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# SYNTHESIS AND CHARACTERIZATION OF NICKEL CALCIUM FERRITE NANOPARTICLES

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## ABSTRACT

Ni-Ca ferrite particles were prepared by co-precipitation method to study its magnetic properties for use in biological applications.XRD pattern obtained has given crystallite size of 14.85, 12.05, 11.74 and 22.72 nm with respective lattice parameter of 8.3618, 8.3495, 8.3811and 8.4632 $A^0$ for $NiFe_2O_4$ ferrite calcined at 700 $^0$C and $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite calcined at 500,700 and 800 $^0$C respectively.XRD of Ni-Ca ferrite showed additional orthorhombic $CaFe_2O_4$phase alongwith the cubic spinel phase at 700 and 800 $^0$C. Particle size measured for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite calcined at 800 $^0$C by TEM and FEG-SEM has given particle size of 20.6 – 26.5 nm and 20.4 -43.9 nm respectively which matches with the crystallite size 22.77nm given by XRD measurement. FTIR spectra measured for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite calcined at 800 $^0$C showed frequency band at 586.60 $cm^{-1}$and at 469.60$cm^{-1}$corresponds to the stretching vibrations of metal –Oxygen (Me-O) bond at tetrahedral and octahedral sites respectively. Presence of these peaks confirms the spinel phase for the ferrite nanoparticles. At 500 $^0$C $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite has exhibited saturation magnetization of 35.92emu/g with coercivity of 41G. At 800 $^0$C same ferrite has showed magnetization of 14.83 emu/g with coercivity of 86G.The Curie temperature ($T_c$) for the same composition was observed above the room temperature.

**Keywords**: saturation magnetization, coercivity, nanoparticles, crystallite size, particle size

## I. INTRODUCTION

Magnetic nanoparticles are nanomaterials consist of magnetic elements such as iron,nickel,cobalt,chromium,manganense,gadolinium and their chemical compounds. Especially ferrite nanoparticles are the most explored magnetic nanoparticles. Spinel ferrite nanoparticles with the formula $MFe_2O_4$ (M = Cu,Mn,Mg,Zn,Ni,Co and other metals) are commonly used in various medical applications such as magnetic resonance imaging (MRI ) constrast agents,as carrier for targeted drug delivery,in the hyperthermia as heat source and as a magnetic separation agents due to their properties. Magnetic nanoparticles show superparamagnetism, have high surface area, nanometric size and ability to produce heat within an alternating magnetic field. In these applications magnetic particles should have higher saturation magnetization with lower coercivity. In this context different magnetic nanoparticles were searched. From the literature survey it was observed that Ni-Ca ferrite particles showed high magnetization , lower coercivity and the Curie temperature in the room temperature range required in hyperthermia[1-2]. Calcium substituted nickel ferrite particles are less studied as seen from the literature. Hence in the present paper calcium substituted nickel ferrite particles were studied. To decrease the Curie temperature($T_C$) of nickel ferrite (which was reported to be 587 $^0$C) calcium concentration, Ca = 0.7 was selected. $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite composition was synthesized by co-precipitation method. To observe the effect of calcining temperature on magnetic properties of the ferrite particles, they were calcined at different temperatures.

## II. METHODOLOGY

### 2.1 Experimental Procedure

$Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite particles were synthesized by co-precipitation of aqueous solutions of Iron (III) Chloride ($FeCl_3$)anhydrous, Nickel Chloride hexahydrate ($NiCl_2.6H_2O$), Calcium Nitrate tetra hydrate $Ca(NO_3)_2.4H_2O$. Initial molar proportions ([Ni]+ [Ca])/ [Fe] was taken as stoichiometric ½. 0.3 mole of Nickel chloride, 0.7 mole

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of Calcium Nitrate, 2 mole of ferric chloride were measured and were dissolved in 12.5 ml distilled water. The solution containing these ions were mixed and added to sodium hydroxide(NaOH) solution at a pH of 12 at the temperature nearly equal to 90 $^0$C. The obtained precipitate was washed till its pH became neutral after that it was dried. The particles were then calcined at different temperatures.

