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AN INVESTIGATION OF STRUCTURAL AND MAGNETIC PROPERTIES OF TRANSITION METALS DOPED MANGANESE FERRITE NANOPARTICLES

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Abstract: **This paper elaborates the synthesis of manganese ferrite nanoparticles and transition metals (Cobalt, Nickel, Cadmium) doped manganese ferrite nanoparticles via co-precipitation technique. The structural and magnetic properties of the obtained nanoferrites are investigated. X-Ray Diffraction (XRD) results substantiated the cubic spinel structure of prepared nanoferrites. Fourier Transform Infrared Spectroscopy (FTIR) is employed for functional group identification. The characteristic frequency of ferrites is found to be 550cm^{-1} - 450cm^{-1}. The results of Energy Dispersive X –ray Spectroscopy (EDX) confirmed the presence of used materials without any impurities. Magnetic parameters are measured by Vibrating sample magnetometer (VSM). All the measured magnetic parameters showed an increasing trend with the content of doping.**

Keywords: **Nanoferrites, Co-precipitation technique,Cubic Spinel and Transition metals.**

I. INTRODUCTION

Ferrites are a large class of oxides with remarkable magnetic properties, which have been investigated and applied during the last 50 years. The possibility of preparing ferrites in the form of nanoparticles has opened a new and exciting research field, with revolutionary applications not only in the field of electronic technology but also in the field of biotechnology [1]. Among various nanoferrites, manganese nanoferrites are gaining much attention because of their magnetocrystalline anisotropy and high saturation magnetization. Furthermore the magnetic nanoparticles are bio-compatible materials [2]. The doping of ferrite has attracted attention because of the possible use of materials in magnetic memories and heterogeneous catalysts etc. Normally the doping of metal affects the physical and chemical properties of the nanomaterials. One can tune the properties of the nanoparticles by the addition of appropriate dopant. For example grain size can be reduced; magnetic property can be enhanced and so on. In this present work transition metals namely, cobalt, nickel and cadmium are substituted into manganese ferrite nanoparticles.

II. MATERIALS AND METHOD

Manganese chloride and ferric chloride are precursors. Cobalt chloride, nickel chloride and cadmium chloride are used as dopants. Aqueous ammonia solution act as a precipitating agent and water is a solvent throughout the entire experimental work. There are so many methods are available to synthesize ferrite nanoparticles such as hydrothermal method, sol-gel method, combustion method, citrate method. Among the various routes co-precipitation method yields more promising results in the synthesis of ferrite nanoparticles [3].

0.1M of manganese chloride and 0.2M of ferric chloride are dissolved in 100ml distilled water. The mixed solution is kept at magnetic stirrer under heating up to 70°C. At 70°C, 10ml of ammonia solution is added in a dropwise manner. The solution is stirred continuously for 2hours at the same temperature. After that the solution is cooled down to room temperature. Then the solution is filtered using whatmann no.1 filter paper. The obtained precipitate is dried at 100°C for

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2hours. Then the dried powder is grained finely by mortor and pestle. Further the powder is calcinated at 800°C for 2hours using muffle furnace. The resultant powder is called as manganese ferrite nanoparticles. Similar procedure is followed for the preparation of cobalt-nickel doped manganese ferrite nanoparticles and cobalt-nickel-cadmium doped manganese ferrite nanoparticles.

III. RESULT AND DISCUSSION

XRD Analysis:

XRD is mainly used to determine the crystal structure [4]. Also one can find the crystallite size, micro strain, lattice parameter etc. Fig 1 depicts the XRD patterns for the prepared samples. The obtained data is very much matched with JCPDS card no 742403. The peaks can be indexed as (311), (222), (422), (333), (531) planes. The obtained nanoferrites are in cubic spinel structure. The crystallite sizes are calculated using Debye Scherrer formula,

$$D = K\lambda/\beta\cos\theta$$

Where K is Scherrer constant (0.9), λ is wavelength of X-rays(1.54Å), β is full width at half maximum and θ is diffraction angle in degree.The calculated average crystallite sizes are in the range of 40-50nm. Also dislocation density and micro strain are calculated by the formulae mentioned below,

$$\delta = 1/D^2$$

Where, δ is the dislocation density and D is the crystallite size.

$$\epsilon = \beta \cos\theta/4$$

Where, ϵ is micro strain, β is full width at half maximum and θ is diffraction angle. The calculated parameters are presented in table1.

