photo catalytically degrade spent wash. Reaction suspension were prepared by adding  $\text{NiFe}_2\text{O}_4$  Photo catalyst powder (25mg) into 50ml of aqueous spent wash solution prior to Irradiation, the suspension were magnetically stirred in a dark Condition for 2 hr to establish

- Degradation of Paint Wash using 'Ni ferrite'

an adsorption desorption equilibrium. The suspension containing spent wash and Photo catalyst were then irradiated under sunlight. At given Time interval 2 hr, the reaction suspension and centrifuged at 1000 rpm for min to remove the partial and the filtrate was Then measured the absorption (O.D.>) on UV spectroscopy at max = 616nm. the percentage degradation efficiency has been calculated as

Before		After	
Wavelength	Absorbance	Wavelength	Absorbance
PH= 2 = 335	0.119	PH= 2 = 335	0.055
PH = 4 = 335	0.124	PH = 4 = 335	0.462
PH = 8 = 335	0.189	PH = 8 = 335	0.071
PH = 10 = 335	0.222	PH = 10 = 335	0.05

Photocatalytic degradation of spent wash

% Degradation of  $\text{NiFe}_2\text{O}_4$

1) PH=2=53.78  
2) PH = 4= 27.5  
3) PH = 8 = 61.41  
4) PH = 10 = 77.47



Nickel Nanoparticle

## Conclusion

Nickel ferrite ( $\text{NiFe}_2\text{O}_4$ ) nanoparticles were successfully synthesized via the oxalate co-precipitation method. IR analysis confirmed bidentate coordination in the precursor, while TGA indicated two-step decomposition leading to ferrite formation. XRD confirmed the formation of a single-phase cubic structure with a crystallite size of 46.04 nm. UV-Visible analysis showed a band gap of 2.38 eV.

The synthesized  $\text{NiFe}_2\text{O}_4$  nanoparticles exhibited significant photocatalytic activity in the degradation of spent wash, with the highest efficiency observed at pH 10.

Catalyst dosage and pH played a critical role in degradation efficiency. These results suggest that  $\text{NiFe}_2\text{O}_4$  is a promising material for environmental remediation applications.

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characterization, and photocatalytic studies of nickel ferrite nanoparticles.

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#### Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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