### 2.2 XRD Measurements

XRD pattern for nickel ferrite and Ni-Ca ferrite calcined at 500 and 700 $^0$C was obtained using XPERT-PRO diffractometer with CuKαradiation (λ= 1.5406 A$^0$), (Step size 0.020 $^0$, measurement time 65.6 s at temperature of 25$^0$C). XRD pattern for Ni-Ca ferrite calcined at 800 $^0$C was obtained by Bruker AXS D8 Advance Diffractometer.

### 2.3 Structural Measurements

Size of Ni-Ca ferrite particles calcined at 800 $^0$C was estimated by Field Electron Gun Scanning Electron Microscope (FEG SEM) JSM-7600 F model and Transmission Electron Microscope (TEM) Hitachi H7500 model. The Energy Dispersive spectra (EDS) for the same ferrite was obtained using JEOL Model JED-2300.

### 2.4 FTIR Measurements

Fourier Transform Infrared (FTIR) Spectroscopy measurements of Ni-Ca ferrite particles calcined at 700 $^0$C was carried with Thermo Nicolet, Avatar 370 model. (range 400-4000 cm$^{-1}$).The dried sample was in KBr matrix, and spectra were measured according to transmission method and were resolved with a resolution of 0.5 cm$^{-1}$.

### 2.5 Magnetic Measurements

Magnetization versus field curves (M-H) for Ni-Ca ferrite particles calcined at 500 and 800 $^0$C was measured using Lakeshore VSM 7410 model. FC-ZFC curve for the ferrite calcined at 800 $^0$C was measured using quantum design SQUID USA MPMS model.

## III. RESULTS AND DISCUSSION

### 3.1 XRD Measurements

Figure – 1 (a) (b), (c) and (d) shows the XRD pattern obtained for $NiFe_2O_4$ ferrite particles calcined at 700 $^0$C and $Ni_{0.3}Ca_{0.7}Fe_2O_4$ferrite particles calcined at 500,700 and 800$^0$C respectively. XRD pattern shows the presence of prominent XRD peaks corresponding to the face centered cubic spinel structure with space group fd3m (#JCPDS -01-080-007).Taking full width at half maximum (FWHM) of the most intense peak (311) and using the Scherrer equation, the average crystallite size for the particles was calculated. The calculated crystallite size of 14.85, 12.05, 11.74 and 22.72 nm with respective lattice parameter of 8.3618, 8.3495, 8.3811and 8.4632 A$^0$was obtained for the nickel ferrite calcined at 700 $^0$C and $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles calcined at 500,700 and 800 $^0$C respectively(Table – 1). With substitution of Ni ions by Ca ions, the crystallite size was reduced from 14.85 nm for $NiFe_2O_4$ to 11.74nm for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ both calcined at 700$^0$C (Table-1). The lattice parameter was increased from 8.3618 to 8.3811 A$^0$. With addition of Ca ions in $NiFe_2O_4$ the 2θ value has shifted to a lower value which indicates increase in the lattice parameter of the ferrite particles. The increase in the lattice parameter with addition of Ca ions indicates accumulation of the Ca ions at preferential B site of the cubic Ni ferrite . With addition of Ca ions in Ni ferrite, the crystallite size was decreased from 14.85 to 12.5nm.Ca ions are supposed to obstruct the crystal growth [3]. At 500 $^0$C the crystallite size of 12.05 nm with lattice constant of 8.3495 A$^0$ was obtained for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite. At 700$^0$C the crystallite size was observed decreased slightly to 11.74 nm compared to 500 $^0$C ferrite particles. The peak width has been increased compared to 500 $^0$C ferrite particles indicating decrease in the crystallite size for the 700 $^0$C ferrite particles. The lattice parameter was found increased at 700 $^0$C. For $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite composition calcined at 800 $^0$C, the crystallite size and lattice parameter both were increased. The peak width has decreased compared to that of 700 $^0$C ferrite nanoparticles indicates increase in the crystallite size. The lattice parameter has increased due to increase in volume of the particles with the calcining temperature. With increase in the calcining temperature, the cell constant was increased continuously for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite composition. At 700 and 800 $^0$C some additional XRD peaks corresponding to the $CaFe_2O_4$ orthorhombic phase have appeared along

