# RESEARCH



# Effect of calcination temperature on structural and magnetic properties of polypropylene glycol stabilized nickel ferrite nanoparticles



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# Abstract

**Background** Aqueous route of sol–gel method was used to synthesize nanocrystalline nickel ferrite (NiFe<sub>2</sub>O<sub>4</sub>) from a stoichiometric mixture of nickel nitrate hexahydtrate and ferric nitrate nonahydrate precursors using aqueous ethanol as solvent and polypropylene glycol as a stabilizing agent. The gel obtained was calcined at various temperatures ranging from 500 °C to 900 °C. The effect of calcination temperature on structural and magnetic properties of nickel ferrite was determined by X–ray diffraction, infrared spectroscopy, thermogravimetry, field emission scanning electron microscopy and vibrating sample magnetometry.

**Results** The results showed a single phase nickel ferrite with a cubic spinel structure having particles of irregular shape and different sizes ranging from 10 to 20 nanometers, randomly distributed to form aggregates.

**Conclusions** The results revealed that the use of polypropylene glycol as a stabilizing agent significantly reduced the agglomeration of nickel ferrite nanoparticles. However, loosely aggregated grains were obtained that got separated with the rise in calcination temperature leading to the formation of more prominent and well–dispersed structures at 900 °C. Moreover, the samples exhibited high coercivity indispensible for the application of nanoparticles in storage and magnetic devices.

**Keywords** Sol–gel, Nickel ferrite, X–ray diffraction, Infrared spectroscopy, Thermogravimetry, Field emission scanning electron microscopy, Vibrating sample magnetometry

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# Introduction

Nickel ferrite exhibits inverse spinel structure having a face–centered cubic lattice with octahedral sub–lattice occupied by Fe<sup>3+</sup> and Ni<sup>2+</sup> and tetrahedral sub–lattice occupied by the remaining Fe<sup>3+</sup>. The magnetic moment originated from antiparallel spins between Fe<sup>3+</sup> at tetrahedral sites and Ni<sup>2+</sup> at octahedral sites results in ferromagnetic nature of nickel ferrite [1, 2]. Nickel ferrite (NiFe<sub>2</sub>O<sub>4</sub>) has attracted much attention among magnetic nanomaterials owing to its distinct magnetic and other physical properties including outstanding permeability at high frequencies, mechanical hardness and electrical properties. Due to its high electrochemical stability, low



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conductivity, reduced coercive field and minor dielectric constant and eddy current losses, nickel ferrite belongs to soft ferrites. It has a high-resistance and shows a resistivity of more than 109  $\Omega$ m. It is used in the wide range of applications in high density magnetic storage apparatus, telecommunication devices, microwave devices, magnetic fluids, magnetically facilitated drug delivery, magnetic resonance imaging and catalysts etc [2]. Though nickel ferrite has been widely investigated as a magnetic material but it has been found to be highly sensitive towards gas-sensing due to its both n-type and p-type semiconducting behaviors; it has been reported that ntype nickel ferrite is sensitive towards reducing gases whereas its p-type leads to sensitivity for oxidizing gases [3]. Moreover, nickel ferrite is the most likely applied matrix of inert anode for aluminum electrolysis due to its high corrosion resistance and electrochemical stability in molten cryolite [2]. Magnetic behavior of these nanoparticles mainly depends on their purity, size, shape and magnetic strength; therefore, they must retain a pure phase, single domain, suitable coercivity, mild magnetization and consistent blocking properties that make them of great significance for their applications. Consequent upon the higher tendency of fine particles to retain magnetization, finer particles are inclined to have a single magnetic domain, whereas larger particles have several domains [4, 5].

