CONTROLLABLE SYNTHESIS AND CHARACTERIZATION OF CU-COZNFE$_2$O$_4$ NANO FERRITES BY AUTO-COMBUSTION ROUTE

Abstract

The methods employed to prepare nano crystalline mixed ferrite materials during experimentation significantly affect the resulting particle size. The quality of ferrite materials greatly influences the performance of devices. The chemical composition and microstructure of ferrites, which determine their properties, are both determined by the preparation process. TheCoCuZnFe$_2$O$_4$ ferrite nanoferites were prepared by sol-gel method. The prepared samples were calcinated at 750^{o}C shows sharper X-ray diffraction peaks, indicating increased crystallinity of the materials. Average crystallite sizes were obtained in the ranges 30.4-42.1 nm for the samples calcinated at 450 ℃ and 750 ℃, respectively. Images using scanning electron microscopy (SEM) showed clumps of particles. The overall acidity was reduced for the samples that were calcinated at higher temperatures. The findings highlight how the preparation circumstances affect nano ferrites' crystallite size, shape, and magnetic characteristics.

Keywords: XRD, DSC, TGA, SEM, Nanoferites

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I. INTRODUCTION

Nanotechnology encompasses a wide range of disciplines, from inventing novel materials with nanoscale dimensions to examining if we can directly alter matter on the atomic scale. It ranges from extensions of standard device physics to whole new methodologies based upon molecule self-assembly. Nanotechnology holds the promise of producing a wide variety of novel materials and gadgets with a diverse range of use, including in medicine, energy production, and electronics. On the other hand, nanotechnology brings many of the same problems as when new technology is introduced, such as worries aboutthe economy [1, 2].When materials are scaled down to the nanoscale, they exhibit different properties than they do at the macroscale. This creates new opportunities for their use. For example, some solids that are normally opaque, such as copper, become translucent at the nanoscale. Similarly, some stable solids, like aluminium, become flammable, and some insoluble solids, like gold, become soluble. When substances like gold are reduced to nanoscales, they can display powerful chemical catalytic properties, despite being chemically inert at larger dimensions. These quantum and surface phenomena observed in matter at the nanoscale are what make nanotechnology an attractive field of study [3-5].

Ferrites, specifically spinel ferrite, possess a crystal structure that comprises two interstitial sites - tetrahedral and octahedral - capable of accommodating various cations. This feature allows for diverse properties in ferrites as other divalent metal ions can replace ferrites. Research has demonstrated that substituting Al^{3+} in cobalt ferrite nanoparticles can modify their electromagnetic properties, making them a promising option for microwave and power device applications. However, replacing Zn in $MgFe_2O_4$ can increase resistivity while decreasing magnetic properties. Semiconducting behaviour becomes a trend after Cr^{3+} substitution. Substituting Cr^{3+} ions in the nickel-zinc ferrite system can also increase resistivity, making the prepared samples suitable for higher frequency ranging systems. Ferrites can also improve the activation energy and thermal decomposition of propellants and can be easily separated using an external magnet [6-9]. Various methods such as sol-gel, hydrothermal, and coprecipitation are utilised to synthesise ferrite nanoparticles. This article reports on the effect of cobalt copper zinc ferrite ($CoCuZnFe_2O_4$) using a low-cost sol-gel method.

II. MATERIALS AND METHODS

All the chemicals used were of analytical grade without further purification and available from commercial sources: cobalt(II)nitrate hexahydrate [$Co\text{-}(NO_3)_2.6H_2O$, 99%], copper(II)nitrate trihydrate [$Cu\text{-}\ (NO_3)_2.3H_2O$, 95%], zinc(II)nitrate hexahydrate [$Zn\text{-}(NO_3)_2.6H_2O$, 99%], ferric(III)nitrate nonahydrate [$Fe\text{-}\ (NO_3)_3.9H_2O$, 98%], and sodium hydroxide (NaOH), Glycine. Laboratory-setup triple distilled water was used as a solvent.

