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ZINC DOPED COBALT FERRITES FOR ENHANCED MAGNETIC HYPERTHERMIA PERFORMANCE

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Article Info

Abstract

Overview of the synthesis, structural characterization, and magnetic hyperthermia performance of Zn-$CoFe_2O_4$ nanoparticles as a future candidate for biomedical applications. Hydrothermal method was used to prepare the nanoparticles with Zn substitution levels from 0.0 until 0.7. The single-phase spinel ferrite structures formation was confirmed by X-ray diffraction analysis. The inclusion of zinc modified the crystal structure of cobalt ferrite affecting fundamental magnetic characteristics including saturation magnetization, coercivity, and specific loss power (SLP). The particle with x = 0.2 exhibited the best magnetic hyperthermia performance among all compositions, owing to its strongest heating capability in external and alternating magnetic fields since it reached new maxima both for specific power losses (SLP) and intrinsic loss power (ILP). Therefore, it is concluded that the Zn-doped $CoFe_2O_4$ nanoparticles may represent ideal candidates for application on magnetic hyperthermia in anti-cancer treatment by generating effective cure temperatures with localized tumor ablation Zn-doped CoFe2O4f about 42–45 °C. The results show that zinc doping is an effective approach to tailoring the magnetic behavior of cobalt ferrite NPs for medical hyperthermia applications. However, additional investigations into biocompatibility, surface functionalization and in vivo performance are necessary before clinical application can be approbated.

Keywords:
Zn-doped $CoFe_2O_4$, Magnetic hyperthermia, Nanoparticles, Saturation magnetization, Specific loss power, Cancer therapy.

Introduction

Nanotechnology is the design, production and application of structures, devices and systems by controlling shape and size at a nanometre scale (from 1 to 100 nm). Materials at this size scale have novel physical, chemical and biological behaviors that are not observed in the corresponding bulk material. These are due to the enhanced surface area-to-volume ratio and quantum effects that become appreciable at the nanoscale. Nanotechnology merges the different scientific fields, such as physics, chemistry, biology, and materials science in order to create nanoscale systems for multifunctional applications. One of the burgeoning fields involving these nanostructures is that mixed nanoparticles, nanomaterials and nanostructures exhibit extreme strength, electrical conductivity, and catalytic performance, which are significantly higher than the corresponding bulk material [1]. Such unique characteristics have prompted a variety of novel applications in electronics, energy storage, and biomedicine related to such nanomaterials [1-2].

Nanotechnology has the potential to reshape modern medicine, particularly diagnosis, treatment and drug delivery. Because they are at the nanoscale, nanoparticles can cross key biological barriers like cell membranes and blood–brain barrier; therefore, they have great potential for targeted drug delivery and enhanced imaging techniques. Iron oxide nanoparticles, which have attracted considerable interest as magnetic hyperthermia agents, can selectively kill cancer cells when they provide localized heating within an alternating magnetic field [3]. Additionally, nanomaterials can be engineered to target specific biological components which helps in early disease diagnosis and patient personalized therapies. By manipulating critical characteristics such as particle size, surface charge and surface functionalization, researchers can design nanocarriers that deliver therapy more precisely and effectively. The applications of nanotechnology in drug formulation also holds tremendous potentiality towards treatment of diseases like cancer to neuro degenerative diseases [4].

Magnetism is an inherent physical characterization originating from flowing electric charge and also the intrinsic magnetic dipole moments related to electron spin as well as orbital activity. It deals with how the forces of magnetism work, what attracts or repels and how magnetic fields interact with matter. In solid-state systems, magnetism originates from the direction and correlation of atomic magnetic moments that exist in a material under the influence of both internal exchange interactions and applied external electromagnetic fields. At macroscopic levels, the magnetic property of a material is generally referred through its magnetization (M), which in essence speaks to the value of magnetic dipole moment per volume [1]. It can be written as:

$$MV = \sum m_i \ldots\ldots\ldots\ldots (1)$$

Where V is the overall volume of material and $m_i$is the magnetic moment of each (i-th) ion or atom. Magnetism motives a very comprehensive variety of physical phenomena -from the possessions of materials up to schemes such as vending machineries and motors[1]. The five core categories of magnetism including Para magnetism, diamagnetism ferromagnetism, antiferromagnetism, and ferrimagnetism take their own features which determine how they act smooth-edged magnetic fields. So, let's discover these forms of magnetism, and the behaviors that they can be put to use in physics practical and theoretical applications. Indeed, magnetic nanoparticles inherently have unique magnetic and surface characteristics, and they are widely used in current technologies. And they are extensively used in biomedicine such as drug transporting, MR contrast enhancing and cancer hyperthermia. When used in environmental engineering, these materials can easily remove the pollutants from wastewater by magnetic separation method [1]. They are also excellent catalysts in chemical reactions because they have high surface area. In a word, the MPs are critical in medical, environmental and industrial fields.

Table 1: Types and Examples of Magnetism

Property	Diamagnetism	Para magnetism	Ferromagnetism	Antiferromagnetism	Ferrimagnetism
Examples	Bi, Si, Cu, inert gases	Al, O_2, MnBi	Fe, Ni, Co, Gd	Cr, MnO, FeO	Fe_3O_4, $MnFe_2O_4$, $NiFe_2O_4$
Susceptibility (χ)	Small, negative	Small, positive	Large (>100)	Small, positive	Large (>100)
Atomic Behavior	No magnetic moments	Randomly oriented moments	Parallel aligned domains	Antiparallel aligned moments	Unequal antiparallel moments
Magnetic Strength	Very weak repulsion	Weak attraction	Strong attraction	Weak (cancelled effect)	Moderate to strong
Magnetization (M vs H)	Slight decrease	Slight increase	Rapid increase, then saturation	Small increase	Rapid increase, then saturation
Temperature Effect	Almost constant	Increases with temperature	Changes near Curie temperature (Tc)	Changes near Néel temperature (TN)	Changes near Curie temperature (Tc)
Domain Structure	No domains	No domains	Strong domains present	Opposite domains cancel	Mixed unequal domains

Ferrites are ceramic materials with iron oxide and divalent or trivalent metal compounds, which generally crystallize in the spinel structure represented by the general formula MFe_2O_4. Their magnetism results from super-exchange between cations at tetrahedral and octahedral sites, supporting the fine tuning of saturation magnetization, coercivity: HC, and magnetic anisotropy through composition variation and nanoscale size adjustment [2]. Ferrite is a type of ceramic compound used in making electrical components, especially as a magnetic core material in transformers at high frequencies, and in the form of the mineral magnetite (Fe2O4). They are chemically unreactive, hard and it is also known that they have electric insulating properties. The most general chemical formula for many of these ferrites is MFe_2O_4, where "M" could be Mn^{2+}, Ni^{2+}, Zn^{2+} or Mg^{2+} [3, 4].

Choice of materials

Cobalt ferrite, CoFe2O4 nanoparticles are a few nanometer-sized magnetic material in the spinel family of ferrites and their size varies between 1-100 nm. They are cobalt (Co^{2+}) and iron (Fe^{3+}) oxides with cubic crystal structure. Since $CoFe_2O_4$ nanoparticles are superparamagnetic and small, it is extensively explored for magnetic, electronic and biomedical applications including magnetics, electronics, biomedicine, sensors and catalytical applications. Also, cobalt ferrite (CoFe2O4) nanoparticles are a class of hard-magnetic ferrites with high coercivity, strong magneto crystalline anisotropy and size-dependent ferrimagnetism that may exhibit superparamagnetic properties at small sizes. The inverse spinel's have a cubic structure and the useful characteristics of high chemical as well as thermal stability, whereas, due to their nano-size they suffer from lattice strain and surface effect. Moreover, $CoFe_2O_4$ nanoparticles are characterized by a high electrical resistivity and semiconducting behavior, which allows for their advanced use in magnetic and electronic devices as well as catalyst support [2].

