SYNTHEIS AND CHARATERIZATION OF NICKEL FERRITE NANOPARTICLES SYNTHESIZED BY SOL-GEL AUTO COMBUSTION METHOD

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ABSRACT

Nickel ferrite nanoparticles were synthesized by sol-gel auto combustion method.XRD petterns expression the existence of a single phase cubic spinel structure.The average grain size estimated from XRD patterns was found to be from 42nm to 43nm VSM study indicates increase in saturation magnetization and decrase in coercivity.FE-SEM images exhibit particles with spherical shape and size ranges from 37 to 46 The two main metal ion vibration of ferrites were observed in FT-IR spetra

Keywords Nickel ferrite sol –gel combustion methodstructure.Magnetic properties

1.INTRODUCTION

Magnetic materials are used in a varicty of applications [1,2] Magnetic spinel ferrite posses best magnetic properties like high magnetocrystalline anisotrophy ,large coercivity and ideal saturation magnetization.They posses exllent structural and chemical stability at expression temperature.substitution of divalent ions nickel ferrite has been made to vary the structural,magnetic and electrical properties [3-6]Though several methods are available to synthesize ferrite ,chemical methods are preferred

due to their simplicity and chemical homogencity Afine particle size is required for uniform sintering and densification which can be obtained easily by chemical methods [7] .Materials synthesized sol-gel auto combustion methods

Have high purity ,chemical homogeneity and uniform particle size .Nickel ferrite ions was synthesized by sol-gel auto combustion method,and their magnetic,structural and morphological features were examined and reported in this work

2. Materials and Methods

Synthesis of nickel ferrite nanoparticles

Nickel ferrite nanoparticles ($NiFe_2O_4$) were synthesized by sol-gel auto combustion method at room temperature. The chemicals used were analytical reagent grade ferric nitrate ($Fe(NO_3)_3.9H_2O$), nickel nitrate ($Ni(NO_3)_2.6H_2O$), citric acid ($C_6H_8O_7.H_2O$) and ammonia (NH_3) solution. Citric acid was employed as the chelating agent. Stoichiometric ratio of nitrates/chelating agent is 1:2. Nitrates and citric acid were dissolved in de-ionized water. This solution was kept in continuous stirring for 24 hrs at 60 °C. The solution become dehydrated and transform into gel. This gel was heat treated in the hot air oven at 250 °C for 8 hrs. This leads to the formation of a dark loose powder. The powder was heated at a rate of 5 °C/min in a muffle furnace and kept at 800 °C for 4 hrs. Finally it was grained finely using mortar and pestle for further analysis. The following instruments were used to synthesis of $NiFe_2O_4$ nanoparticles.

S. No.	Instruments	Specifications
1.	Beakers	- 500 ml borosilicate
2.	Magnetic stirrers with hot plate	- Stirrer speed 0 to 1000 rpm Room Temperature to 100 °C
3.	Digital weight balance	- Accuracy 0.001 g
4.	Digital pH meter (with reference electrode)	- Room temperature to 100 °C
5.	Hot air oven	- RT to 300 °C
6.	High temperature muffle furnace	- RT to 1050 °C Accuracy ± 1 °C

Fig. 20: Synthesis part of $NiFe_2O_4$ nanoparticles

3.Result and Discussion

3.1 Powder X-ray diffraction (XRD) analysis

Structural and phase analysis of nickel ferrite samples were investigated by powder XRD patterns obtained from X'Pert-PRO Pan Analytical X-ray diffractometer operated at 45 kV and 30 mA, Cu Kα, wavelength 1.5406 Å. These are shown in Fig. 21. These patterns confirms the presence of a single phase cubic spinel structure. The prominent hkl planes (220), (311), (222), (400), (422), (511) and (440) are identified and indexed. The results are in good agreement with literature values [18-20].