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with the cubic spinel phase (Figure-1 c and d).The XRD peaks corresponding to the $CaFe_2O_4$ orthorhombic phase were indexed[4]. These peaks were observed due to completion of $CaFe_2O_4$ phase at higher calcining temperature >600 $^0$C[5]. Calculated values of the X-ray densities for Ni-Ca ferrite particles are given in Table 1.The observed and the calculated interplanar spacing values obtained for Ni-Ca ferrite particles are tabulated in Table 2.

### 3.2 Microstructural Analysis

Particle morphology of Ni-Ca ferrite particles was studied using TEM and FEG-SEM measurements. FEG-SEM image obtained for $Ni_{0.3}Ca_{0.7}$-$Fe_2O_4$ ferrite particles calcined at 800 $^0$C(Figure -2) shows the uniform particle size distribution. The particles are seen spherical and slightly aggregated Particles are in the range of 20.4-43.9 nm which agrees with the crystallite size given by the XRD pattern 22.72nm (Table-3).

Figure-3 shows TEM micrograph for $Ni_{0.3}Ca_{0.7}Fe_2O_4$composition calcined at 800$^0$C.The micrograph gives spherical ferrite particles with aggregations formed between them which is due to the dipolar magnetic interactions among the particles. Particles are 20.4 - 26.5 nm in size. The crystallite size given by XRD pattern (22.72nm) agrees with the particle size shown by TEM image.

