

# Synthesis and Characterization of Nanocrystalline Nickel Ferrite by Co-precipitation Technique

Dr. S. S. Modhave

AISSMS College of Engineering, Pune

Corresponding Author E-mail: modhavesujata0@gmail.com

**Abstract:** Nanocrystalline nickel ferrite ( $\text{NiFe}_2\text{O}_4$ ) powders were synthesized using a simple and cost-effective co-precipitation technique. Stoichiometric amounts of nickel and iron salts were dissolved in aqueous medium and co-precipitated by the controlled addition of a base under constant stirring. The precipitates were thoroughly washed, dried, and subsequently calcined at different temperatures to obtain phase-pure nickel ferrite nanoparticles. Structural characterization using X-ray diffraction (XRD) confirmed the formation of a single-phase cubic spinel structure, and the average crystallite size was estimated using the Scherrer formula, indicating nanometer-scale dimensions. Fourier transform infrared (FTIR) spectroscopy revealed characteristic metal–oxygen vibrational bands corresponding to tetrahedral and octahedral sites of the spinel lattice. The morphology and particle size distribution were examined by scanning electron microscopy (SEM), showing agglomerated but uniformly distributed nanoparticles. Magnetic properties studied using a vibrating sample magnetometer (VSM) demonstrated ferrimagnetic behavior with size-dependent saturation magnetization and coercivity. The results indicate that the co-precipitation method is an efficient route for synthesizing nanocrystalline nickel ferrites with controlled structural and magnetic properties, making them suitable for applications in magnetic storage, sensors, and biomedical fields.

**Keywords:** Nanocrystalline, characterization, Nickel ferrite, Stoichiometric.

## I. INTRODUCTION

Spinel ferrites with the general chemical formula  $\text{AB}_2\text{O}_4$  represent an important class of magnetic ceramic materials widely used in electronic and electromagnetic applications. Nickel ferrite ( $\text{NiFe}_2\text{O}_4$ ) is a well-known soft magnetic material characterized by high electrical resistivity, moderate saturation magnetization, low eddy current losses, good chemical stability, and mechanical strength.

$\text{NiFe}_2\text{O}_4$  crystallizes in an inverse cubic spinel structure, where  $\text{Ni}^{2+}$  ions predominantly occupy octahedral (B) sites and  $\text{Fe}^{3+}$  ions are distributed between tetrahedral (A) and octahedral (B) sites. The magnetic properties originate from super-exchange interactions between cations at A and B sites mediated by oxygen ions.

When the particle size is reduced to the nanometer scale, significant modifications occur in magnetic and structural behavior due to finite size effects, surface spin disorder, and increased surface-to-volume ratio. These nanoscale characteristics enhance the functional performance of nickel ferrite in applications such as magnetic storage devices, microwave absorbers, ferrofluids, gas sensors, catalysts, and biomedical systems.

Several synthesis routes including sol-gel, hydrothermal, combustion, microemulsion, and mechanical alloying have been reported for the preparation of  $\text{NiFe}_2\text{O}_4$  nanoparticles. Among these, the co-precipitation technique offers advantages such as simplicity, cost-effectiveness, low synthesis temperature, scalability, and good compositional control. The technique enables uniform distribution of cations at the molecular level under controlled pH conditions.

The present work focuses on synthesizing nanocrystalline nickel ferrite via co-precipitation and systematically studying its structural, morphological, and magnetic properties.



## II. EXPERIMENTAL PROCEDURE

### 2.1 Materials

Analytical grade chemicals were used without further purification:

Nickel nitrate hexahydrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ )

Ferric nitrate nonahydrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ )

Sodium hydroxide (NaOH)

Ethanol

Deionized water

The molar ratio of  $\text{Ni}^{2+}$  to  $\text{Fe}^{3+}$  was maintained at 1:2.

### 2.2 Synthesis Method

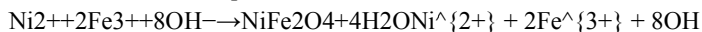
#### Step 1: Preparation of Precursor Solution

Calculated amounts of nickel and iron nitrates were dissolved separately in deionized water and then mixed to obtain a homogeneous solution. The mixture was stirred continuously for 30 minutes.

#### Step 2: Precipitation

A 1 M NaOH solution was added dropwise to the mixed solution under constant stirring until the pH reached 10–11. A dark brown precipitate formed, indicating hydroxide formation. The mixture was maintained at 80 °C and stirred for 2 hours to promote nucleation and growth.

