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Study of the structural and magnetic properties of Gd doped $CoFe_2O_4$ nanoparticles synthesized via Sol-gel route

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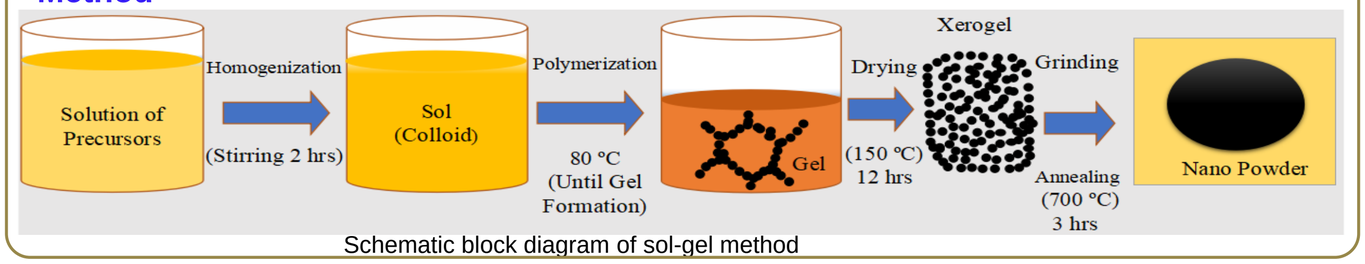
Motivation: Cobalt ferrite nano-particles has been attracting increasing interest owing to their remarkable chemical stability, high Curie temperature, large magnetocrystalline anisotropy, mechanical hardness and moderate to high saturation magnetization and coercivity. These properties make it suitable for variety of technological applications, such as high-density digital recording disks, magnetic resonance imaging (MRI), hyperthermia, magnetically guided drug delivery etc. Doping of rare-earth ions in spinel ferrite can alter the structural and magnetic properties of nanoparticles. Hence the motivation of this research is to study the structural and magnetic properties of Gd doped $CoFe_2O_4$ nanoparticles through sol-gel route.

Raw Materials

- $Co(NO_3)_2.6H_2O$,$Fe(NO_3)_3.9H_2O$ and $Gd(NO_3)_3.6H_2O$ (Metal precursors)
- Citric acid (Chelating agent)
- $NH_3.H_2O$ (to control pH)
- Water (Solvent)

Method



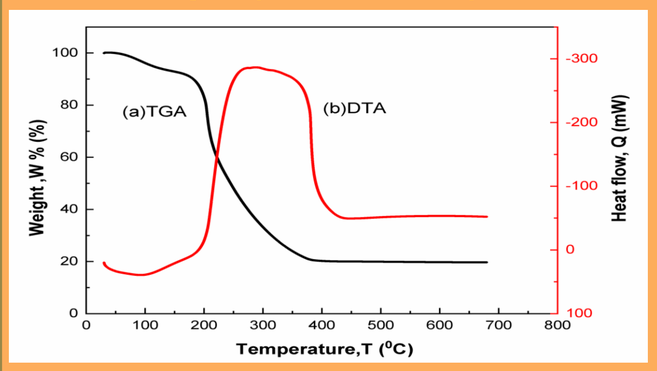
Schematic block diagram of sol-gel method

Characterization

- TGA and DTA
- TEM
- FTIR
- XRD
- VSM

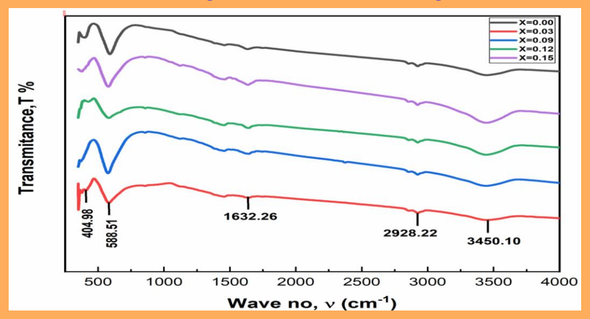
Results and Discussion

Thermal Analysis (TGA-DTA curve)



- Major weight loss occurred from 200 °C - 370 °C due to water vaporization and decomposition of organic materials.
- Stable phase after 400 °C indicate crystallization of spinel ferrite.