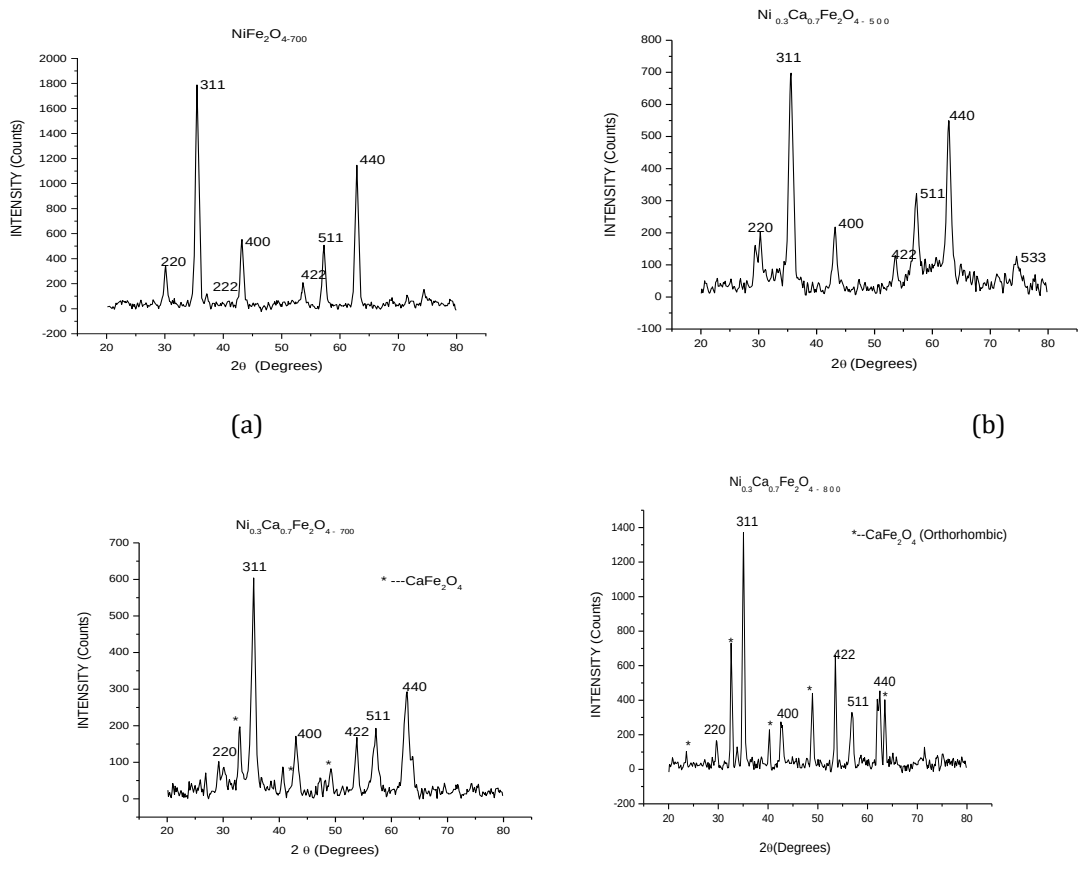


**Figure 1:** - XRD pattern obtained for a) $NiFe_2O_4$ferrite calcined at 700 $^0$C and $Ni_{0.3}Ca_{0.7}Fe_2O_4$ferrite particles calcined at (b) 500, (c) 700 and (d) 800 $^0$C

Table 4 gives the values of mass% and atomic % of the constituent elements for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles calcined at 800 $^0$C.EDS spectra (Figure4) for the sample shows the presence of O, Fe, Ca and Ni elements inside the sample. The compositional molar ratio of Me ([Me] = ([Ni] + [Ca]) to [Fe] was found < 0.5 shows that $Ni_{0.3}Ca_{0.7}Fe_2O_4$ferrite was not stoichiometric.This is because of the appearance of $CaFe_2O_4$ phase along with cubic spinel phase at 800 $^0$C inside the sample as shown by the XRD.

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### 3.3 Spectral Measurements

Figure-5 shows FTIR spectra recorded for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles calcined at 800 $^0$C. The spectra exhibits frequency band at 586.60 $cm^{-1}$and at 469.60$cm^{-1}$corresponds to the stretching vibrations of metal – Oxygen (Me-O) bond at tetrahedral and octahedral sites respectively. Presence of these peaks confirms the spinel phase for the ferrite nanoparticles

**Table 1.**Structural properties of Ni-Ca ferrite particles obtained from XRD pattern (suffix denotes the calcining temperature)

| Nameof ompound | crystallitesize nm)XRD | Lattice parameter ($A^0$) | X-Ray density (g/cc) | Volume (cc) |
|---|---|---|---|---|
| $NiFe_2O_{4\_700}$ | 14.85 | 8.3618 | 5.347 | 584.38 |
| $Ni_{0.3}Ca_{0.7}Fe_2O_{4\_500}$ | 12.05 | 8.3495 | 5.070 | 582.07 |
| $Ni_{0.3}Ca_{0.7}Fe_2O_{4\_700}$ | 11.74 | 8.3811 | 5.012 | 588.71 |
| $Ni_{0.3}Ca_{0.7}Fe_2O_{4\_800}$ | 22.72 | 8.4632 | 4.846 | 608.89 |