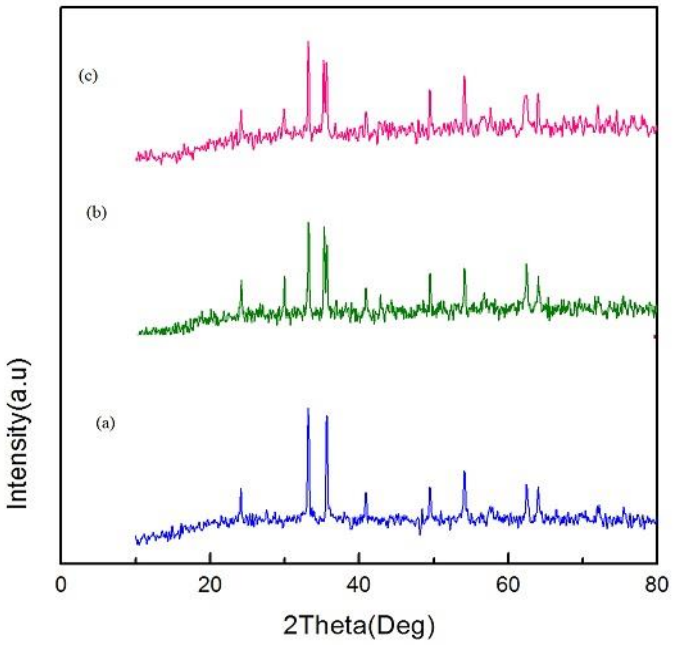

Fig 1: XRD patterns of the synthesized samples a) $MnFe_2O_4$NPs, b) Co-Ni-$MnFe_2O_4$NPs, c) Co-Ni-Cd-$MnFe_2O_4$NPs

Table 1: Calculated parameters

Samples	Avg D (nm)	Avg δ (nm^{-2})	Avg strain
$MnFe_2O_4$	47	6.04×10^{14}	0.0008
$CoNiMnFe_2O_4$	50	5.40×10^{14}	0.0007
$CoNiCdMn\ Fe_2O_4$	40	2.71×10^{15}	0.0012

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FTIR Analysis:

FTIR spectroscopy is employed to identify the functional groups present in the prepared samples. FTIR characterization is carried out at room temperature in the wave number range of 4000 to 400cm^{-1}. Figure 2 represents FTIR spectra for the synthesized nanoferrites.

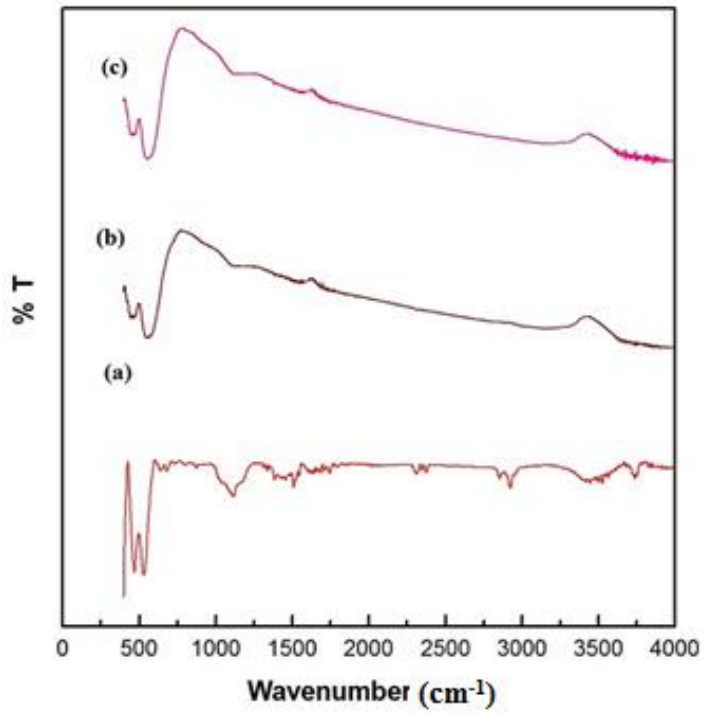

Fig 2: FTIR spectra for a) $MnFe_2O_4$ NPs, b) Co-Ni-$MnFe_2O_4$ NPs, c) Co-Ni-Cd-$MnFe_2O_4$ NPs

In all ferrites two prominent peaks are observed. They are the bands around 550cm^{-1} and 450cm^{-1}. These two are characteristic freuqency of all the ferrites attributed to the intrinsic stretching vibrations of tetrahedral sites (A sites) and octahedral sites (B sites) respectively [8]. Some less intensed extra peaks were observed. The peaks around 3200-3800cm^{-1} ascribed to OH stretching and the bands around 1600 -1700cm^{-1} may be attributed to OH bending [5]. Eventhough the synthesized nanoferrites dried well, water molecules are present in the samples. So it is concluded that the drying process should be extented further. The intensed vibration bands presented in the following table2

