

Infrared Spectroscopic Studies of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ Synthesis of nanoparticles by considering effect of annealing temperature

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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. With increase in sintering temperature, the intensity of the peaks was observed to be increased. The presence of the two characteristics peaks belonging to the tetrahedral and octahedral stretching vibrations were analyzed by Fourier transform infrared spectroscopy (FTIR) in the wavenumber range of 400 to 4000 cm^{-1} . Using FTIR data, the force constant values and bond length were determined by using standard relations.

Keyword: XRD, Sol Gel, green Fuel, IR.

I. INTRODUCTION

Many researchers are drawn to ferrites with different crystal forms because of their intriguing electrical and magnetic characteristics. Hexagonal ferrite, rare earth garnet, and spinel ferrite are classified according to their crystal structures; among these, spinel ferrites are the most promising option for technological uses [1]. The features of spinel ferrite include high electrical resistivity of 106 to 109 cm and high saturation magnetisation of 75 emu/g , among others. Typically, the ceramic method is used to create spinel ferrites, and the resulting particle size is on the order of μm [2]. The preparation of materials at nanoscale dimensions is the result of advancements in nanoscience and nanotechnology. Because of their unique and intriguing characteristics that set them apart from bulk spinel ferrite, spinel ferrite nanoparticles have

become more significant in recent decades[3]. Numerous biomedical sectors might benefit greatly from the use of magnetic nanoparticles, including cell separation, hyperthermia tumour treatment, enhanced contrast agents for magnetic resonance imaging (MRI), and magnetic field-guided carriers for drug or radiation therapy localisation[4, 5]. The formula for a ferrite with a spinel structure, which is made up of an almost closely packed FCC array of anions with holes partially filled by the cations, is AB_2O_4 , where A stands for metallic ions in interstitial (tetrahedral) sites A and B for metallic ions in interstitial (octahedral) sites B[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. Different methods have been established to synthesize spinel ferrite nanoparticles, such as chemical co-precipitation, microemulsion, the sol-gel technique, sol-gel auto-combustion, hydrothermal processes, solvothermal methods, the organic precursor approach, and ball milling, among others. [8].

Currently, researchers are increasingly focused on magnetic nanoparticles because of their promising

uses in high-density magnetic recording, microwave technology, and medical applications. Within the category of magnetic materials, cobalt ferrite (CoFe_2O_4) has garnered significant interest because of its exceptional characteristics, including high coercivity, moderate saturation magnetization, as well as strong mechanical strength and chemical durability. Cobalt ferrite is classified as a hard ferrite based on its magnetic properties.

II. LITERATURE SURVEY

The synthesized cobalt-chromium ferrite, $\text{CoCr}_x\text{Fe}_{2-x}\text{O}_4$ (with x ranging from 0 to 1.2), was produced using the sol-gel auto-combustion technique. X-ray diffraction (XRD) analysis shows that the samples calcined at 800°C for 3 hours exhibited a single cubic phase. The lattice parameter was observed to decrease as the concentration of chromium increased, according to Jinpei Lin et al [9]. Nickel-doped cobalt ferrite nanoparticles [$\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ ($0 \leq x \leq 1$)] were produced using a hydrothermal technique. Characterization of the structure, morphology, and microstructure indicated that the crystallite size appeared roughly spherical at lower nickel concentrations, whereas at higher concentrations, the shape was diamond-like, consisting of nanoscale grains, as noted by R. S. Melo et al [10]. Cobalt ferrite nanoparticles through microwave hydrothermal synthesis and the adsorption effectiveness for organic dyes: Isotherms, thermodynamics, and kinetic analyses were developed by S. Mishra et al [11]. The sol-gel method in creating cobalt ferrite amorphous powder and indicated that certain heat treatment is essential to thoroughly decompose the organic and nitrate components found in the amorphous powder were prepared by M. Sajjia et al [12]. The impact of fuel properties on the processing of nano-sized cobalt ferrite fine powders through the combustion method. By utilizing various mixtures of glycine fuel and metal nitrates, the adiabatic flame temperature (T_{ad}) of the process along with product characteristics could be effectively managed as noted by A. B. Salunkhe et al [13].

Nanostructured cobalt ferrite powders were synthesized via the sol-gel method to achieve the ideal crystallite size and magnetic characteristics. The influence of sol-gel variables, such as the pH level, the mole ratio of ethylene diamine tetra acetic 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 characteristics of the specimens, was examined using Taguchi statistical experiments Mohammad Ali Zandi Khajeh et al [14].