**Table 2.** Comparison between some of the observed and calculated d values for Ni -Ca Ferrite system for cubic phase

| | $Ni_{0.3}Ca_{0.7}Fe_2O_{4\ \_500}$ | | | $Ni_{0.3}Ca_{0.7}Fe_2O_{4\ \_800}$ | | |
|---|---|---|---|---|---|---|
| **hkl** | **2θ** | **$d_{obs}$** | **$d_{hkl}$** | **2θ** | **$d_{obs}$** | **$d_{hkl}$** |
| 220 | 30.214 | 2.9556 | 2.9558 | 29.660 | 3.0095 | 3.0136 |
| 311 | 35.559 | 2.5226 | 2.5245 | 35.056 | 2.5576 | 2.5581 |
| 400 | 43.179 | 2.0934 | 2.0952 | 42.686 | 2.1164 | 2.1182 |
| 511 | 53.634 | 1.7074 | 1.7073 | 53.546 | 1.7100 | 1.7111 |
| 440 | 57.212 | 1.4776 | 1.4770 | 56.899 | 1.6169 | 1.6176 |

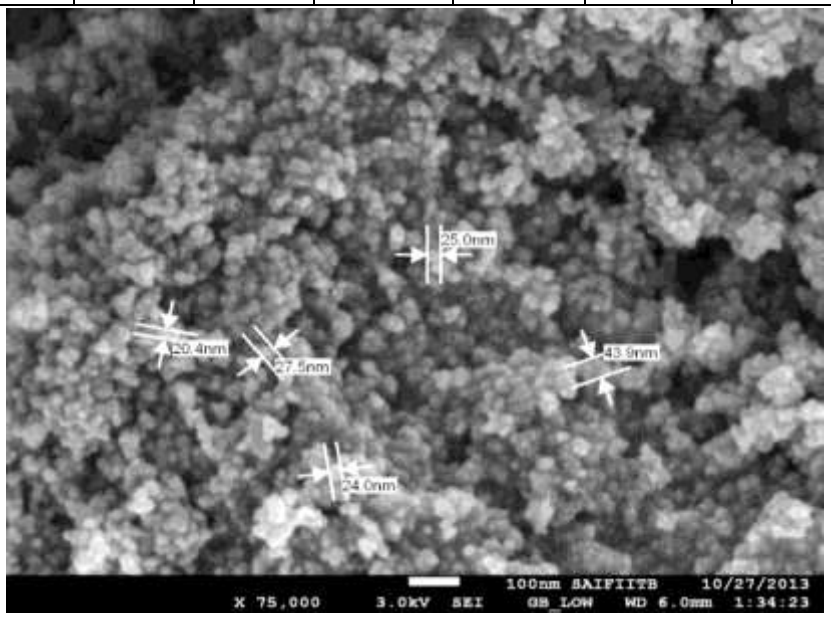


**Figure2:** Field Electron Gun Scanning Electron Microscopy image obtained for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles.

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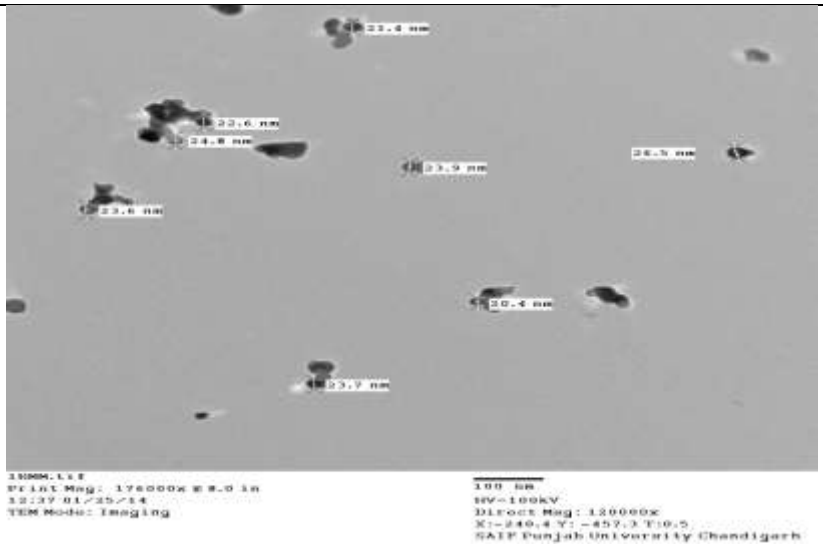

**Figure:3** Transmission Electron Microscopy image obtained for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles.