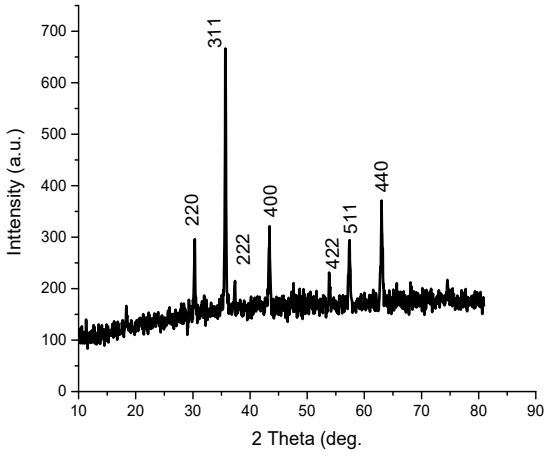

Fig. 22: XRD pattern of $NiFe_2O_4$ nanoparticles

The lattice parameters were calculated using the formula,

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

where, "d" is inter-planar distance, "h, k and l" are the miller indices [24]. The determined lattice constant values are given in table1. With the help of Scherer equation

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

The average crystallite size (D) was calculated. Here, λ is wavelength of the X-ray radiation used, β is full width at half maximum (FWHM) measured in radians and θ is Bragg angle. The crystallite sizes were found to vary from 42 nm.

X-ray density (ρ_x) of nickel ferrite samples were calculated using,

$$\rho_x = \frac{8M}{Na^3}$$

where, 8 denotes the number of atoms in a unit cell of spinel lattice, M is molecular mass of the articular ferrite samples, N is the Avogadro's number (6.02252×10^{26} $kmol^{-1}$) and 'a' is the lattice constant. The calculated values of X-ray density is 5.415 g/cm^3 for nickel ferrite samples.

Table 3: structural and magnetic properties of Nickel ferrite nanoparticles

Properties		Nickel ferrite
Lattice constant (a) (Å)		8.346
Volume (V) (Å^3)		581.34
Crystallite size (nm)	XRD data	42.43
	FE-SEM image	37-46
X-ray density (g/cm^3)		5.415
Saturation magnetization (Ms) (emu/g)		25.37
Coercivity (Hc) (Oe)		198
Remanence magnetization (Mr) (emu/g)		7.6
Bohr magneton μ_B		1.06

3.2 Vibrating Sample Magnetometer (VSM) analysis

Magnetization measurements of nickel ferrite samples were done using vibrating sample magnetometer (VSM-Lake Shore model 7404, operated at a maximum applied field of 12.5 kOe at room temperature). The observed hysteresis (M-H) curves are presented in Figs. 22 and 23. From the plotted M-H curves, the saturation magnetization (Ms), coercivity (Hc) and remanent magnetization (Mr) values are measured.

