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Effects of Annealing Temperature on the Size of Zinc Doped Cobalt Ferrite Nanoparticles Synthesized using Green Binder

Sajid F. Shaikh[1], Bhagwan V. Jadhav[2], R. P. Patil[3]
Department of Chemistry
Anjuman Islam Janjira Degree College of Science, Murud-Janjira, Raigad, Maharashtra, India
Changu Kana Thakur College, New Panvel, Raigad, Maharashtra, India
M. H. Shinde Mahavidyalay, Tisangi, Kolhapur Maharashtra, India
sajidoshaikh@gmail.com, bvjadhav02@yahoo.com, patilraj_2005@rediffmail.com

Abstract: *Zinc doped cobalt ferrite nanoparticles have been synthesized by sol-gel auto combustion method from nitrate salts of respective metal ions using lemon as a green binder. The nanoparticles synthesized were annealed at different annealing temperatures of 873K, 973K, 1073K, 1173K and 1273K. The size of nanoparticles was also analyzed at a constant annealing temperature for different duration of time. X-ray diffraction pattern confirmed the formation of single-phase nanoparticles of Zinc doped cobalt ferrite. Fourier transform infrared study was performed to ascertain the structure of the nanoparticles. FT-IR studies also supported the trend of increasing size as shown by XRD results. Scanning Electron Microscopic (SEM) studies revealed nano crystalline nature of the sample. Energy dispersion X-ray analysis (EDAX) was performed to know an elemental composition of the sample and to confirm the stoichiometry.The study revealed that crystallinity enhanced and size of the nanoparticles increased with increasing annealing temperature due to coercivity.*

Keywords: Zinc Doped cobalt Ferrite Nanoparticles, sol-gel technique, XRD, FT-IR, EDAX

I. INTRODUCTION

Polycrystalline spinel ferrites are widely used in many electronic devices. They are preferred because of their high permeability in the radio- frequency (RF) region, high electrical resistivity, mechanical hardness and chemical stability. Ferrite nanoparticles are under intense research because of their unique chemical, mechanical, structural magnetic and electric properties.(1,2) Ferrite nanoparticles have versatile application in catalysis, electronics, photonics, sensors and Ferro fluids.(3) Ferrite nanoparticles are also used in biomedical sciences. Due to their unique size and shape, they can easily reach to the body parts where other conventional drugs find hard to reach.

Cobalt ferrite (CoFe2O4) is a well-known hard magnetic material with high coercivity and moderate magnetization. These properties, along with their great physical and chemical stability, make CoFe2O4 nanoparticles suitable for magnetic recording applications (4). Many efforts have been made to improve the basic properties of these ferrites by substituting or adding various cations of different valence states depending on the applications of interest. Among spinel ferrites, Zn^{2+} substituted CoFe2O4 nanoparticles exhibit improved properties such as excellent chemical stability, high corrosion resistivity, magnetocrystalline anisotropy, magnetostriction, and magneto-optical properties (5-7).

Size of the particles depends mostly on the preparation method and conditions. In The literature, there are a number of methods like as hydrothermal synthesis, mechanical Milling and hydrolysis of metal carboxylate in organic solvent (8-9), sol–gel pyrolysis method (10),the microwave hydrothermal method (11), template-assisted hydrothermal method (12), and combustion technique are used to prepare ferrites nanoparticles. However sol-gel auto combustion method is considered as an economical way of producing fine particles. Every method has the merits and demerits regarding the control of particles size and durability.

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In the current study, focus was placed on the $Co_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles prepared via sol-gel auto combustion method using lemon as green binder. The dependence of the morphology on annealing temperatures are investigated taking into account that mixed ferrites chosen here are highly sensitive to temperature (6).

II. Experimental

A. Materials

Metal nitrates and chelating agent used for synthesis were A.R. grade. Iron nitrate nonahydrate $Fe(NO_3)_3.9H_2O$, Zinc nitrate hexahydrate $Zn(NO_3)_2.6H_2O$, Cobalt nitrate hexahydrate $Co(NO_3)_2.6H_2O$, used were of purity 98-99%. The green binder is fresh lemon juice. The chemicals were used without further purification.