Zn doping: Zinc doping in cobalt ferrite ($CoFe_2O_4$, abbreviated as Zn-doped $CoFe_2O_4$) is defined by the replacement of Fe^{3+} or Co^{2+} with equivalent Zn^{2+} ions at octahedral [B] sites of the spinel structure with a site preference for tetrahedral [A] sites. The cation distribution drives the Fe^{3} ions further into octahedral (B) sites, altering A–B super exchange interactions and resulting generally in lower coercivity but increased saturation magnetization at low Zn composition[5], therefore for increasing Zn degree $CoFe_2O_4$ with greater variation becomes a softer magnetic material which shall also be interesting for high frequency and sensor applications[6].

Zn as an Appropriate Dopant in $CoFe_2O_4$ Ferrites: Zinc's (Zn^{2+}) non-magnetic properties, ionic size and high Favor into the A-site (tetrahedral) of the spinel structure creates a good base layered above cobalt ferrite ($CoFe_2O_4$) [6]. The occupancy of co-substituted such as Ti^{4} in the octahedral (B) sites tends to increase A–B super exchange interaction strength and interatomic interaction, and thereby increases saturation magnetization at low content concentration but decreases its coercivity and magneto crystalline anisotropy [7, 8]. World Academy of Science, Engineering and Technology | 67 On top of that, enlargement in lattice as well as improved crystallinity is indicated by the influence of Zn^{2+}ions (having greater radius compared to Co^{2+}) but does not break stability of spinel phase. Besides, the decreasing manganic losses and gain in possibility to tune magnetic properties due to Zn-doping is exceptionally beneficial for APL. Moreover, the chemical inertness and biocompatibility of Zn also suggest its potential application in biological fields such as magnetic hyperthermia [9], where both temperature control-efficiency and minimized hysteresis field are issues.

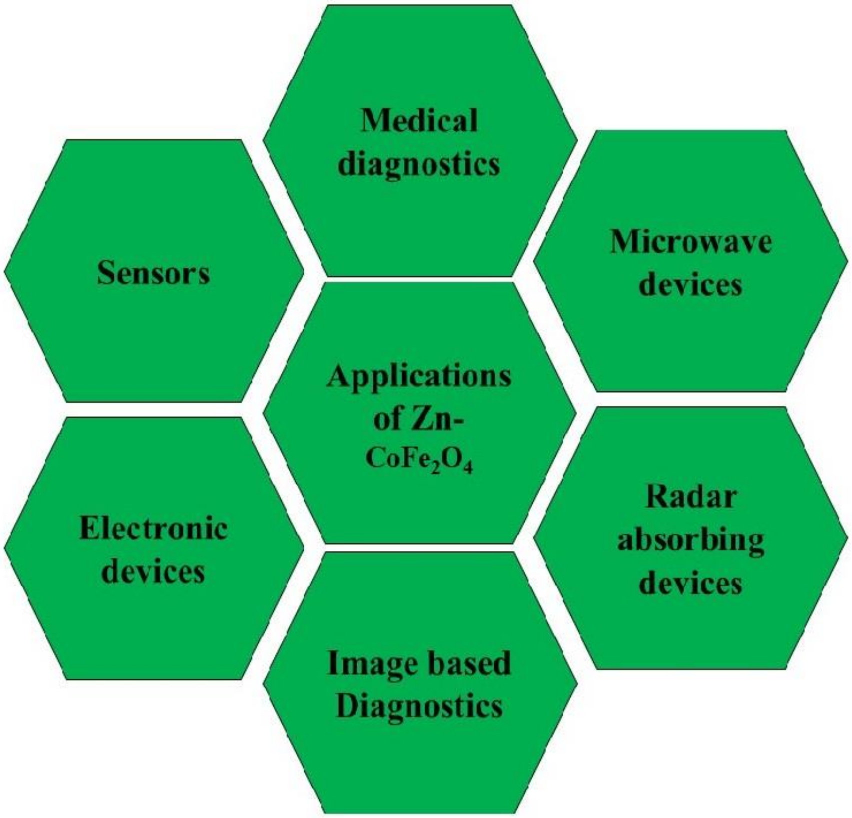

Figure 1: Applications of Zn-substituted $CoFe_2O_4$ [5].