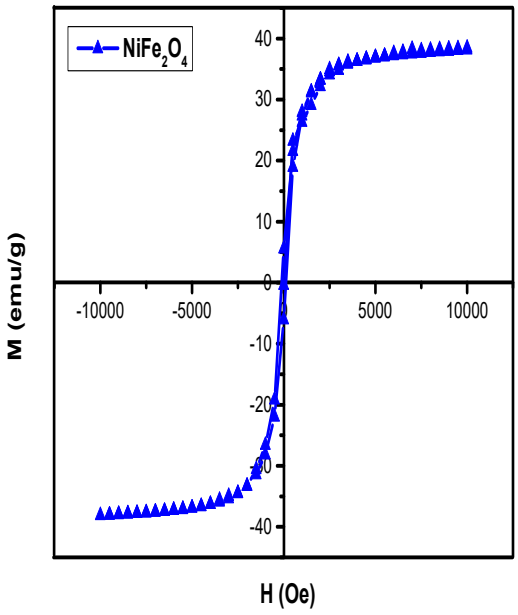

Fig. 23: M-H plots of $NiFe_2O_4$ nanoparticles

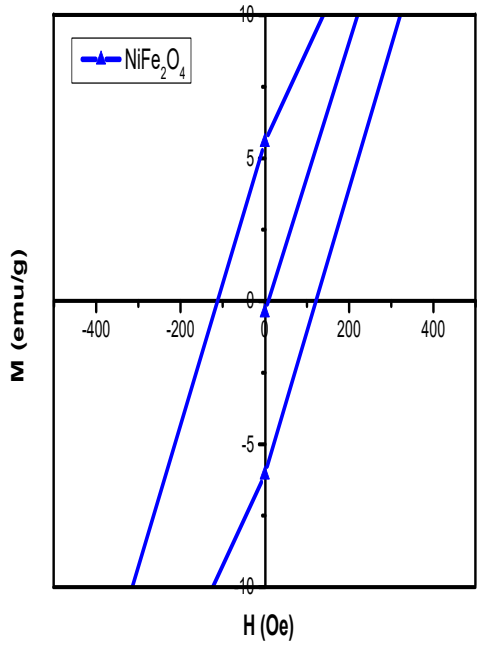

Fig. 24: M-H enlarged plot of $NiFe_2O_4$ nanoparticles

The experimental magnetic moment per formula unit in Bohr magneton (μ_B) was calculated from the saturation magnetization values using the equation,

$$\mu_B = \frac{M_S M_W}{5585}$$

where, M_S is saturation magnetization, M_W is molecular weight of the sample and 5585 is magnetic factor. The observed values are listed in Table 3. The M-H curves revealed that the magnetic properties of the samples are affected by composition and different cation distribution. Various cations can be occupied in tetrahedral sites and octahedral sites to change the magnetic properties [25]. All the hysteresis curves indicate soft ferromagnetic nature of the samples. The saturation magnetization value of nickel ferrite sample is 25.37 emu/g with maximum applied field 12.5 kOe at room temperature. Which is agree with results reported by Mahmoud Goodarz et al [21].

3.3 Field Emission Scanning Electron Microscopy (FESEM) analysis

The morphological features of nickel ferrite samples were analyzed by FE-SEM analysis (Model-JEOL/JSM-5610 NE instrument) and are shown in Fig. 24. The grain size of the heat treated samples are found to be from 37 to 46 nm for nickel ferrite. FE-SEM images revealed the spherical nature of the particles. Observed particle size closely matches with the values obtained from XRD measurement.