The reaction can be represented as:



#### Step 3: Aging

The precipitate was allowed to age for 12–24 hours to enhance crystallinity.

#### Step 4: Washing and Drying

The product was filtered and washed repeatedly with deionized water and ethanol to remove impurities. The washed precipitate was dried at 100 °C for 12 hours.

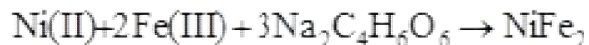
#### Step 5: Calcination

The dried powder was calcined at 500 °C and 700 °C for 3 hours with a heating rate of 5 °C/min to obtain crystalline  $\text{NiFe}_2\text{O}_4$  nanoparticles.

## III. RESULTS AND DISCUSSION

### 3.1 Synthesis of Precursor :

The metal tartarate precursor was prepared from the reaction between metal ion solutions and sodium tartarate in solution state. When metal ion solution and sodium tartarate were mixed in appropriate proportion and it resulted into the formation of precipitate, for which reaction can be written as follows:



#### Conversion of Precursor to Oxides:

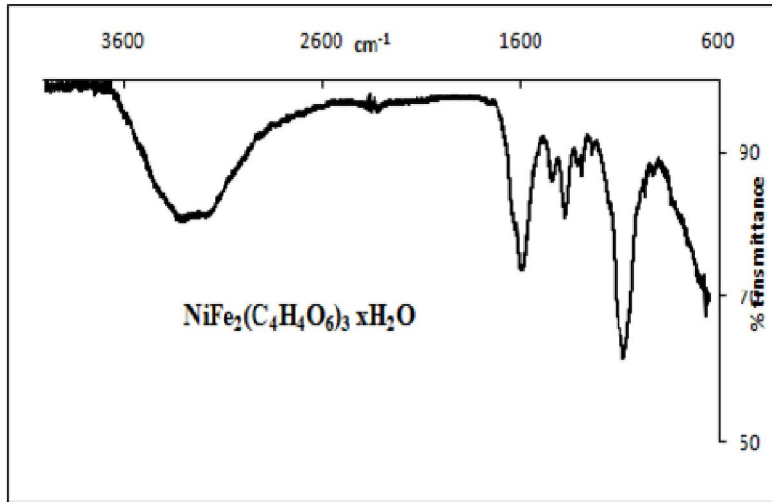
When precursor was heated at 600 °C its converted to oxide form



### 3.2 IR Spectral Analysis of Precursor:

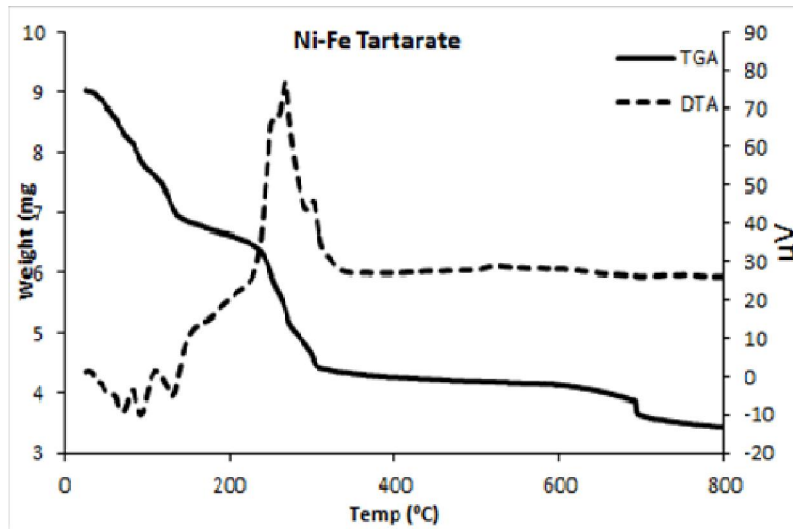
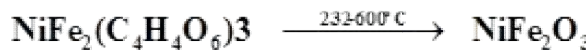
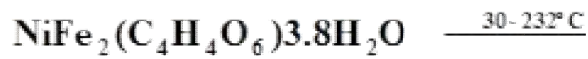
IR spectroscopy shows the signal of different functional group present in compound at particular frequency





### 3.3 Thermal analysis:

On the basis of TGA and DTA studies, the following tentative scheme is proposed for the thermal decomposition of tartarate precursor in air.