Review of Literature

Here is a summary based on the above sentence: This review helps as an overview of scientific development, discoveries and information from numerous different studies related to cobalt ferrite ($CoFe_2O_4$) nanoparticles along with their amendments for usage in biomedicine. Due to its intriguing structural, magnetic and chemical properties, cobalt ferrite has been understood very extensively and is considered as a promising material in magnetic hyperthermia therapy. Unfortunately, pure $CoFe_2O_4$ usually suffers from high magnetic anisotropy and lower biocompatibility, limiting its direct applications in medicine. In order to address these issues, extensive research is being carried out in doping techniques. Zn-doped $CoFe_2O_4$ nanoparticles have been shown to improve heating efficiency, stability and lower toxicity for applications in targeted cancer therapy via hyperthermia. This chapter reviews the relevant literature on synthesis processes, structural and magnetic properties, as well as biomedical applications of CoFe2O4 and Zn-doped CoFe2O4 nanoparticles which serves as a basis for this research.

Andhare et al, prepared $Co_{1-x}Zn_xFe_2O_4$ (x = 0.0–1.0) nanoparticles through chemical co-precipitation followed by sintering at a temperature of 900 °C. XRD analysis confirmed the formation of a single-phase cubic spinel structure, while FTIR spectra verified the presence of the ferrite phase. SEM observations revealed spherical grains with agglomeration, and EDX confirmed the elemental composition of Co, Zn, Fe, and O. Optical analysis by UV–Vi's spectroscopy indicated a variation in the energy band gap with increasing Zn substitution. Magnetic measurements from M–H loops showed a decreasing trend in saturation magnetization, remanence, coercivity, magnetic moment, and M_r/M_s ratio with higher Zn^{2+} content, suggesting that zinc doping weakens the magnetic interactions in cobalt ferrite nanoparticles [10].

Jameela et al, synthesized zinc-doped cobalt ferrites ($CoFe_{2-x}Zn_xO_4$, x = 0.1–0.5) using the co-precipitation method to explore their structural, optical, and magnetic characteristics. XRD confirmed the formation of a single-phase cubic spinel structure with crystallite sizes ranging from 42–53 nm, while SEM micrographs revealed irregularly shaped particles. Based on FTIR spectral analysis, the presence of absorption bands between 300–600 cm^{-1} represent metal–oxygen vibrations at tetrahedral and octahedral sites. Strong absorbance in the 660–900 nm range indicates promising optical properties, as revealed by UV–Vi's analysis. In addition, the study revealed that higher concentrations of Zn improved the crystallinity of the cubic spinel phase, indicating that cobalt ferrites doped with Zn may exhibit promising structural and functional characteristics suitable for use in electrical devices [11].

Sharon et al. investigated the structural, dielectric and magnetic properties of zinc-substituted cobalt ferrite nanostructures ($Co_{1-x}Zn_xFe_2O_4$) fabricated via double sol–gel method. Rietveld refinement of XRD data confirmed the cubic spinel structure in the space group Fd-3m; crystallite sizes varied from 52 to 16 nm with increasing lattice parameters as a function of zinc doping. On the other hand, higher Zn concentrations resulted in reduced particle size, more dislocation density and increasing packing factor, due to further stability without any sacrifice of structural integrity having tolerance factor l0∼1.

Morphological and elemental composition were confirmed by SEM and EDAX, while the increase in bandgap energy from 2.95 to 4.33 eV was displayed through UV–Vi's analysis. High dielectric constant value suggests application in capacitors and gate dielectrics as also shown by dielectric studies. All samples presented ferromagnetic character based on magnetic characterization, with the highest saturation magnetization (73.72 emu/g) obtained for $Co_{0.5}Zn_{0.5}Fe_2O_4$, while $Co_{0.3}Zn_{0.7}Fe_2O_4$ and $ZnFe_2O_4$ were showed superparamagnetic behaviour [12].