Cobalt copper zinc ferrite powder was synthesized bythe auto-combustion method. The stoichiometric amounts of nitrates to the fuel are taken as 1:3. Initially, all the nitrates were mixedinto the distilled water and fuel is added to the solution. These solutions are mixed using a magnetic stirrer and heated for about 45 min. The solution is continuously stirred till the temperature reaches 80 ^{0}C and pH 7 is maintained by using ammonia of suitable quantity, then, the mixed solution is transferred into a heating mantel which is maintained at a temperature of about 100 ^{0}C. Upon heating, the solution is turned into dried gel. The dried gel undergoes auto-combustion and forms a foam-like structure. After cooling, the obtained foam is converted into ash, poured into the mortar, and will be grinded to form a fine powder. The obtained fine powder is kept under sintered at 600 ^{0}C temperature for about 6 hours using a muffle furnace. Finally, the Cobalt Copper Zinc Ferritesare obtained.

III. CHARACTERIZATION

We used the Panalytical X-Pert Pro MPD instrument to record the X-ray diffraction patterns of the samples we synthesized. The samples were analyzed in the 2θ range of 10-80º, with a scanning speed of 5º/min and step size of 0.02º. To analyze the morphology of the samples, we used the FESEM CARL ZEISS instrument. This microscope has a field emission gun and can operate with accelerating voltage variable from 0.5 to 30 kV, with 2 nm resolution. We also carried out thermal analysis of the samples using TGA/DTA.

1. **X-Ray Diffraction Studies on as prepared $CuZnFe_2O_4$ and $CoZnFe_2O_4$ Nano Ferrites:** Structural analysis of the $CoZnFe_2O_4$ and $CuZnFe_2O_4$ were obtained from powder XRD which is depicted in Figures 1 and 2.A single-phase spinel cubic structure can be seen in the prepared sample according to the XRD peaks (1 1 1), (0 2 2), (1 1 3), (2 2 2), (2 2 4), (3 3 3), and (0 4 4).In some of the samples, a secondary phase of hematite with a cubic spinel structure is forming partially. Previous studies have found single-phase Co-Zn ferrite nanoparticles created through the sol-gel process and annealed at temperatures below 600°C. These nanoparticles correspond with the JCPDS card numbers 52-0277 and 89-0599, which represent spinel Co-Zn ferrite and secondary hematite phase, respectively, and the diffraction peaks are in good agreement [10].At high concentrations of cobalt doping, the secondary hematite phase's peak strength decreased. To determine the average crystallite size of all the synthesized samples, Scherer's equation [11, 12] was used, and the full-width half maximum (FWHM) of the most prominent peak (1 1 3) of the XRD patterns was analyzed.

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

where D is the average crystallite size, β is the intensity FWHM in radians and $\lambda = 1.54\,\mathring{A}$ is the wavelength of X-ray andθ is the Bragg's angle [13].

It is discovered that cobalt doping results in an increase in crystallite size (D) from 25 to 31 nm. Nearly the same crystallite size (D) was achieved at x=0.5% (28 nm) and x=0.5% (30 nm). Due to this slight dopant fluctuation, other estimated structural parameters at x = 0.03 and 0.09 have values that are nearly identical. The spacing (d) and lattice constant (a) [14, 15] are two structural parameters that are impacted by Co doping and have been determined by using the relation:

$$2d \sin \theta = n\lambda$$

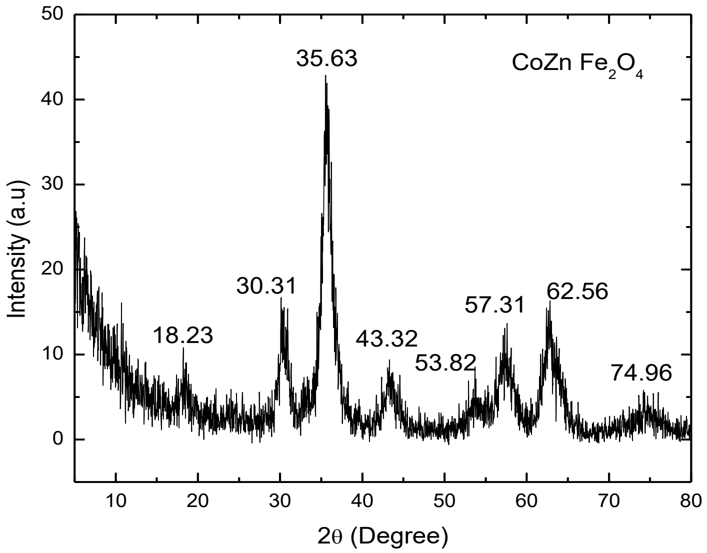

Figure 1: XRD pattern of $CoZnFe_2O_4$Nanoferrite

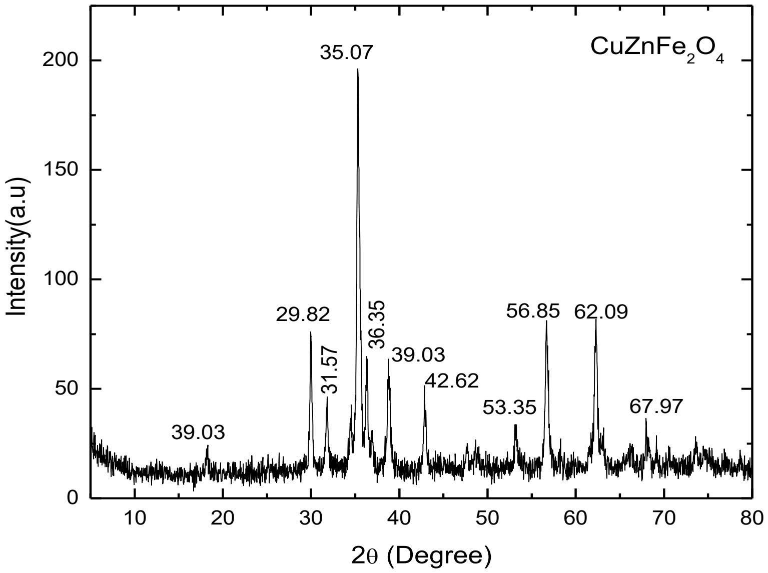

Figure2: XRD pattern of $CuZnFe_2O_4$Nanoferrite

2. **FESEM Analysis:** $CuZnFe_2O_4$ and $CoZnFe_2O_4$ Nano Ferrites were synthesized and dried at 90 ^{0}C in a vacuum and calcined at 500 ^{0}C for 45 min, respectively, in Figure 3. It is evident that crystal agglomeration occurs as can be seen from Figure 3 (a). It typically results from the formation of smaller crystals. There are several spherical crystals that are substantially smaller or have nanometer dimensions that are below 100 nm. This is consistent with the XRD pattern, which showed the peak broadening for these powder specimens. The grain size was assessed from a SEM micrograph, the crystal size increased (Fig. 3b).This value is consistent with the XRD data results. The extremely narrow XRD peaks show that larger grains are larger than the nanoscale area. Low-resolution micrographs clearly show the impact of heat treatment on the fine-grainsize of the specimens (Fig. 3b).