Table 2: Intensed vibration bands

Samples	**U_1 (Stretching vibrations by A sites) cm^{-1}**	**U_2 (Stretching vibrations by B sites) cm^{-1}**
$MnFe_2O_4$	532	470
$CoNiMnFe_2O_4$	564	439
$CoNiCdMnFe_2O_4$	577	443

Normally the vibration band in the tetrahedral sites occurs at higher wave number than octahedral sites, which is due to the smaller bond length of tetrahedral positions compared to the octahedral sites [6]. The wavenumber U_1 increased whereas U_2 decreased with the concentration of doping. This behaviour is attributed to the stretching of Fe-O bonds on the substitution of cations in A and B sites [7].

EDX Analysis:

Energy Dispersive X-ray Spectroscopy is used to find the elemental compositions of the prepared nanoferrites. All the spectra and data proved the presence of used materials without any impuities. The obtained spectra and data are presented below.

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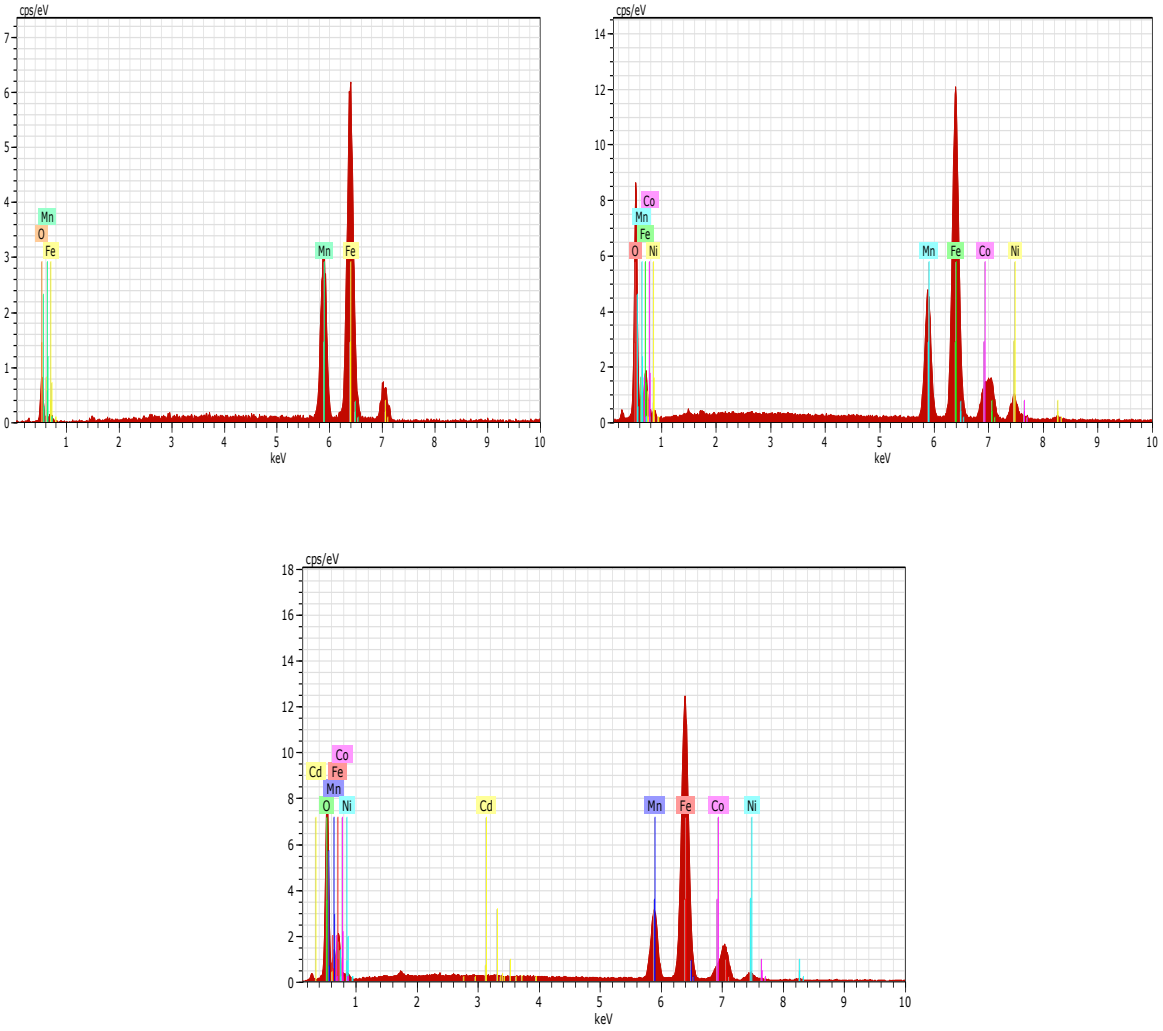