Among various nanosize metal oxides, nickel ferrite is considered as one of the most promising metal oxides due to its simplicity of synthesis [6]. Nickel ferrite can be obtained by a variety of methods such as hydrothermal, sol-gel, co-precipitation, reverse micelle, solvothermal and polymeric precursor [7]. Among these techniques, sol-gel is simple and inexpensive as it operates at a lower reaction temperature. It is believed that the reaction parameters including reaction temperature and pH of the reactants are important in regulating the particle nucleation and growth rates and hence, the subsequent particle morphology. The alternative method for controlling the particle morphology that specifically results in reduced agglomeration involves the use of surfactants or some organic solvents, because of their properties to induce electrostatic repulsion and steric hindrance among nanoparticles. Sivakumar et al. used polyvinyl alcohol (PVA) for controlling particle size in the synthesis of nickel ferrite through sol-gel auto-combustion method [8]. Researchers have also reported the use of various chelating agents such as citric acid, cellulose, cellulose-citric acid mixture, maleic acid and urea for regulating particle size and controlling agglomeration of particles during synthesis of nanosize nickel ferrite through sol-gel method but high degree of agglomeration was observed [9, 10]. Therefore, sol-gel method for the synthesis of nickel ferrite nanoparticles still needs further investigation.

In the present study, nickel ferrite nanoparticles were synthesized via the aqueous route of sol-gel method using polypropylene glycol for controlling the agglomeration. The structural and magnetic properties of the prepared nickel ferrite nanoparticles calcined at temperatures ranging from 500 °C to 900 °C were studied by XRD, IR, TGA, FESEM and VSM techniques.

## **Experimental**

# Reagents

Nickel nitrate hexahydrate (Thermo Scientific 99%), iron (III) nitrate nonahydrate (Uni–Chem 98%), ethyl alcohol (RCI Labscan>99.8%), polypropylene glycol (99%) and nitric acid (Merck 70%) were used.

### Synthesis of nickel ferrite nanoparticles

Nickel ferrite nanoparticles were synthesized by aqueous sol-gel route in acidic medium. 39.3 g of nickel nitrate hexahydrate and 51.2 g of ferric nitrate nonahydtrate were added as precursors to a mixture of solvents i.e. ethyl alcohol and water in 1:1 ratio. 10 mL of propylene glycol were added and a few drops of nitric acid were used to maintain pH of the solution at 1. The mixture was stirred constantly for four hours at 50 °C. The sol thus formed was dried at 105 °C for twenty four hours to obtain gel. The gel after fine grinding was divided into five portions that were calcined at 500 °C, 600 °C, 700 °C, 800 °C and 900 °C for two hours and were labeled as NF-1, NF-2, NF-3, NF-4 and NF-5 respectively. The graphical representation of the process is shown in Fig. 1.

#### Characterization

The phase composition of synthesized nickel ferrite nanoparticles was investigated by means of X-ray diffractometer Bruker D8 Advance. The diffractograms were recorded between 0° and 100° 20 range using monochromatised CuKα radiation having wavelength of 1.5406Å. Infrared spectroscopy was carried out with infrared spectrometer Agilent Technologies. Thermogravimetric analysis was conducted using differential scanning calorimeter Universal V4.5 A, TA instruments USA, in nitrogen atmosphere from room temperature to 1000 °C. Surface morphology studies were performed using field emission scanning electron microscope FEI Nova 450 NanoSEM. The magnetic properties of the samples were measured at room temperature with a vibrating sample magnetometer (VSM), Cryogen-Free High Field Measurement System, applying a maximum magnetic field of 6 T and the values of parameters such as the saturation magnetization  $(M_r)$ , remanent magnetization  $(M_r)$  and the coercivity  $(H_c)$  were calculated.