B. Experimental Procedure

$Co_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles were synthesized by sol-gel auto combustion method, using starting material of high purity AR grade metal nitrate and fresh lemon juice. The metal nitrate solutions were mixed in the required stoichiometric ratios in minimum quantity of distilled water. The solutions will be mixed on a magnetic stirrer at 353K.The pH of the solution was maintained between 9 and 9.5 using ammonia solution. The solution mixture was slowly heated around 373K with constant stirring to obtain a fluppy mass.On further heating, colored powder will obtained. The powder will cooled for some time. The obtained powder nanoparticles were annealed at different temperatures of 873K, 973K, 1073K, 1173K and 1273K for 4 hours.

C. Characterization

Synthesized nanoparticles was analyzed by X-ray diffraction technique (XRD) for structure and crystallinity. The formation of mixed metal ferrite nanoparticles was confirmed by Fourier transform infrared (FT-IR) studies. X-ray diffraction (XRD) data was collected at room temperature. Crystallographic properties e.g. phase of the material and crystal structure was determined using the same data.

FT-IR analysis was carried out in the range of **4500-100** cm^{-1}. The samples were pelleted with KBr. SEM-EDAX analysis was also performed to assure the prepared nanomaterials.

III. Results and Discussion

A. X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) patterns for the as prepared and annealed polycrystalline $Co_{0.4}Zn_{0.4}Mn_{0.2}Fe_2O_4$ferrite powders are presented in Figure 1. The XRD spectra showed all the characteristics peaks corresponding to the characteristic planes (311), (511)and (440) appear at 35°, 57° and 64° on comparing with the patterns of all the investigated samples with that of standard JCPDS card, a single phase $Co_{0.4}Zn_{0.4}Mn_{0.2}Fe_2O_4$has formed with no extra peaks. At 973K fully crystallized $Co_{0.4}Zn_{0.4}Mn_{0.2}Fe_2O_4$has formed with sharp peaks indexed as (220), (311), (400), (422), (500) planes of spinel structure. In the present work, Figure 1 reveals the presence of the spinel structure for the as prepared $Co_{0.5}Zn_{0.5}$ and the noticed broadening in the peaks of the as prepared sample could be attributed to the formation of ferrite particles in nano range. The sharpness of the peaks is also a good indicator for the increased size of the nanoparticles, as the particle size is increased by increasing the sharpness of the peak. The peak width decreases with the increase of annealing temperature which reflects the coarsening of particles. By increasing the annealing temperature, the diffraction peaks become sharper and narrower and increase in intensity. This is because of the amplification in crystallinity that leads to the increased particle size of the nuclei. It is evident from the XRD Pattern that crystallinity of ferrite nanoparticles is increased by increasing the annealing temperature.