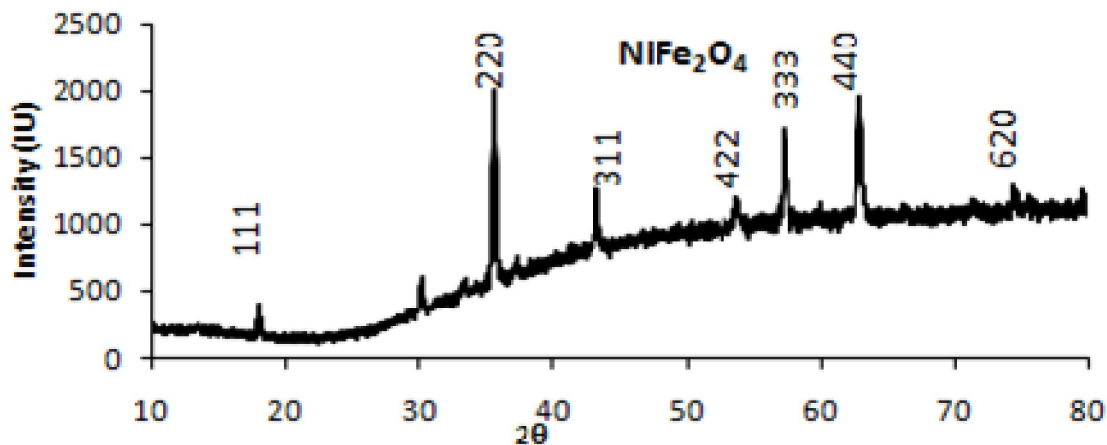
### 3.4 Chemical Analysis:

Percent content of metal ions in Nickel Ferrites is shown in following table:

Ferrite	Percentage of Fe(III)	Percentage of Ni(II)		
	Observed (%)	Expected (%)	Observed (%)	Expected(%)
NiFe <sub>2</sub> O <sub>4</sub>	43.63	47.65	26.11	25.04

### 3.5 X-ray powder diffraction analysis of Ferrites:

Powder XRD patterns of synthesized ferrites were recorded and analysed for 2 $\theta$  and d values. From 2 $\theta$  and d values, planes in crystalline phase were assigned. From planes in crystalline phase was identified. The typical XRD of ferrites are represented in following figure:



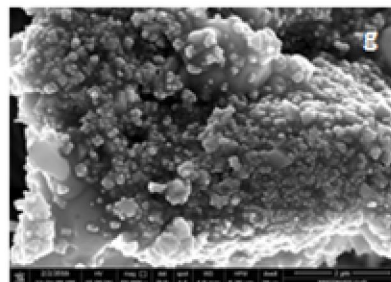
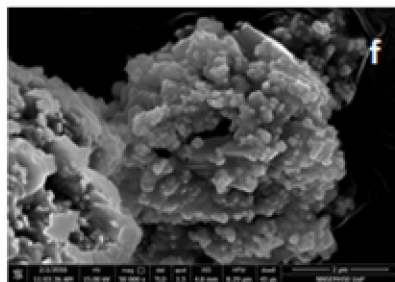
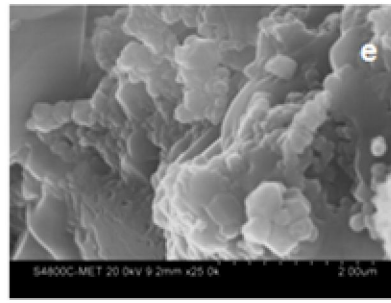
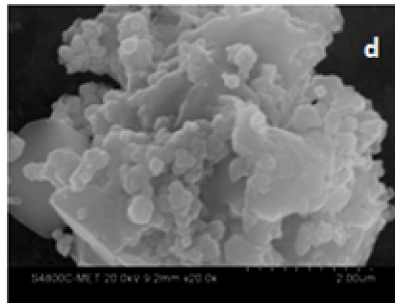
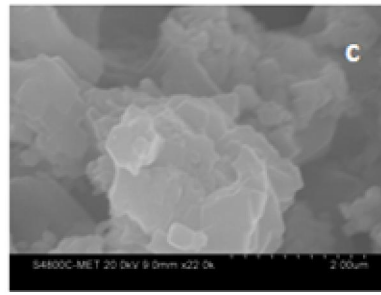
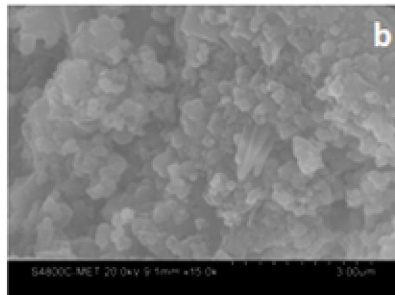
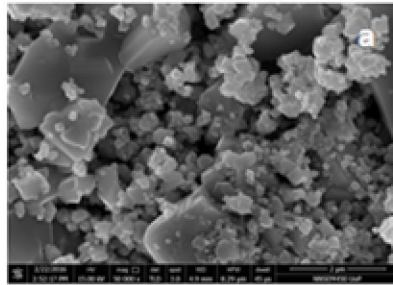
Comparison of observed d values of nickel ferrite from XRD pattern with reported values and respectively assigned planes:

NiFe <sub>2</sub> O <sub>4</sub>	Reported d-values	Reported planes
4.845	4.8138	[111]
2.959	2.9478	[220]
2.526*	2.5139*	[311]
2.382	2.4069	[222]
2.089	2.0844	[400]
1.901	1.9128	[331]
1.703	1.7019	[422]
1.608	1.6048	[511]
1.542	1.4739	[440]
1.476	1.4093	[531]
1.413	1.3896	[442]
1.321	1.3183	[620]
1.270	1.2715	[533]
1.262	1.2569	[622]
1.205	1.2034	[444]



**3.6 Crystal parameter of nickel ferrite:**

Sr. No.	Ferrite	a (Å°)	Crystal Volume (Å° <sup>3</sup> )	X-ray density (D <sub>x</sub> ) g/cm <sup>3</sup>	Bulk density g/cm <sup>3</sup>	Porosity	Crystallite size (nm)
	NiFe <sub>2</sub> O <sub>4</sub>	8.333	578.63	5.380	2.54	0.528	36.6
	JCPDS	8.337	579.47	5.373			----



### 3.7 Surface Morphology by FFSEM:

FESEM images of nickel shows very high agglomeration of crystallite into mixed shaped grains and it is reflected in terms of very high grain size than that of crystallite sizes. Grain sizes are found from 30 nm to 200 nm for same ferrite material. The grain sizes of doped nickel ferrites are found **less than that of pure nickel ferrite**.

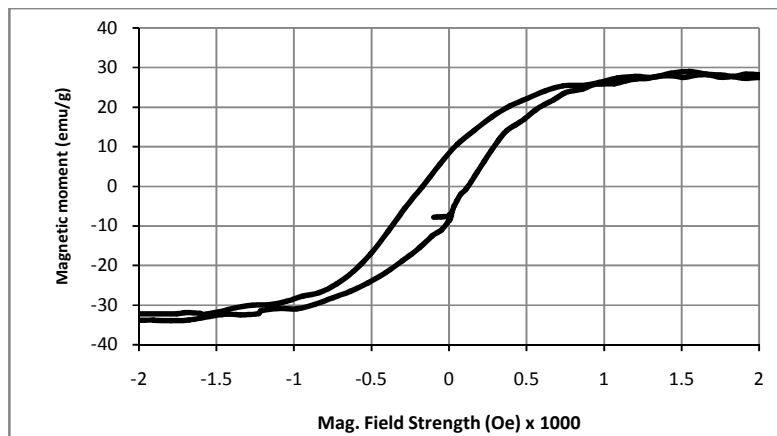
### 3.8 Electrical conductivity data of nickel ferrite:

Compound	Temperature corresponding to desorption of adsorbed water (K)	$\sigma$ at 500 K ( $\Omega^{-1}\text{cm}^{-1}$ )	Ferrimagnetic region	Paramagnetic region	Transition temperature ( $T_c$ ) K
$\text{NiFe}_2\text{O}_4$	365	$2.66 \times 10^{-5}$	0.273	0.427	560

### 3.9 Magnetic properties for nickel ferrite:

Compound	Coercive force $H_c \pm 0.5$ Oe	Saturation magnetization (Ms) $\pm 2\text{emu/g}^{-1}$	Ratio of $M_R/M_S$	Magnetic moment $n_B \pm 0.1 \mu_B$		Magnetic moment $n_B \pm 0.1 \mu_B$
				Observed	Calculated	
$\text{NiFe}_2\text{O}_4$	59.57	28.98	0.392	1.263		2.83

### Hysteresis loops for nickel ferrites



## IV. CONCLUSION

Nanocrystalline  $\text{NiFe}_2\text{O}_4$  was successfully synthesized using a controlled co-precipitation method. Structural studies confirmed single-phase cubic spinel formation with nanoscale crystallite size.

Magnetic analysis revealed ferrimagnetic behavior influenced by particle size and crystallinity. The co-precipitation technique provides an effective and economical method for producing nickel ferrite nanoparticles suitable for magnetic and electronic applications.



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