Fig. 1 Graphical representation of sol-gel synthesis of nickel ferrite nanoparticles



Fig. 2 (a) XRD pattern of NF-1. (b) XRD pattern of NF-2. (c) XRD pattern of NF-3. (d) XRD pattern of NF-4. (e) XRD pattern of NF-5

## **Results and discussion**

Nickel ferrite nanoparticles have great tendency to agglomerate due to strong magnetic attraction among particles, van der Waals forces and high surface energy. Consequently, they form larger assemblies as observed by most of the researchers in the past; they obtained agglomerates of nickel ferrite nanoparticles having size in micrometric range [11, 12]. In order to enhance the dispersibility of nanosize particles in an aqueous medium and prevent agglomeration, addition of an appropriate stabilizing agent during synthesis can play a significant role. Present study is therefore aimed at the sol-gel synthesis of nickel ferrite nanoparticles using polypropylene glycol as a stabilizing agent. Polypropylene glycol being a long chain compound inhibits crystal growth and prevents agglomeration through stearic interactions. Further, it is a high boiling solvent as compared to water and ethanol; its slow evaporation controls the nucleation rate and particle growth in the aqueous medium. In fact, quick particle growth leads to increased particle size due to fast nucleation rate because of accelerated core formation. So preferably, the nucleation rate must be higher than the growth rate for attaining smaller particle size.

## X-ray diffraction studies

X-ray diffraction patterns of the nickel ferrite nanoparticles calcined at 500 °C, 600 °C, 700 °C, 800 °C and 900 °C are shown in Fig. 2a-e. The patterns show distinct peaks in the scanned  $2\theta$  range from 0° to 100°. All the peaks indexed on the basis of JCPDS data (Card No. 10-0325) correspond to the single phase face-centered cubic structure of inverse spinel nickel ferrite. The patterns show well–defined reflections at  $2\theta$  values of 30.5, 35.4, 37.4, 43.6, 54.0, 57.7 and 63.3 corresponding to the crystal planes (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) respectively. Furthermore, the slight broadening observed among the diffraction peaks is attributed to the nanocrystalline nature of the particles. It is also evident from the results that the variation in calcination temperature strongly influences the intensity of diffraction peaks; the higher calcination temperatures lead to more intense diffraction peaks owing to larger grain size and greater crystallinity. No additional diffraction peaks related to impurities such as Fe<sub>2</sub>O<sub>3</sub> and NiO can be observed. All

these results are in close agreement with those reported in literature [13–15].

The XRD data was used to obtain average crystallite size (t) calculated by using Scherrer's formula (1):

$$t = \frac{9.0\lambda}{\beta \cos\theta} \tag{1}$$

where,  $\lambda$  denotes the wavelength,  $\beta$  stands for FWHM and  $\theta$  is the diffraction angle. The results given in Table 1 show that the average crystallite size ranges between 13 and 15 nm.

## Infrared spectroscopy

The IR absorption spectra of nickel ferrite samples recorded in the range of 4000–650 cm<sup>-1</sup> are illustrated in Fig. 3a-e. The figures show broad band of weak intensity for each NF sample in the region between 3800 and 3000 cm<sup>-1</sup> ascribed to the stretching vibrations of the O-H bonds of hydroxyl groups due to the adsorption of water molecules on the surface of nanoparticles as well as hydrogen bonded hydroxyl groups. Another weak band observed in the range of  $2400-2200 \text{ cm}^{-1}$  appears due to the presence of hydrogen bonding between OH groups [16]. Other weak bands of quite low intensity at 1640 cm<sup>-1</sup> and 1476 cm<sup>-1</sup> correspond to the O-H bending vibrational modes and stretching vibrations of entrapped nitrate groups respectively [5]. The distinct absorption band in the region below 1000 cm<sup>-1</sup> is assigned to the stretching modes of metal-oxygen bonds that confirms the formation of nickel ferrite. Similar strong absorption bands around 600 cm<sup>-1</sup> have been reported in the literature for Fe-O stretching modes of nickel ferrite nanoparticles [17-19]. The broadness of this band specifies the nanosize of particles [20].