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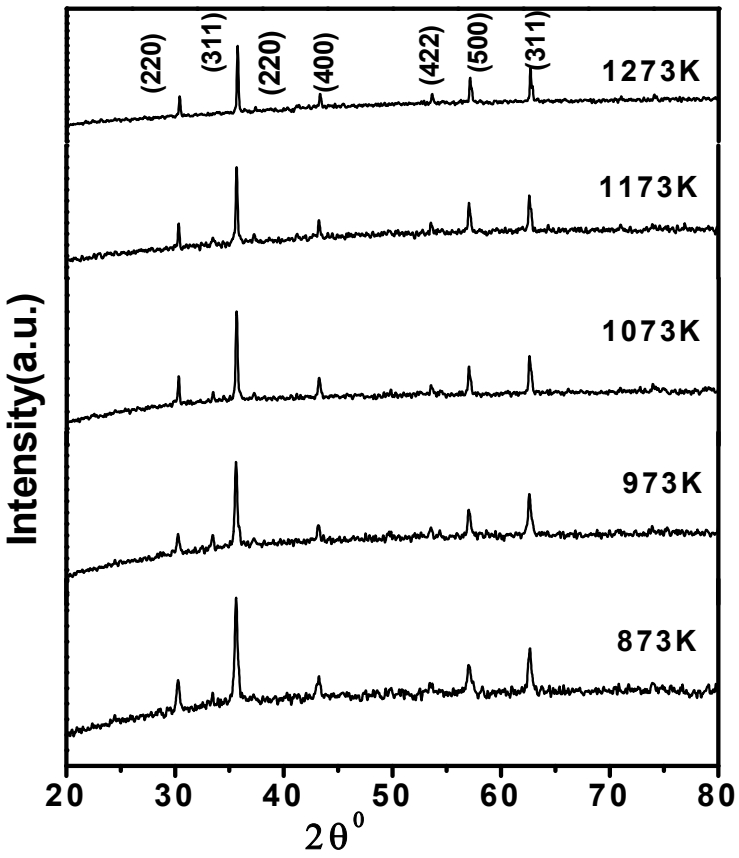

Figure 1: The X-ray diffraction (XRD) patterns for the polycrystalline $Co_{0.5}Zn_{0.5}Fe_2O_4$ferrites powders as prepared and that was annealed at different temperatures (873K-1273K)

B. Particle Size Calculation of Zinc Ferrite Nanoparticles by Scherer Formula

The sizes of the nanoparticles at different annealing temperatures have been determined using Scherer equation from the Full width at Half Maximum (FWHM) value of [311] diffraction peak.

$$D = 0.9\lambda/\beta \cos\theta$$

Where D is the particle size, λ is the X-ray wavelength (1.5418), θ is the Bragg angel and β is the half maximum width. The size of nanoparticles obtained at different annealing temperature was measured as 12.6 nm, 17.7 nm, 19.3nm, 24.9 nm and 27.2 nm for 873K, 973K, 1073K, 1173K and 1273K annealing temperature for 4 hours respectively.

The particle size increases from 12.6 nm to 27.2 nm with the systematic variation of annealing temperature calculated by Scherer formula as shown in Figure 2.

Table 1: The variation of the particle size of $Co_{0.4}Zn_{0.4}Mn_{0.2}Fe_2O_4$ferrites with different annealing Temperatures.

Sample ID	Annealing Temperature (K)	Size (nm)
SFS103600	873	12.6
SFS103700	973	17.7
SFS103800	1073	19.3
SFS103900	1173	24.9
SFS1031000	1273	27.2

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C. Fourier Transform Infrared (FT-IR) Studies

IR spectrum is considered an important tool to get information about the structure and the positions of ions in the crystal through the crystal's vibration modes (15).The formation of spinel $Co_{0.5}Zn_{0.5}Fe_2O_4$ structure in the calcined zinc ferrite nanoparticles is further supported by FT-IR spectra shown (Figure 2-4). The peaks at **556 cm^{-1}** correspond to the metal–oxygen (Fe–O) stretching vibrations and it is the characteristic peak of the spinel structure of $Co_{0.4}Zn_{0.4}Mn_{0.2}Fe_2O_4$nanoparticles. The peak at **3450** corresponds to the vibration of O-H and the light band at **1640** could be attributed to the adsorbed water or humidity. This was further supported by disappearance of these bands at higher temperature. The strong absorption band at **426 cm^{-1}** is described as the stretching modes of Zn–O (16).

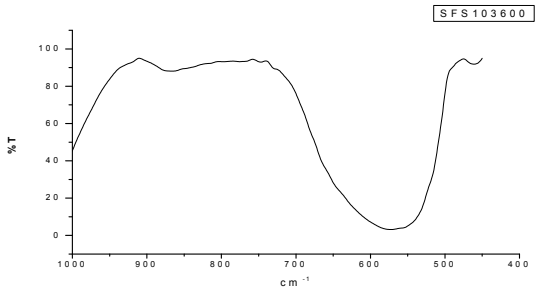

Figure 2: The FTIR spectra for the polycrystalline $Co_{0.5}Zn_{0.5}Fe_2O_4$ferrites powders as prepared and that was annealed at 873K.

