

XRD Studies of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ Synthesis of Nanoparticles by Using Inorganic and Biogenic Fuel

S. A. Rode¹, P. S. Salunke², S K Fasate³, S. T. Alone^{4*}, K M Jadhav⁵

^aPratishthan College Paithan Tq. Paithan Dist. Chhatrapati Sambhajanagar 431107

^bC. T. Bora College of arts, Commerce and Science, Shirur, Dist. Pune 412210

^cVinayakrao Patil College Vaijapur, Dist. Chhatrapati Sambhajanagar 423701

^dRajarshi Shahu A C S College Pathri, Tq. Phulambri Dist. Chhatrapati Sambhajanagar 431111

^eMGM University, Chhatrapati Sambhajanagar

*Corresponding Author: drsureshtalone@gmail.com

Abstract: In the present study, $\text{Co}_{0.7}\text{Ca}_{0.3}\text{Fe}_2\text{O}_4$ nano ferrite was prepared by auto-combustion sol gel methods. X-ray powder diffraction (XRD) patterns of citric acid and garlic assisted $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles were studied systematically. Two different type of fuels i.e. citric acid as an organic fuel and garlic extract as a green fuel were used with a optimized metal nitrate to fuel ratio. The structural analysis performed by X-ray diffraction (XRD) technique confirmed the pure phase formation with spinel cubic crystal structure.

Keyword: XRD, Sol Gel, green Fuel

I. INTRODUCTION

Ferrites with various crystal structures are attractive to many researchers because of their interesting electrical and magnetic properties. On the basis of crystal structure are grouped into spinel ferrite, rare earth garnet, hexagonal ferrite among this, spinel ferrites are most promising candidate for the technological applications [1]. High electrical resistivity of the order of 10^6 to $10^9 \Omega\text{cm}$, high saturation magnetization of the order of 75 emu/g etc are the characteristics properties of the spinel ferrite. Generally, spinel ferrites are prepared by ceramic technique in which size obtained of the particle is of the order of μm level [2]. The developments in the nanoscience and nanotechnology have lead to prepare the materials in nanoscale dimensions. Therefore, in the current decades spinel ferrite nanoparticles have gained

much importance because of their interesting an unusual properties which are differ from bulk spinel ferrite [3].

Magnetic nanoparticles offer great potential application in a variety of biomedical fields, such as improved contrast agents for magnetic resonance imaging (MRI), cell separation, hyperthermia tumor treatment and as magnetic field-guided carriers for localizing drugs or radioactive therapies [4, 5]. A ferrite with a spinel structure which is formed by a nearly close packed FCC array of anions with holes partly filled by the cations can be represented by the formula AB_2O_4 , where A represents metallic ions located in A interstitial (tetrahedral) sites and B metallic ions located in B (octahedral) sites [6]. Due to the large electronegativity of oxygen, the ionic type of bonds prevails in almost all oxide spinel. The exchange interaction in spinel ferrites in which the antiparallel alignment of magnetic moments of the A-site with B-site is mediated by oxygen ions is called super-exchange interaction [7]. The strength of the super-exchange interaction between the cations depends on the A-O-B bond angle, which is the largest for an angle of 180° .

It is worth mentioning that, most of the magnetic properties of spinel ferrite strongly depend on the size and the shape of the nanoparticles, which are closely related to the preparation method. Various preparation techniques have been accordingly developed to produce spinel ferrite nanoparticles including chemical co-precipitation, microemulsion, sol-gel method, sol-gel auto-

combustion, hydrothermal, solvothermal, organic precursor method, ball milling, etc [8].

Nowadays, magnetic nanoparticles have got considerable attention of the researchers due to their potential applications in the field of high-density magnetic recording, microwave devices and medical sciences. Among the magnetic materials, cobalt ferrite (CoFe_2O_4) has drawn considerable attention due to remarkable properties, such as high coercivity, moderate saturation magnetization along with good mechanical hardness and chemical stability. On the basis of magnetic properties the cobalt ferrite considered as hard ferrites.

II. LITERATURE SURVEY

Jinpei Lin et al [9] synthesized cobalt-chromium ferrite, $\text{CoCr}_x\text{Fe}_{2-x}\text{O}_4$ ($x = 0-1.2$), has been by the sol-gel auto-combustion method. X-ray diffraction (XRD) indicates that samples calcined at 800°C for 3 h were a single-cubic phase. The lattice parameter decreased with increasing Cr concentration.

R. S. Melo et al [10] synthesized Nickel-doped cobalt ferrite $[\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4 (0 \leq x \leq 1)]$ nanoparticles by means of hydrothermal method. The structural, morphological and microstructural characterization revealed crystallite size was roughly spherical for lower nickel concentration while for higher ones in diamond shape consisting of nanosized grains.

S. Mishra et al [11] prepared cobalt ferrite nanoparticles by microwave hydrothermal synthesis and adsorption efficiency for organic dyes: Isotherms, thermodynamics and kinetic studies.

M. Sajjia et al [12] followed the sol-gel technique in the preparation of cobalt ferrite amorphous powder and reported that some heat treatment is necessary to completely decompose the organic and nitrate contents present in the amorphous powder.

A.B.Salunkhe et al [13] reported the effect of fuel characteristics on the processing of nano-sized cobalt ferrite fine powders by the combustion technique. By using

different combinations of glycine fuel and metal nitrates, the adiabatic flame temperature (T_{ad}) of the process as well as product characteristics could be controlled easily.