#### Thermogravimetric analysis

The thermal behavior of NF samples was investigated by thermogravimetric analysis heating in the temperature range from room temperature to 1000 °C in nitrogen atmosphere (Fig. 4a–e). The TGA curves show similar behavior among all the NF samples. In the first step, a continuous mass loss of 5% up to 450 °C can be attributed to the removal of adsorbed water and organic

 Table 1
 Crystallite size of NF samples calculated by Scherrer's formula

lennala				
Crystallite size (t) (nm)				
13.9241				
14.4877				
14.4810				
13.5617				
14.8811				

residues entrapped in the nanoparticles during their synthesis [21]. A second step of significant weight gain i.e. 47–54% between 480 °C and 520 °C can be seen indicating the phase transformation of metal oxides [22]. In fact the reducing atmosphere in the presence of nitrogen results in a rapid weight gain by the samples leading to the conversion of Fe<sub>2</sub>O<sub>3</sub> to Fe<sub>3</sub>O<sub>4</sub> [23]. Further increase in temperature up to 1000 °C had negligible effect on the thermal stability of NF samples. The thermal behavior of intermediate formed as gel before calcination of NF samples (Fig. 4f) confirms the complete transformation of precursors to metal oxides below the lowest calcination temperature i.e. 500 °C. Hence, the thermogravimetric analysis demonstrates the complete conversion of precursors into metal oxides.

#### Morphological characterization

The surface morphology of NF samples was studied by field emission scanning electron microscopy at 50,000× magnification and micrographs are illustrated in Fig. 5ae. The micrographs show nonhomogeneous structures with aggregated particles of different sizes and irregular shapes. It can be noticed that more aggregation is found in NF-1 calcined at 500 °C as compared to the other NF samples calcined at higher temperatures. The results reveal that loosely aggregated grains got separated and the grain size increased with the rise in calcination temperature leading to the formation of more prominent and well-dispersed structures at 900 °C. The average grain size was found to be in the range of 10-20 nm. In the past, researchers obtained agglomerates of nickel ferrite having size in micrometric range owing to high surface energies of nanoparticles resulting in the formation of larger assemblies [11, 12].

### Magnetic characterization

The magnetic properties of nickel ferrite samples were recorded by using hysteresis loops obtained at room temperature with the applied field ranging up to 6 T as shown in Fig. 6a-e. Saturation magnetization (M<sub>s</sub>), remanent magnetization (M<sub>r</sub>) and coercivity (H<sub>c</sub>) values are summarized in Table 2. The magnetic hysteresis (M-H) loops typical for soft magnetic materials confirm the ferromagnetic nature of the NF samples. The results show that the magnetic parameters vary with the change in particle size. In fact the magnetic properties and application fields of the ferrite nanoparticles are highly dependent on the magnetic anisotropy and interparticle interactions [24]. Hence, the lower value of M<sub>s</sub> exhibited by NF–1 can be attributed to the dense aggregation and anisotropy of the powdered sample since the disordered spins present on the surface of the nanoparticles inhibit the alignment of core spin with the field direction resulting in lower M<sub>s</sub> values [25]. An



Fig. 3 (a) IR spectrum of NF-1. (b) IR spectrum of NF-2. (c) IR spectrum of NF-3. (d) IR spectrum of NF-4. (e) IR spectrum of NF-5

increasing trend with the increasing calcination temperature can be seen among NF–2, NF–3 and NF–4 followed by the highest  $M_s$  value for NF–5. Greater values of saturation magnetization are usually associated with the high crystallinity of the nanoparticles at higher calcination temperatures [2]. The remanent magnetization also showed the lowest value for NF–1 reaching a

maximum for NF–5. High coercivity can be observed from the hysteresis loops, which has been reported to be essential for preventing the recorded bits from demagnetization while applying the nanoparticles for data storage or magnetic recording [24]. The results show higher coercivity value for NF–3 sample calcined at 700 °C as compared to the other samples. A decrease



Fig. 4 (a) TGA curve of NF-1. (b) TGA curve of NF-2. (c) TGA curve of NF-3. (d) TGA curve of NF-4. (e) TGA curve of NF-5. (f) TGA curve of NF-intermediate gel

in coercivity at lower calcination temperatures may occur as the particles lose their domain wall boundaries due to the formation of larger aggregates whereas at 700 °C defined grain boundaries are formed and free surface is attained due to reduced aggregation with the rise in calcination temperature resulting in improved magnetic properties. Further increase in the calcination temperature up to 800 °C and 900 °C on the other hand, results in decreased coercivity though particles seem to be separated at these temperatures. It happens due to the fact that the thermal energy for smaller particles becomes sufficient to suppress the entire anisotropy energy which stimulates the reversal of moments leading to the lower critical fields [11, 26] (Table 3).