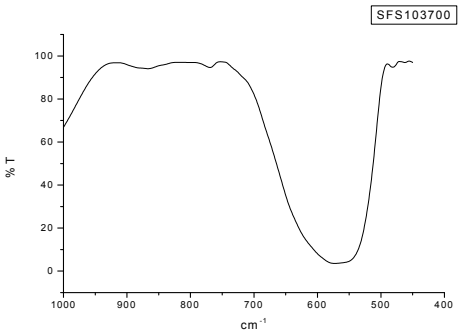

Figure 3: The FTIR spectra for the polycrystalline $Co_{0.5}Zn_{0.5}Fe_2O_4$ferrites powders as prepared and that was annealed at 973K.

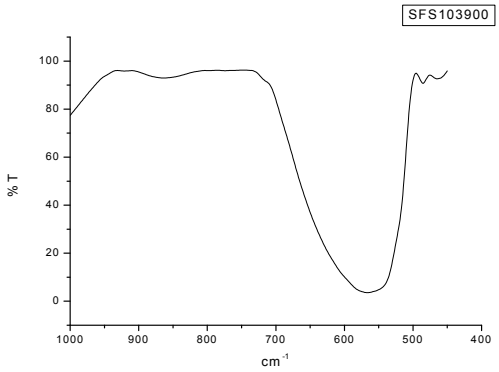

Figure 4: The FTIR spectra for the polycrystalline $Co_{0.5}Zn_{0.5}Fe_2O_4$ferrites powders as prepared and that was annealed at 1173K.

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D. Sem Analysis

- The scanning electron microscope (SEM) images of $Co_{0.5}Zn_{0.5}Fe_2O_4$ ferrites sintered at 700^0C is shown in Fig.5 & 6.
- In Fig.5 & 6, it can be seen that particles are well distributed and agglomerated.
- Usually, these agglomerates are formed by smaller size particles.
- The size of the grain is in the range of below 30 nm from Fig.5 & 6.
- SEM image reveal dense microstructures.

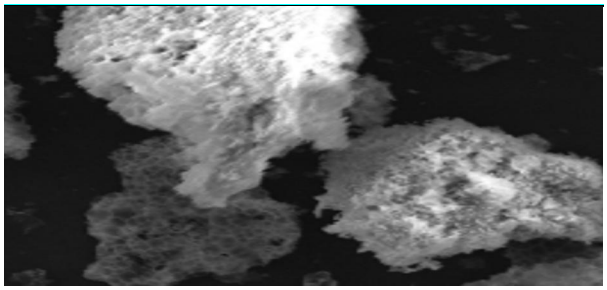

Figure 5: SEM micrograph of $Co_{0.5}Zn_{0.5}Fe_2O_4$ sintered at 973K

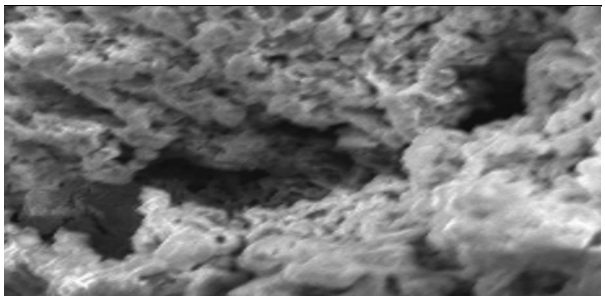

Figure 6: SEM micrograph of $Co_{0.5}Zn_{0.5}Fe_2O_4$sintered at 1073K

E. Energy dispersive x-ray spectroscopy (EDAX)

- Energy dispersive x-ray spectroscopy (EDAX) spectra of **$Co_{0.5}Zn_{0.5}Fe_2O_4$**sample sintered at 973K & 1073K are shown in Fig. 7 & 8.
- The presence of Co, Zn and Fe in the samples is depicted in the spectra.
- The EDAX analysis indicates the wt% of cobalt and zinc in these samples.
- The EDAX analysis does not show any variation in combination with respect to temperature.