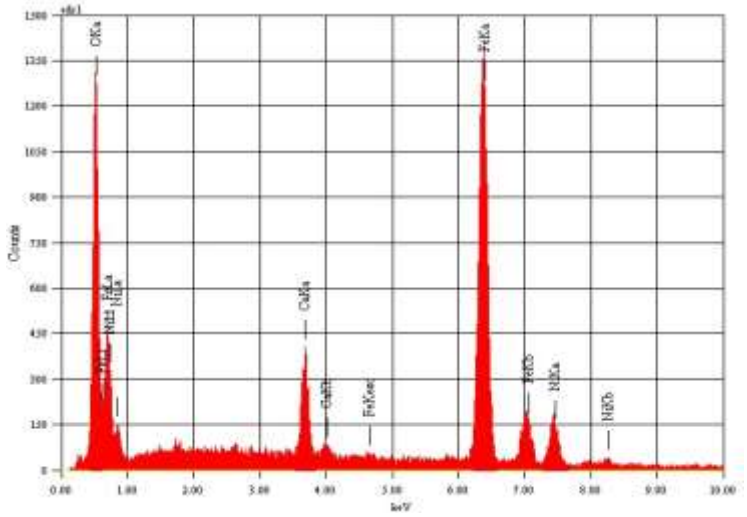


**Figure: 4** Energy Dispersive spectra (EDS) obtained for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles

**Table 3.** Structural Properties of Ni-Ca ferrite particles

| Name of Compound | Crystallite ize(nm)(XRD) | Particle size(nm) (FEG-SEM) | Particlesize nm)(TEM) |
|---|---|---|---|
| $Ni_{0.3}Ca_{0.7}Fe_2O_{4\text{-}800}$ | 22.73 | 20.4-43.9 | 20.6-26.5 |

**Table 4.** Elemental analysis for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles

| Element | (keV) | Mass% | Atom% | K |
|---|---|---|---|---|
| O K | 0.525 | 17.36 | 41.67 | 0.4247 |
| Ca K | 3.69 | 6.71 | 6.43 | 0.5089 |
| Fe K | 6.398 | 67.05 | 46.1 | 1 |
| Ni K | 7.471 | 8.87 | 5.8 | 1.2976 |
| Total | | 100 | 100 | |

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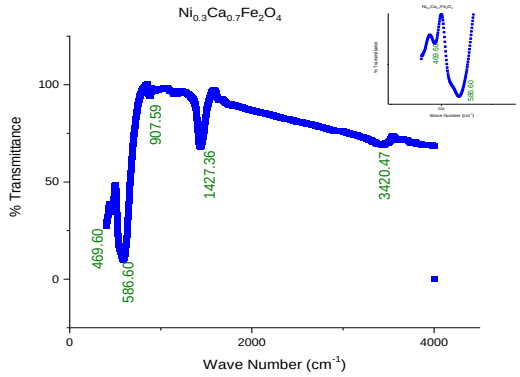


**Figure:5** Fourier Transform Infrared spectra obtained for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles

Additional frequency peaks appeared in FTIR spectra in case of our ferrite nano-particles shown in Figure-5 corresponds to the stretching vibrations of hydroxyl (OH) group that shows the presence of the water content adsorbed on the ferrite particle surface.