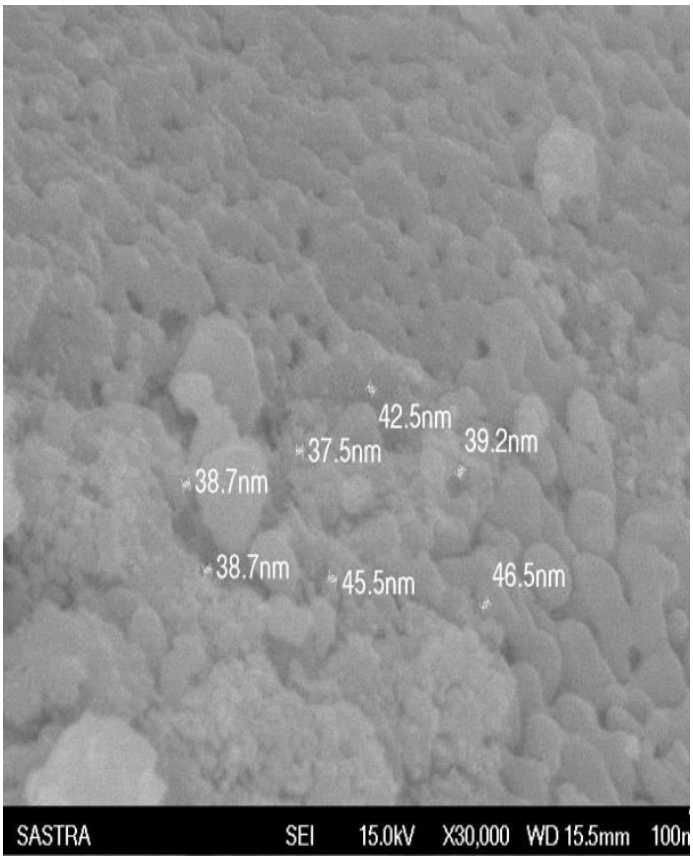

Fig. 25: FE-SEM image of $NiFe_2O_4$ nanoparticles

3.4 FT-IR spectral study

FT-IR spectra of nickel ferrite samples were recorded using Perkin Elmer FT-IR spectrometer in the range 4000 to 400 cm^{-1} and are shown in Fig.25. The unit cell of nickel ferrite contains 8 molecules. There are 32 divalent oxygen ions, 16 trivalent iron ions and 8 divalent nickel ions in the unit cell. 32 oxygen atoms arrange themselves in fcc structure and this leads to 8 tetrahedral voids (A-sites) and 16 octahedral voids (B-sites). The nickel ions occupy half of the B-sites. The remaining B-sites and A-sites are occupied by iron ions. Ni^{2+} ion is expected to occupy B-sites. This divalent metal ion may also occupy A-sites and B-sites which results in the movement of iron ions from A-sites to B-sites [22].

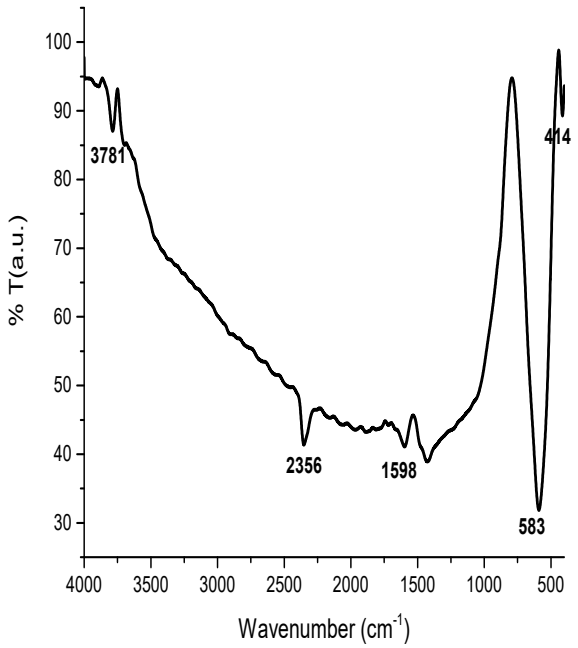

Fig. 26: FT-IR spectrum of $NiFe_2O_4$ nanoparticles

The two main vibrations of tetrahedral and octahedral metal-oxygen bonds will be observed in the range of 800-400 cm^{-1}. The band observed

around 583-598 cm^{-1} and 414-421 cm^{-1} is due to tetrahedral sites and octahedral sites respectively. In $NiFe_2O_4$, the tetrahedral M-O vibration occurs at 583 cm^{-1} and the octahedral M-O vibration of $NiFe_2O_4$ occurs at 414 cm^{-1}. The occurrence of two metal-oxygen vibrations at two different wavenumbers is due to the change in metal-oxygen bond length for tetrahedral and octahedral sites. The observed absorption bands closely matches with earlier literature values [26, 27].

CONCLUSIONS

- Nickel ferrite nanoparticles were synthesized at room temperature by sol-gel auto combustion method.
- They synthesized sample crystallize in cubic spinel phase.
- The lattice parameter and average crystallite size values are calculated.
- The saturation magnetization (Ms), coercivity (Hc) and remanence magnetization (Mr) values are calculated.
- FE-SEM image shows the synthesized nickel ferrite nanoparticles are spherical in shape and size of the particles was in the range 37-46 nm, which matches with XRD pattern.
- Presence of two main metal ion vibrations of tetrahedral (A) and octahedral (B) sites were observed in FT-IR spectra.

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