Magnetic Characterizations of Spinel Cobalt Ferrite Nanoparticles using Pulse Field Hysteresis Loop Tracer

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Abstract: The present paper reports magnetic characterizations of spinel cobalt ferrite nanoparticles using pulse field hysteresis loop tracer. The magnetic nanoparticles with 23 nm size were produced by sol-gel auto combustion synthesis which was annealed at 500°C for 4 h for better crystallinity. The obtained nanoparticles were first characterized by X-ray diffraction (XRD) technique. The XRD pattern resembles well with the reported pattern. The analysis of XRD data proves the formation of single phase cubic spinel structure of prepared nanoparticles. The XRD data was used to find a particles size, lattice constant, X-Ray density, lattice strain, dislocation density etc. All these structural parameters show good consistency with the reported values. The magnetic characterizations of the prepared cobalt ferrite nanoparticles were carried out at room temperature using pulse field hysteresis loop tracer. The important magnetic parameters such as saturation magnetization, coercivity, remenance magnetization and magneton number were deduced from M-H hysteresis curve. The obtained values of magnetic parameters show enhanced values as compare to the bulk cobalt ferrite sample. The obtained data can be useful in biomedical applications.

Index Terms - Cobalt ferrite nanoparticles, X-ray diffraction, M-H plot.

I. INTRODUCTION

In recent years the magneto-resistance ceramic iron particles, such as spinel-based structures, have attracted considerable attention because of their soft magnetic characteristics and magnetic dependency with the cation distribution in the crystal lattice [1]. Because of their relatively high electrical resistivity, low eddy current losses, high-frequency performance, significant chemical inertia, strong thermal stabilities, relatively large saturation magnetization and satisfactory magnetocrystalline anisotropy. Owing to their important electrical and magnetic properties ferrites are used to manufacture high-density magnetic recording media, high-frequency transformers cores, gas sensors etc.

Spinel ferrites are composed of iron oxides, which may be altered using other transition metal oxides with a general formula of AB_2O_4, where A represents a divalent metal ion. It is well known that the ferrites with the spinel structure are based on a face-center cubic lattice of the oxygen ions. Each spinel unit cell contains eight formula units. In each unit cell, there are 64 tetrahedral sites (A sites) and 32 octahedral sites (B sites). Therefore, the chemical, structural, magnetic, electrical and dielectric properties of ferrite are strongly influenced by their composition and microstructure. It is well known that the intrinsic properties of ferrites depend on the chemical composition, preparative conditions and substitutions. Important modification in structural and magnetic properties can be obtained by introduction of a relatively small amount of foreign ions [2]. In small particles, the saturation magnetization (Ms), magnetocrystalline anisotropy (K), Curie temperature (Tc) and coercivity (Hc) values are found to differ from their bulk behavior [3].

The research interest lies on cobalt ferrite-based material because of their potential applications in high-density information storage, magneto-optical devices and biomedical applications [4, 5]. This is because of various interesting properties possessed by the materials, e.g. strong anisotropy, high saturation, coercivity, etc. The magnetic properties of the $CoFe_2O_4$ are greatly affected by the size of the particles, by cation substitution and distribution [6-8].

In view of the above facts, the present work deals with the synthesis and magnetic properties evaluation of spinel cobalt ferrite nanoparticles synthesized by simple and cost effective sol-gel auto combustion technique. The prepared nanoparticles may show their suitability in the biomedical applications.

II. EXPERIMENTAL METHOD

Materials

Cobalt nitrate ($Co(NO_3)_2 \cdot 6H_2O$), ferric nitrate ($Fe(NO_3)_3 \cdot 9H_2O$) and citric acid were used as a raw materials for sol-gel auto combustion synthesis of $CoFe_2O_4$ spinel ferrite nanoparticles. All the reagents used for the synthesis were of analytical grade (AR) and used as received without further purification.

Preparation

Spinel cobalt ferrite nanoparticles were synthesized by sol-gel auto combustion method using citric acid as a fuel. The stoichiometric proportions of metal nitrates to fuel (citric acid) ratio as 1:3 were taken into separate glass beakers. The mixed solution was stirred for 30 - 35 minutes to dissolve completely into the double distilled water. Then they were mixed together after complete dissolution. Ammonia was added drop by drop into the solution to adjust pH value at 7. Then the neutralized solution was constantly magnetically stirred and heated at 90°C for 6 h on a hot plate. On the formation of sol, gel, very viscous gel the temperature was further raised up to 110°C so that the auto combustion of the dried gel started and finally powder was obtained. The as prepared loose cobalt ferrite powder was grinded for 40 minutes and annealed at 500°C for 4 h in muffle furnace.

Characterizations

The prepared spinel cobalt ferrite sample was characterized by X-ray diffraction (XRD) technique by Regaku model. The XRD patterns were recorded at room temperature in the 2θ range of 20° to 80° using Cu-Kα radiation (λ = 1.54056 Å). Using XRD data various structural parameters such as lattice constant, crystallite size, lattice strain and dislocation density. The magnetic properties of the sample were measured using pulse field hysteresis loop technique (Magnata Company) at room temperature. Using recorded M-H loop the values of saturation magnetization, remenance magnetization and coercivity of the cobalt ferrite nanoparticles were obtained.