Fig 3: EDX spectra of $MnFe_2O_4$NPs, $CoNiMnFe_2O_4$NPs and $CoNiCdMnFe_2O_4$NPs

Table 4: EDX Data of $MnFe_2O_4$NPs

Elements	Wt%	Astm%
Iron	62.21	49.18
Oxygen	10.45	28.85
Manganese	27.35	21.97
Total	100	100

Table 5: EDX Data of $CoNiMnFe_2O_4$NPs

Elements	Wt%	Atm%
Iron	41.02	21.73
Oxygen	35.76	66.12
Manganese	13.29	7.16
Cobalt	5.29	2.66
Nickel	4.64	2.34
Total	100	100

Table 5: EDX Data of $CoNiCdMnFe_2O_4$NPs

Elements	Wt%	Atm%
Iron	47.94	24.60
Oxygen	38.14	68.30
Manganese	9.43	4.92
Cobalt	2.66	1.29
Cadmium	0.02	0.10
Nickel	1.81	0.88
Total	100	100

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VSM Analysis:

VSM (Vibrating Sample Magnetometer) is a technique used to measure the magnetic parameters such as saturation magnetization, coercivity and retentivity as a function of magnetic field, temperature and time. Here VSM studies are carried out at room temperature and shown in figure 4.

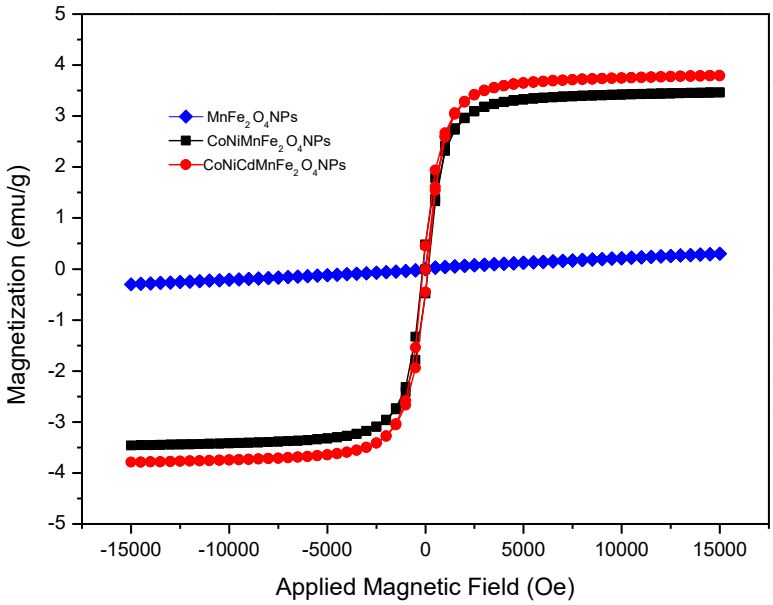

Fig 4: Hysteresis loops of the samples

Table 6: Magnetic parameters

Samples	Coercivity (G)	Saturation magnetic field(emu/g)	Retentivity (emu/g)
$MnFe_2O_4$	68.86	0.29	3.5E-9
$CoNiMnFe_2O_4$	133.07	12.11	1.68
$CoNiCdMnFe_2O_4$	113.64	13.27	1.58

For the undoped sample, all the measured values like coercivity, saturation magnetization and retentivity are lower than that of doped samples. When cobalt and nickel is added to manganese ferrite nanoparticles, its magnetic parameters are enlarged. The increase in coercivity may be due to increase in magnetic anisotropy [9]. The increase in saturation magnetization value may be attributed to the increasing of crystallinity and particle size of the nanoferrites [10]. When cobalt, nickel and cadmium are added as dopants in $MnFe_2O_4$NPs, its coercivity and retentivity decreased slightly. This is because of diamagnetic nature of cadmium ions.

IV. CONCLUSION

From the present work, manganese ferrite nanoparticles and transition metals (Co, Ni, Cd) doped manganese ferrite nanoparticles have been synthesized and investigated their structural and magnetic properties successfully. XRD results confirmed the cubic spinel structure of the nanoparticles in the range of 40-50nm. FTIR spectra showed two main peaks around 550 and 450cm^{-1} due to intrinsic stretching vibrations of A sites and B sites respectively. The compositions of the obtained nanoferrites were determined by EDX studies. According to VSM results, the magnetic parameters namely, saturation magnetization, coercivity and retentivity were found to be increased with the concentration of dopants.

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