Fig. 5 (a) FESEM micrograph of NF-1. (b) FESEM micrograph of NF-2. (c) FESEM micrograph of NF-3. (d) FESEM micrograph of NF-4. (e) FESEM micrograph of NF-5

# Conclusion

Nickel ferrite nanoparticles were synthesized by a simple and cost-effective sol-gel method using polypropylene glycol as a stabilizing agent. The effect of various calcination temperatures ranging from 500 °C to 900 °C on structural and magnetic properties of synthesized nanoparticles was studied. The diffraction patterns of ferrite samples proved the formation of single phase face-centered cubic inverse spinel structures with larger grain size and greater crystallinity at higher calcination temperatures. The IR spectra and TGA curves also confirmed the formation of nickel ferrite. FESEM suggested nonhomogeneous structures with aggregated particles having irregular shapes and average grain size in the



Fig. 6 (a) Hysteresis loop of NF-1. (b) Hysteresis loop of NF-2. (c) Hysteresis loop of NF-3. (d) Hysteresis loop of NF-4. (e) Hysteresis loop of NF-5

**Table 2** Magnetic parameters obtained from the results ofhysteresis loops in Fig. 6

Sample	Ms	M <sub>r</sub>	Hc
	(emug <sup>-1</sup> )	(emug <sup>-1</sup> )	(T)
NF-1	5.746	2.231	0.0452
NF-2	7.554	4.030	0.0492
NF-3	7.637	3.137	0.0518
NF-4	7.792	3.991	0.0458
NF-5	9.803	5.038	0.0419

**Table 3** Comparison of coercivity of nickel ferrite nanoparticles

 prepared by different routes

Synthesis method	Average Particle Size (nm)	Нс (T)	Reference
Sol-gel autocombustion	22	0.017700	Gopale et al. [14]
Sol-gel autocombustion	22	0.000197	Bharati et al. [27]
Glycine assisted sol–gel autocombustion	24	0.005967	Bajaj et al. [28]
Urea assisted sol–gel autocombustion	20.87	0.017007	Deshmukh et al. [29]
Urea assisted sol-gel autocombustion	84	0.019260	Mostafa et al. [30]
Polyvinyl alcohol assisted sol-gel autocombustion	30	0.025080	Sivakumar et al. [8]
Glycolic acid assisted sol-gel	35–45	0.009300	Srivastava et al. [31]
Polypropylene glycol assisted sol-gel	10–20	0.051800	Present study

range of 10–20 nm. The results revealed that the use of polypropylene glycol as a stabilizing agent significantly reduced the agglomeration of nickel ferrite nanoparticles. However, loosely aggregated grains were obtained that got separated with the rise in calcination temperature leading to the formation of more prominent and well–dispersed structures at 900 °C. VSM established the ferromagnetic nature of soft magnetic nickel ferrite with a significant effect of particle size on magnetic parameters due to variation in calcination temperature. The samples exhibited high coercivity indispensible for the application of nanoparticles in storage and magnetic devices.

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#### Author contributions

Conceptualization: Samreen Zahra; Methodology: Uzma Naz; Validation: Samreen Zahra; Investigation: Uzma Naz; Resources: Sarwat Zahra; Data Curation: Asma Sheikh; Writing - Original Draft: Samreen Zahra; Writing -Review: All authors reviewed the manuscript; Editing: Samreen Zahra.

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#### Data availability

The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

#### Declarations

**Ethics approval and consent to participate** Not applicable.

#### **Consent for publication**

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#### **Competing interests**

The authors declare no competing interests.

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