Mohammad Ali Zandi Khajeh et al [14] Nanostructured cobalt ferrite powders were prepared using the sol-gel process to obtain the optimal crystallite size and magnetic properties. The effect of sol-gel parameters, including the pH value, the mole ratio of ethylenediaminetetraacetic acid (EDTA) to metal ions, the mole ratio of ethylene glycol (EG) to metal ions and the heat-treatment temperature on the crystallite size and the magnetic properties of the samples, was investigated by Taguchi statistical experiments.

Many researchers have investigated the structural, morphological optical, magnetic, electrical, dielectric properties of pure and substituted cobalt ferrite. Very few researchers have focused their attention on the influence of varying fuel, varying annealing temperature, varying metal to nitrate ratio, varying pH etc synthesis parameters on the properties of mixed cobalt cadmium spinel ferrite. The selected composition of cobalt cadmium spinel ferrite that is $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ show enhanced magnetic properties. The use of inorganic fuels like citric acid, glycine etc produces toxic gases which are harmful to the environment. To overcome this problem, green synthesis approach has been emerged as a effective method for the preparation of spinel ferrite nanoparticles. The properties of spinel ferrite are more sensitive to crystallite size, which depends on method of preparation and annealing temperature.

In view of the above facts, the aim of the present work is to understand the influence of varying inorganic and organic fuel as well as annealing temperature on the synthesis, structure, morphology, dielectric and magnetic properties of mixed spinel ferrite of cobalt and cadmium having the formula $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$.

All the reagents used for the synthesis of cobalt ferrite nanoparticles were analytical grade and used as received without further purification. Cobalt ferrite $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, cadmium nitrate ($\text{Cd}(\text{NO}_3)_2$), ferric nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), citric acid ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$), garlic (*Allium sativum*), ammonia (NH_3) and distilled water was used as raw materials

III. SYNTHESIS PROCEDURE

Synthesis of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles was carried out by using sol-gel autocombustion method. The stoichiometric amounts of cobalt ferrite, cadmium nitrate and ferric nitrate were dissolved in distilled water under magnetic stirring. The inorganic fuel (citric acid) biogenic fuel (garlic) was mixed in the metal nitrate solution to chelate Co^{2+} , Cd^{2+} and Fe^{3+} ions in the solution. A small amount of ammonia was added drop-wise into the solution to adjust pH value to about 7 and stabilize the nitrate-citrate solution. The neutralities solution was evaporated to dryness by heating at 80°C on a hot plate with continuous stirring until it becomes viscous and finally formed a very viscous gel. The temperature is further raised up to 110°C so that the ignition of the gel starts. The dried gel burnt completely in a self propagating combustion manner to form a loose powder. Finally the as burnt powders were annealed at temperature 650°C for 6 h.

IV. CHARACTERIZATION TOOLS

The structural properties of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles was investigated by using X-ray diffraction (XRD) technique (Bruker D8 advance) at room temperature using $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$).

V. RESULTS AND DISCUSSION

X-ray powder diffraction (XRD)

X-ray powder diffraction (XRD) patterns of citric acid and garlic assisted $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles are shown in Fig 1. XRD pattern shows the reflections (220), (311), (222), (400), (422), (511), (440) and (533). These reflections are the indication of the presence of a cubic spinel structure. The diffraction line corresponding to a cubic spinel type and crystalline phase provide clear evidence of the single phase formation of a $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles spinel ferrite.

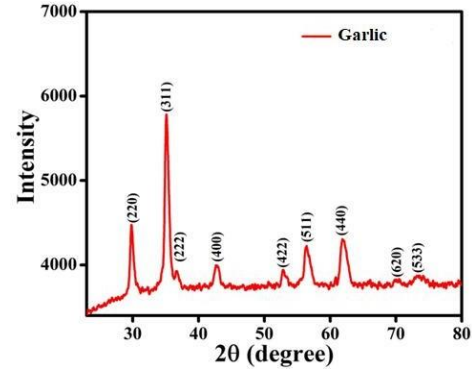
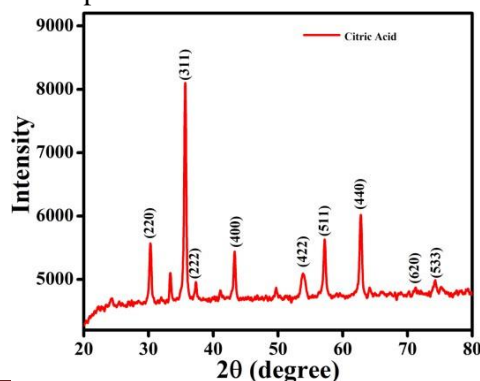


Fig. 1 XRD pattern of citric acid and garlic assisted $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles.

The XRD patterns are sharp and intense. The values of inter planer spacing (d) along with the Millar indices (hkl) are used to calculate the lattice constant and other structural parameters. The obtained values of lattice constant are in good agreement with the reported literature.

VI. CONCLUSIONS

The mixed spinel ferrite nanoparticles with composition $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ were prepared by well known sol-gel autocombustion method. The effect of the fuel type and sintering temperature on the structural, morphological, magnetic and dielectric properties of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles were studied systematically. Two different type of fuels i.e. citric acid as an organic fuel and garlic extract as a green fuel were used with a optimized metal nitrate to fuel ratio. The structural analysis performed by X-ray diffraction (XRD) technique confirmed the pure phase formation with spinel cubic crystal structure. Using XRD data, the different structural parameters such as lattice constant, crystallite size, unit cell volume etc. were calculated from standard relations and are found to be in the reported range. Further, the effect of the sintering temperature on structural properties of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles was undertaken by XRD technique. With increase in sintering temperature, the intensity of the peaks was observed to be increased.

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