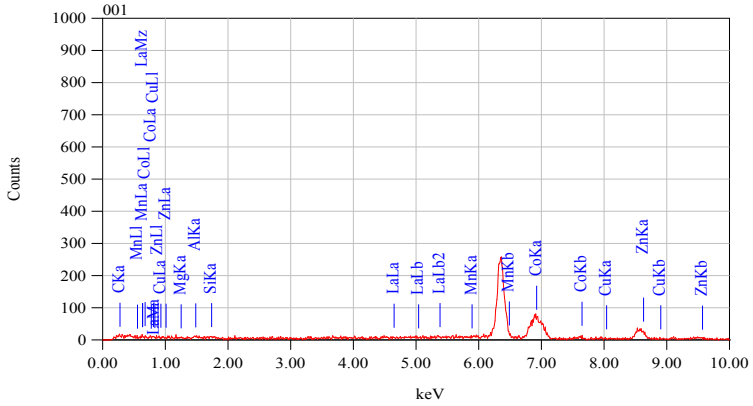

Figure 7: EDAX spectra of $Co_{0.5}Zn_{0.5}Fe_2O_4$sintered at 973K

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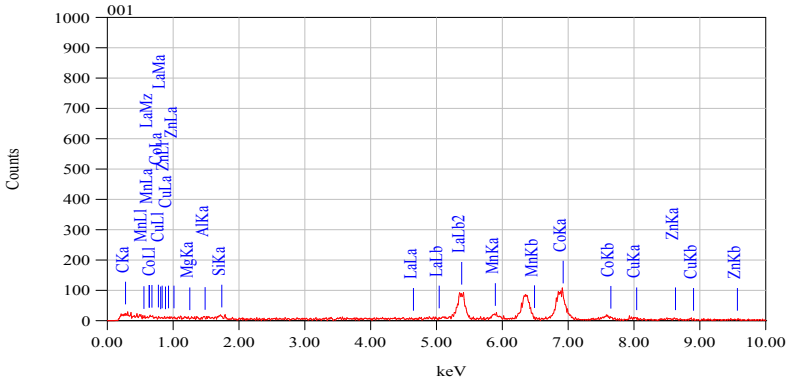

Figure 8: EDAX spectra of $Co_{0.5}Zn_{0.5}Fe_2O_4$ sintered at 1073K

IV. CONCLUSION

Nanosized $Co_{0.5}Zn_{0.5}Fe_2O_4$particles were synthesized by sol-gel autocombustion methods at different annealing temperatures to analyze the effect of annealing temperature on the particle size of the compound. The structure of the compounds was confirmed by XRD. FT-IR analysis supported the formation of spinel structure of $Co_{0.5}Zn_{0.5}Fe_2O_4$nanoparticles. The FT-IR spectra showed **two** characteristic metal oxygen vibrational bands, a Fe-O band, and Zn-O band.Scherer formula was used for the estimation of size of the $Co_{0.5}Zn_{0.5}Fe_2O_4$nanoparticles. Particles size of 11.6 nm, 15.7 nm, 18.8 nm, 23.6 nm and 26.5 nm were found at annealing temperature of 873K, 973K, 1073K, 1173K and 1273K respectively. The study shows that the particle size increases with increasing annealing temperature. SEM image of prepared sample show that the particles have an almost homogeneous distribution, and an agglomeration of nanoparticles with inhomogeneous broader grain size distribution. EDAX data give the elemental % and atomic % in the mixed $Co_{0.5}Zn_{0.5}Fe_2O_4$ system and it shows the presence of Zn, Co, Fe and O without precipitating cations.

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