III. RESULTS AND DISCUSSION

X-Ray diffraction studies

Fig. 1 represents room temperature X-Ray diffraction pattern of cobalt ferrite nanoparticles. The XRD pattern show the Bragg's reflections (220), (311), (222), (400), (422), (511) and (440). All these reflections belong to cubic spinel structure. No extra peak was found in the XRD pattern, indicating the formation of homogeneous, single phase cubic spinel structure compound. The values of Bragg's angle, intensity, interplanner spacing d (observed and calculated) etc. are listed in table 1. Using these values, lattice constant (a) was determined and its value is given in table 2. To confirm the nanocrystalline nature of the prepared samples, the crystallite size was evaluated using Scherrer's formula [9]. Using lattice constant values X-Ray density and unit cell volume was obtained. Also, the lattice strain and dislocation density were also obtained using the value of crystallite size. All these structural data are listed in table 2.

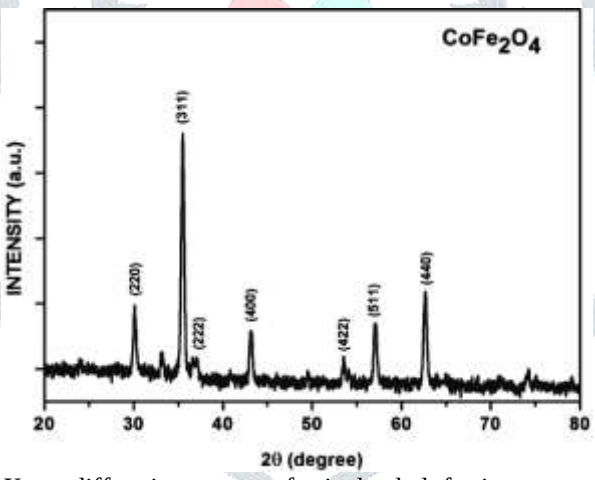

Fig. 1 X-ray diffraction pattern of spinel cobalt ferrite nanoparticles

Table 1 Values of Miller indices (h k l), Bragg's angle (θ), interplanar spacing (d), Intensity (I) and Relative intensity ratio (I/Io) for spinel cobalt ferrite nanoparticles

Parameters	(220)	(311)	(400)	(511)	(440)
2θ °	30.12	35.48	43.10	57.15	62.66
θ °	15.06	17.74	21.55	28.58	31.33
Sin θ	0.260	0.305	0.367	0.478	0.520
Sin θ / λ	0.169	0.198	0.238	0.310	0.338
d (Å)	2.964	2.528	2.097	1.610	1.481
I (a.u.)	2497.4	7761.0	2019.2	2587.5	3358.7
I / Io	32.18	100.00	26.02	33.34	43.28

Table 2 Values of Lattice constant (a), crystallite size (t), Unit cell volume (V), X-ray density (d_X), dislocation density (δ) and lattice strain (ε) for spinel cobalt ferrite nanoparticles

Parameters	$CoFe_2O_4$
a (Å)	8.376
t (nm)	23
V ($Å^3$)	587.63
d_X (gm/cm^3)	5.307
δ ($lines/m^2$) × 10^{14}	18.90
ε (%)	0.091

Magnetic properties studies

The magnetic characterizations of the prepared cobalt ferrite nanoparticles were done through pulse field hysteresis loop tracer. The measurements were recorded at room temperature. The plot of magnetization (M) vs applied field (H) exhibits typical hysteresis curve with greater coercivity. The M-H plot provides the information about the saturation magnetization (M_s), remenance magnetization (M_r) and coercivity (H_C). For the present hysteresis curve, the values of these magnetic parameters are listed in table 3. The values of saturation magnetization, coercivity and remenance magnetization are found to be greater as compared to bulk cobalt ferrite [10]. The comparative study of the magnetic parameters of bulk cobalt ferrite and cobalt ferrite nanoparticles clearly indicates the crystallite size effect. The nanosize particles exhibits enhanced magnetic properties compared to bulk material and these enhanced values of magnetic parameters can be useful for biomedical applications.

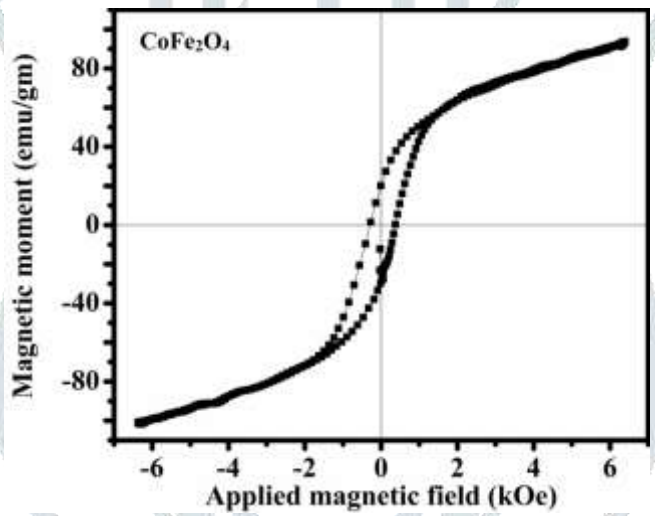

Fig. 2 M-H hysteresis loop for spinel cobalt ferrite nanoparticles

Table 3 Values of saturation magnetization (M_S), remenance magnetization (M_r), remenance ratio (M_r/M_S) coercivity (H_C) and magneton number for spinel cobalt ferrite nanoparticles

Parameters	$CoFe_2O_4$
M_S (emu/gm)	83.87
M_r (emu/gm)	21.15
M_r/M_S	0.252
H_C (Oe)	285.67
n_B (μ_B)	3.524

IV. CONCLUSION

Single phase cubic spinel structured cobalt ferrite nanoparticles with nanosized dimensions can be easily obtained through easy and low cost sol-gel auto combustion technique. The lattice constant and magnetic parameters are in good agreement with the reported literature values. The prepared cobalt ferrite nanoparticles can be useful for biomedical applications.

V. ACKNOWLEDGMENT

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