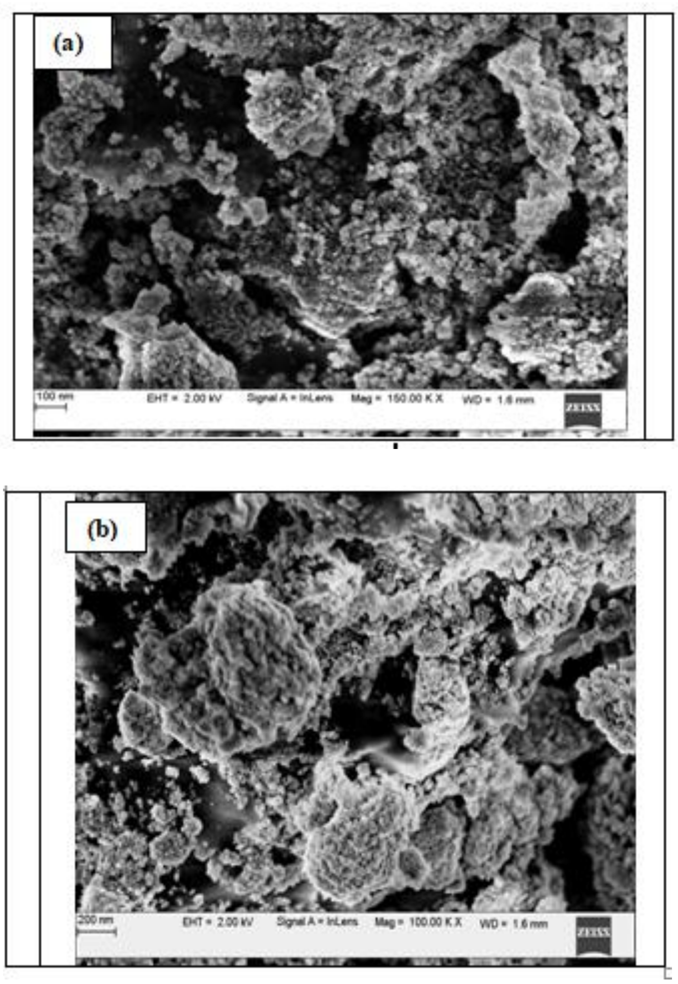

Figure 3: FESEM micrograph of (a) $CuZnFe_2O_4$ and (b)$CoZnFe_2O_4$ sintered at 500 ^{0}C.

3. DSC and DTG Analysis:

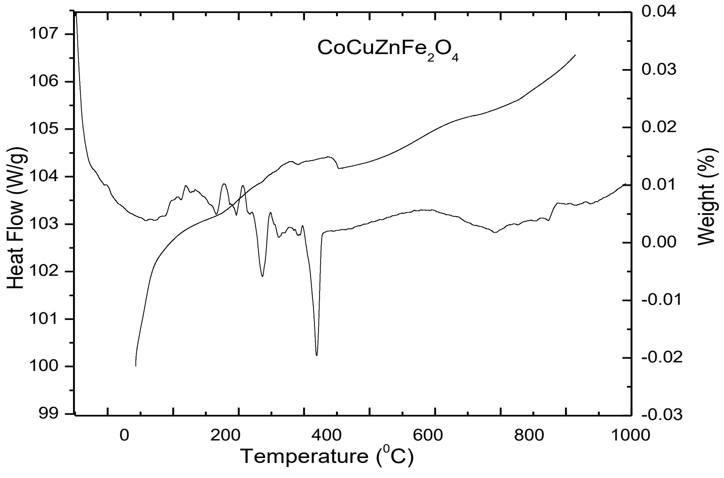

Figure 4: DSC/TGA Spectra of$CoCuZnFe_2O_4$Nanoferrite.

The DSC/TGA curve of $CoCuZnFe_2O_4$nanoferrites is shown in Figure 4. The initial weight loss in the $CuZnFe_2O_4$and $CoZnFe_2O_4$nanoferrites was around 10% when the temperature was less than 100 ^oC. The sample was thermally stable and the weight change was insignificant. Due to the degradation of the polymer chains, there is a weight loss of about 50% in the second phase in the temperature range of 100 ^oC to 450 ^oC and a greater weight loss (about 68%) in the temperature range of 500^oC to 800 ^oC. It was observed that the quantity of $CuCoZnFe_2O_4$nanoferrites contained in the composite affected the breakdown temperature of the nanoferrites [16-18].

IV. CONCLUSIONS

The current work illustrated the structural, thermal and chemical characteristics of prepared $CuZnFe_2O_4$ and $CoZnFe_2O_4$nanoferrites. XRD, FE-SEM, and DSC analysis techniques were used to investigate the preparedsamples. Samples exhibit a single-phase spinel phase cubic structure according to XRD analysis. $CuZnFe_2O_4$ and $CoZnFe_2O_4$nanoferrites create a spinel phase cubic structure with a partial secondary hematite phase (α-Fe_2O_3) development. Because Co^{+2} ions have higher ionic radii than Cu^{+2} ions, the crystallite size (D) increases with Co and Cu doping. FESEM gave information about the crystallinity of the prepared samples. From TGA, the thermal stability was studied.

Conflict of interest

The author declares that they have no conflict of interest.

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