### 3.4 Magnetic measurements

To study the magnetic properties of $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles its magnetization versus field (M-H) curves were measured at room temperature. Figure 6- a gives M-H curve for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles calcined at 500 $^0$C. The curve has displayed saturation magnetization ($M_s$) of 35.92 emu/g with coercivity ($H_c$) of 41G having remanence of 1.49emu/g. Same composition calcined at 800 $^0$C has given $M_s$ of 14.83 emu/g with coercivity of 86 G having remanence of 1.50 emu/g(Figure 6-b) (Table-5). These observations shows that magnetization of Ni-Ca ferrite particles is decreased and the coercivity is increased with increase in calcining temperature. This was due to appearance of additional paramagnetic

**Table 5.** Magnetic parameters obtained from M-H curves for Ni-Ca ferrite nanoparticles

| Name of Compound | Saturation Magnetization $M_s$(emu/g) | Coercivity $H_c$ (G) | Anisotropy constant K(erg/G) | Remanence ($H_r$) (emu/g) | Remanence ratio ($M_r/M_s$) |
|---|---|---|---|---|---|
| $Ni_{0.3}Ca_{0.7}Fe_2O_{4\ \_500}$ | 35.92 | 41 | 1502.77 | 1.49 | 0.041 |
| $Ni_{0.3}Ca_{0.7}Fe_2O_{4\ \_800}$ | 14.83 | 86 | 1301.40 | 1.50 | 0.101 |

$CaFe_2O_4$ phase appeared at 800 $^0$C (Figure 1 d).At lower calcining temperature higher magnetization is displayed by the ferrite particles as at low calcining temperature orthorhombic $CaFe_2O_4$ phase is amorphous and does not affect the magnetic properties [5] same observations were obtained by Remirez et al in case of $Mg_{0.4}Ca_{0.6}Fe_2O_4$ ferrite[6]. For $Mg_{0.4}Ca_{0.6}Fe_2O_4$ ferrite the Ms values treated at 350 and 600 $^0$C were 23.1 and 15.1 emu/g and $H_c$ values were 5.7 and 14 Oe respectively. Ni-Ca ferrite nanoparticles with small coercivity were observed ferrimagnetic at room temperature. The saturation magnetization obtained for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles at 500 $^0$C was 35.92 emu/g .This value was less than the value obtained by Prasad et al [2] for $Ni_{0.2}Ca_{0.8}Gd_{\ 0.08}Fe_{1.92}O_4$ ferrite which is 55 emu/g. This shows that magnetization of the Ni-Ca ferrite particles increase with increase in the Ca ions. Nickel ferrite has a mixed spinel structure in which $Ni^{2+}$ions ( ionic radius,0.72A$^0$) having magnetic moment of 2μB (where μB is Bohr magneton) occupy both A and B sites. $NiFe_2O_4$ obeys cation distribution $(Ni_{1-x}\ Fe_x)^A[Ni_xFe_{2-x}]^BO_4$ ,where x is the inversion factor which indicates the fraction of $Fe^{3+}$ ions accumulated at A sites[7-8]. The super exchange A-B interactions between $Fe^{3+}$and $Ni^{2+}$ ions at both sites gives the net magnetization to the Ni ferrite particles. Incoming nonmagnetic $Ca^{2+}$ions (ionic radius,0.99A$^0$) prefer to occupy B site due to large ionic radius. Addition of $Ca^{2+}$ions causes redistribution of the cations at two sub lattice sites which gives the net magnetization to the sample.