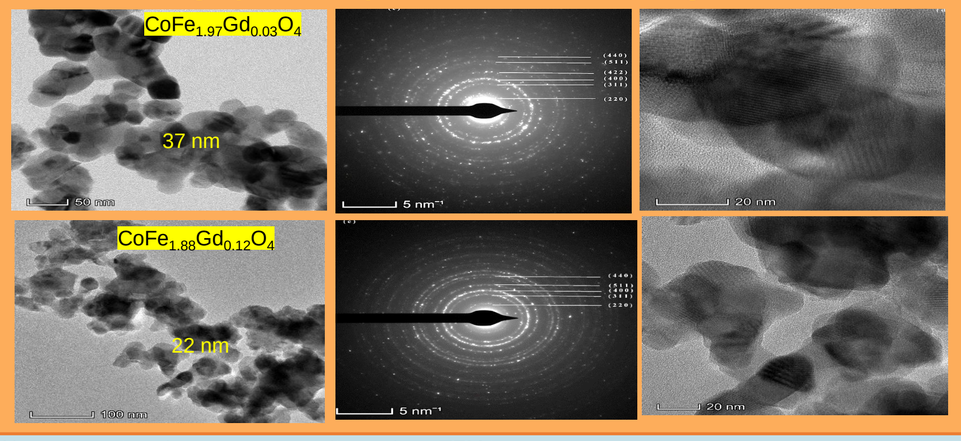
Structural analysis of the NPs by FTIR



Wave Number (cm^{-1})	Bond Vibration
404.98	Co-O
588.51	Fe-O
1632.26	N-H
3450.10	O-H

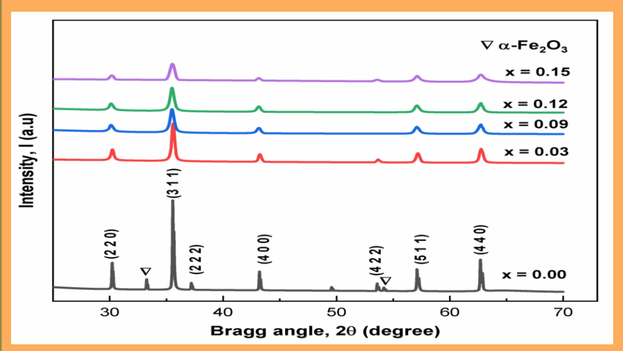
The wave number corresponding to the bond vibrations confirm the formation of spinel phase of Gd doped cobalt ferrite

TEM micrograph SAED pattern and HRTEM images

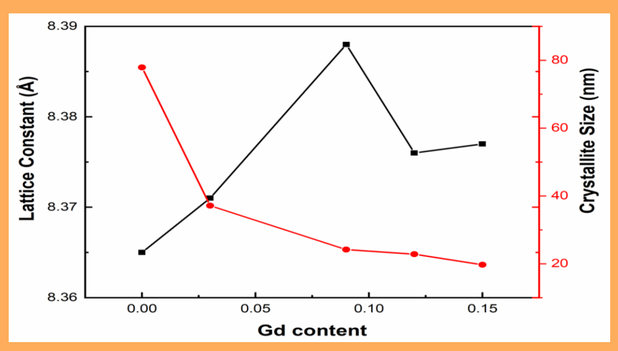


- Nanometer size of particles are confirmed from TEM images.
- Clear rings in SAED pattern confirm crystalline nature and fringes in HRTEM images ensure poly crystalline nature of the samples.