Many researchers have explored the characteristics, morphology, optical, magnetic, electrical, and dielectric attributes of pure and substituted cobalt ferrite. A limited number of researchers have concentrated on the effects of different fuel types, diverse annealing temperatures, various metal to nitrate ratios, changing pH levels, and other synthesis parameters on the characteristics of mixed cobalt cadmium spinel ferrite. The chosen composition of cobalt cadmium spinel ferrite, which is $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$, demonstrates improved magnetic properties. The employment of inorganic fuels such as citric acid and glycine releases toxic gases that pose a threat to the environment. To address this issue, a green synthesis method has emerged as an effective technique for synthesizing spinel ferrite nanoparticles. The attributes of spinel ferrite are increasingly influenced by crystallite size, which is dependent on the preparation method 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 $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$.

III. EXPERIMENTAL TECHNIQUE

i) Materials

All the chemical reagents for the synthesis of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles such as cobalt nitrate hexahydrate, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and iron nitrate nonahydrate, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, ammonia hydroxide, malachite green, and ethanol were commercially available and employed without further purification.

ii) Synthesis of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles

The synthesis of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles were done by sol-gel autocombustion method using citric acid as a fuel. The stoichiometric amounts of metal nitrates were mixed in a minimum amount of water required to dissolve it completely. Further the pH of the solution adjusted at 7. The pH adjusted solution then heated at 80°C for 2-3h.

Further the temperature was raised to 120°C to convert into gel. The gel formed solution starts to combust to convert it to a loose power. The loose power was collected and grinded using mortar pestle. The well grinded sintered at 600°C to get pure phase nanocrystalline power.

iii) Characterizations techniques

$\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles were characterized by a series of technologies involving field emission scanning electron microscopy (FE-SEM).

IV. RESULTS AND DISCUSSION

Fourier transform infrared spectroscopy (FTIR)

In general the information about the vibrational modes in crystals and the position of the ions is probed by Fourier transform infrared spectroscopy (FTIR). In particular to spinel ferrites, FTIR is considered a very important tool to provide the auxiliary confirmation to preparation of the crystal structure by the characteristic bands of metal oxygen (M-O) at tetrahedral (ν_1) and octahedral (ν_2) sites. FT-IR spectra of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles sintered at different temperature are presented in the Fig.1. Fig. 1 reveals the two absorption bands at around 600 and 400 cm^{-1} corresponding to tetrahedral and octahedral vibrations, which provides the information about the formation of cubic structure. The vibration ν_1 at tetrahedral site is caused by the stretching if M-O and oxygen bond while that of ν_2 at octahedral site by bending of M-O.

V. CONCLUSIONS

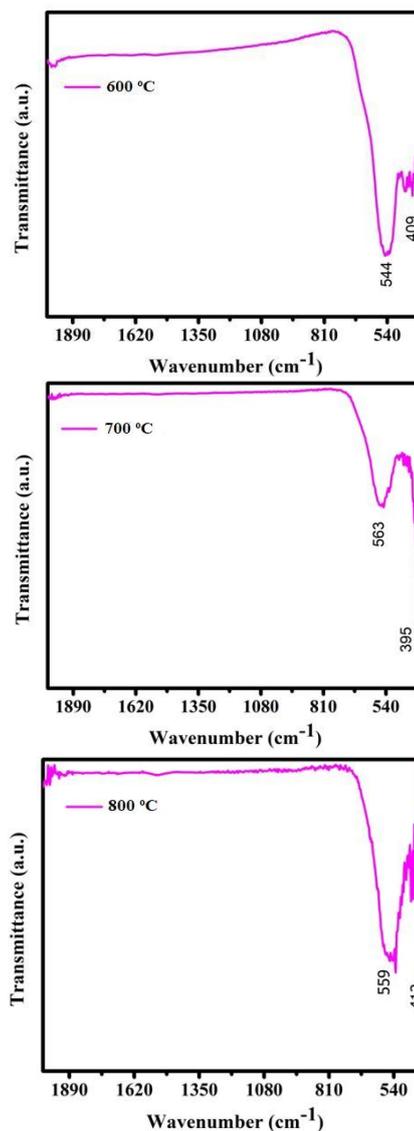
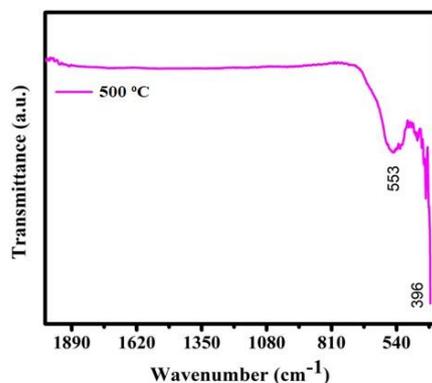


Fig. 1 FT-IR spectra of $\text{Co}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles sintered at different temperature

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. With increase in sintering temperature, the intensity of the peaks was observed to be increased. The presence of the two characteristics peaks belonging to the tetrahedral and octahedral stretching vibrations were analyzed by Fourier transform infrared spectroscopy (FTIR) in the wavenumber range of 400 to 4000 cm^{-1} . Using FTIR data, the force constant values and bond length were determined by using standard relations.

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