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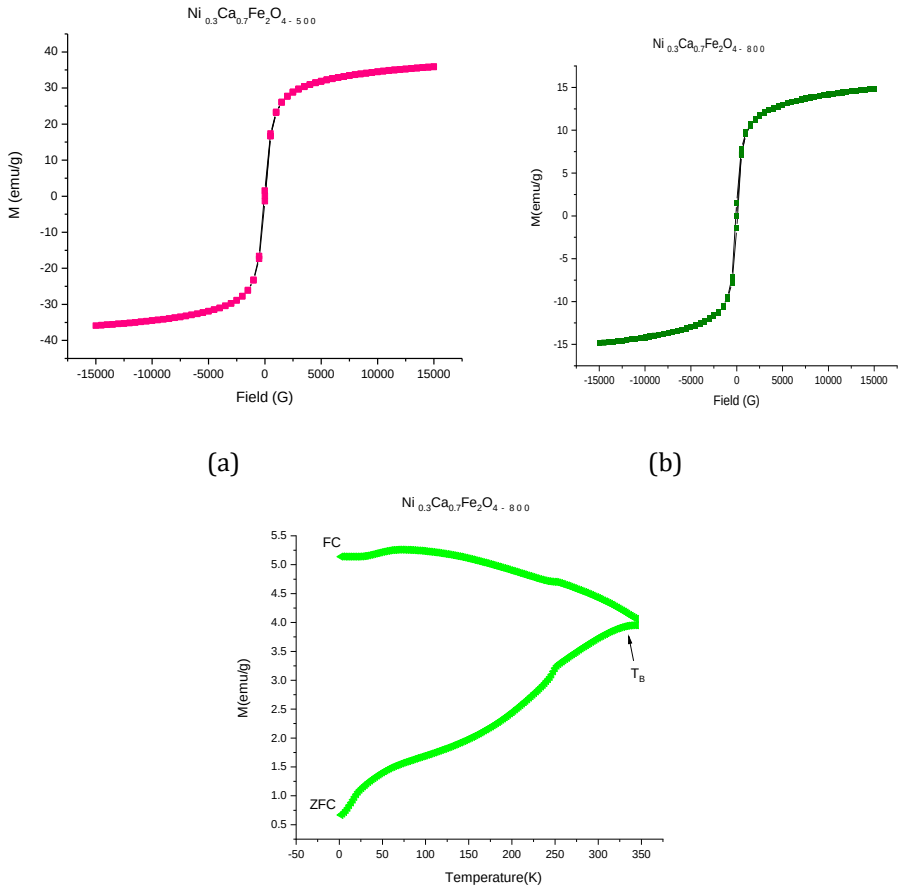


**Figure:6** Magnetization vs. field (M-H) curves obtained for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles calcined at (a) 500 $^0$C(b) 800 $^0$C,(C) Field cooled - Zero Field cooled(FC-ZFC) curve obtained for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite nanoparticles. Calculated values of anisotropy constant K (calculated using Hc = 0.98 K/$M_s$) are given in Table-5. K values are proportional to the coercivity and remanence. As increase in thermal energy with temperature overcomes the anisotropy energy barrier of the particles.

Figure 6 - c shows the FC-ZFC curve measured for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite particles (calcined at 800 $^0$C) at 100 Oe .In the ZFC mode the sample was cooled in the zero field mode from 350 K to 6 K and after stabilization of the temperature a measuring field of 100 Oe was applied. The blocking temperature ($T_B$) for the assembly of nanoparticles has obtained near 334.30 K. It is not possible to find the Curie temperature for $Ni_{0.3}Ca_{0.7}Fe_2O_4$ composition from obtained FC-ZFC curve. Prasad et al has obtained $T_c$ of 50$^0$C for $Ni_{0.2}Ca_{0.8}Gd_{0.08}Fe_{1.92}O_4$ ferrite. In case of our $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite the Ca concentration ,Ca = 0.7 is not able to decrease the curie temperature of $NiFe_2O_4$ in the room temperature range.

## IV. CONCLUSION

$Ni_{0.3}Ca_{0.7}Fe_2O_4$ composition was synthesized by the co precipitation route and calcined at different temperatures. At lower calcining temperature 500 $^0$C, the ferrite particles showed single spinel cubic phase whereas at higher temperature (> 600 $^0$C) the ferrite particles have displayed orthorhombic $CaFe_2O_4$ phase along with the cubic spinel phase. $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite has shown saturation magnetization of 35.92 and 14.83 emu/g with coercivity of 41 G and 86 G at respective calcining temperatures of 500 and 800 $^0$C. $Ni_{0.3}Ca_{0.7}Fe_2O_4$ ferrite with obtained magnetic properties might be useful in hyperthermia, in targeted drug delivery or as magnetic resonance imaging contrast agent.

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