Structural analysis of the NPs by XRD



XRD patterns of the $CoFe_{2-x}Gd_xO_4$ (x= 0.0 to 0.15) samples annealed at 700 °C



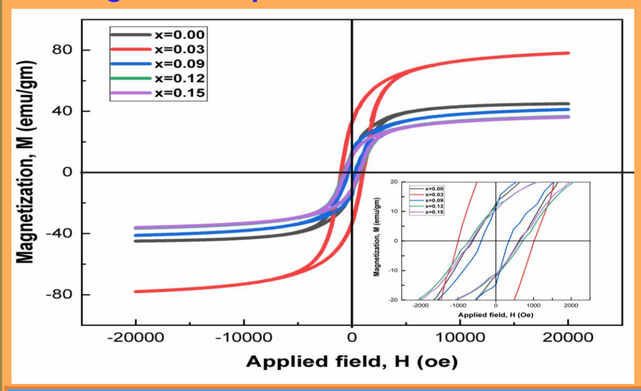
Variation of lattice constant and crystallite size with Gd content

Table: Parameters obtained from XRD data

Gd content (%)	Lattice constant, a (Å)	Crystalline size, D (nm)	Strain, ε	Dislocation density, δ	Packing factor, p
0.00	8.365	77.87	0.2645	0.000692	302.614
0.03	8.371	37.13	0.2672	0.000864	128.994
0.09	8.388	24.16	0.2672	0.001202	91.963
0.12	8.376	22.80	0.2672	0.001268	87.622
0.15	8.377	19.68	0.2672	0.001348	82.422

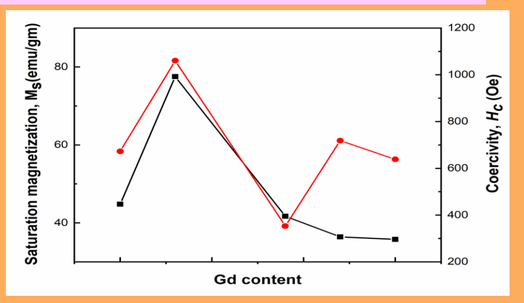
- XRD patterns confirm the formation of cubic spinel structure of Gd doped cobalt ferrite.
- The decrease in crystallite size with Gd substitution is due to suppression of grain growth at octahedral site.
- The increase in lattice constant is due to replacement of smaller Fe^{3+} (0.69 Å) ions by larger Gd^{3+} ions (0.938 Å)
- The crystallite size measured from XRD data is comparable with the particles size measured from TEM images.

Magnetic Properties



Room temperature hysteresis loops for the $CoFe_{2-x}Gd_xO_4$ (x=0.0 to 0.15) nanoparticles

The magnetization curves for all samples is '*S*' shaped which are the characteristics of ferromagnetic materials.



Variation of saturation magnetization and coercivity with Gd content

Table: Parameters estimated from hysteresis loop

Gd content x	Saturation Magnetization M_s (emu/gm)	Magnetic moment, n_B	Coercivity H_c (Oe)	Magnetic anisotropy constant, K_1(10^4 erg/gm)	Remnant magnetization M_r (emu/gm)	Remnant ratio, M_r/M_s
0.00	44.78	1.8812	672.59	1.5059	12.32	0.2751
0.03	77.53	3.2991	1060.19	4.1098	32.38	0.4176
0.09	41.71	1.7967	353.09	1.4727	10.41	0.2528
0.12	36.41	1.6088	718.33	2.6154	11.18	0.3070
0.15	35.75	1.5991	638.43	1.1411	9.21	0.2576

- The increase of Ms for x = 0.03 is due to the cation distribution in octahedral and tetrahedral sites.
- The increase of Hc for x = 0.03 is due to the anisotropy originated from the strong spin orbit coupling of Gd^{3+}.
- For further doping the coercivity is reduced due to the thermal effect and reduction of particle size.

Concluding remarks: Nanocrystalline $CoFe_{2-x}Gd_xO_4$, (x= 0.0 to 0.15) have been successfully synthesized via conventional sol-gel route. We provide a detail study of the structural, morphological and magnetic properties of the samples. The XRD analysis confirms the crystalline phase of cubic spinel structure. TEM images confirm the formation of nanometer size of the particles and SAED patterns indicate the higher crystallinity of the samples. The magnetic analysis shows the soft ferromagnetic behavior of all the samples. The change of saturation magnetization as well as coercivity with Gd content are owing to the cation distribution and spin orbit coupling of Gd^{3+} ions.