Parus, Büdenbender, Subaric et al. $_4Co_{0.6-x}Mg_xFe_{1.9}La_{0.1}O_4$ (x = 0.0–0.6) nano ferrites (prepared via co-precipitation method, crystallite sizes: Sherrer:29.6–39.2 nm & Williamson–Hall:31.6–36.3 nm). Structural investigation ascertained the characteristics of cubic spinel phase and FTIR, Raman spectroscopy not only confirmed vibrational band due to metal–oxygen vibrations at tetrahedral and octahedral site. With increasing Mg^{2+} substitution studies the decrease of DC resistivity, activation energy, and bandgap were observed by electrical and optical measurements. With the substitution of the Mg^{2+} ions, a decrease in saturation magnetization, remanence and coercivity was inferred from magnetic measurements by parent $Zn_{0.4}Co_{0.6}Fe_{1.9}La_{0.1}O_4$ composition which has proved to be the most promising for microwave applications[13].

Pic siblings and co-authors discovered Zn-substituted cobalt ferrite (ZnxCo1−xFe2O4, x = 0.0–0.7) nanoparticles synthesized using the hydrothermal technique in order to characterize their structural, magnetic, and hyperthermia characteristics [74]. The XRD and Rietveld analysis confirmed the spinel structure with cation redistribution, while magnetic measurements showed a transition from ferrimagnetic to superparamagnetic behavior as Zn concentration increased. The x = 0.6 sample had the highest heating efficiency, with the specific and intrinsic loss power values within clinically safe limits. These results suggest the prospective applications of Zn-doped CoFe2O4, particularly that of Zn0. 6Co0[14].

Marsalin et al. prepared nanoclusters of Fe_3O_4 and $CoFe_2O_4$ using a modified solvothermal method intended for medical care, as well as energy storage applications. XRD and SEM confirmed phase purity and spherical morphology, whereas VSM established effective magnetic properties. Higher saturation magnetization (87.17 emu/g) and lower coercivity value (190 Oe) than $CoFe_2O_4$ which have reduced magnetization (59.12 emu/g), maintaining greater coercivity component 1132 Oe owing to increased estimated linear dimensions/magneto-crystalline/uniaxial anisotropies. Cobalt ferrite has been recognized as an important candidate for special advanced biomedical applications with its nanofibrous hierarchical structure and high magnetic property,[15] especially for the application in magnetic hyperthermia.

Synthesis protocol

In this article, experimental methods, instruments and techniques employed for the synthesis, characterization of Zn-doped $CoFe_2O_4$ nanoparticles will be discussed. Methods Although there are many studies of magnetic nanoparticles for biomedical applications, particularly indirect methods such as magnetic hyperthermia (mHT), it is critical that this work be reliable and reproducible. These methods provide control of the nanoparticle structure, composition, and magnetic properties for therapeutic studies[16]. This research work describes a chemical synthesis route, in which the selected synthetic strategy seeks to obtain uniformly sized nanoparticles with controlled Zn substitution and stable magnetic performances. Widely used wet-chemical approaches such as sol–gel, co-precipitation or hydrothermal synthesis grant fine control over dopant concentration and crystallinity, known to impact magnetic heating performance [17].

A variety of methods can be employed to synthesized nanomaterials, including combustion, microwave synthesis, gas phase synthesis, hydrothermal synthesis, solvothermal synthesis, thermal degradation, chemical vapor deposition, template synthesis, pulsed laser ablation, and the conventional sol-gel method [18].

Synthesis methods of nanomaterials: There are two main ways to make nanoparticles: the "top-down" method and the "bottom-up" method. These two strategies use different ideas to create nanoscale materials. Each one has its own set of benefits and drawbacks, and the best choice depends on what the material will be used for [19].

Two main approaches are used for the synthesis of nanomaterials.

- Bottom-up approaches.
- Top-down approaches

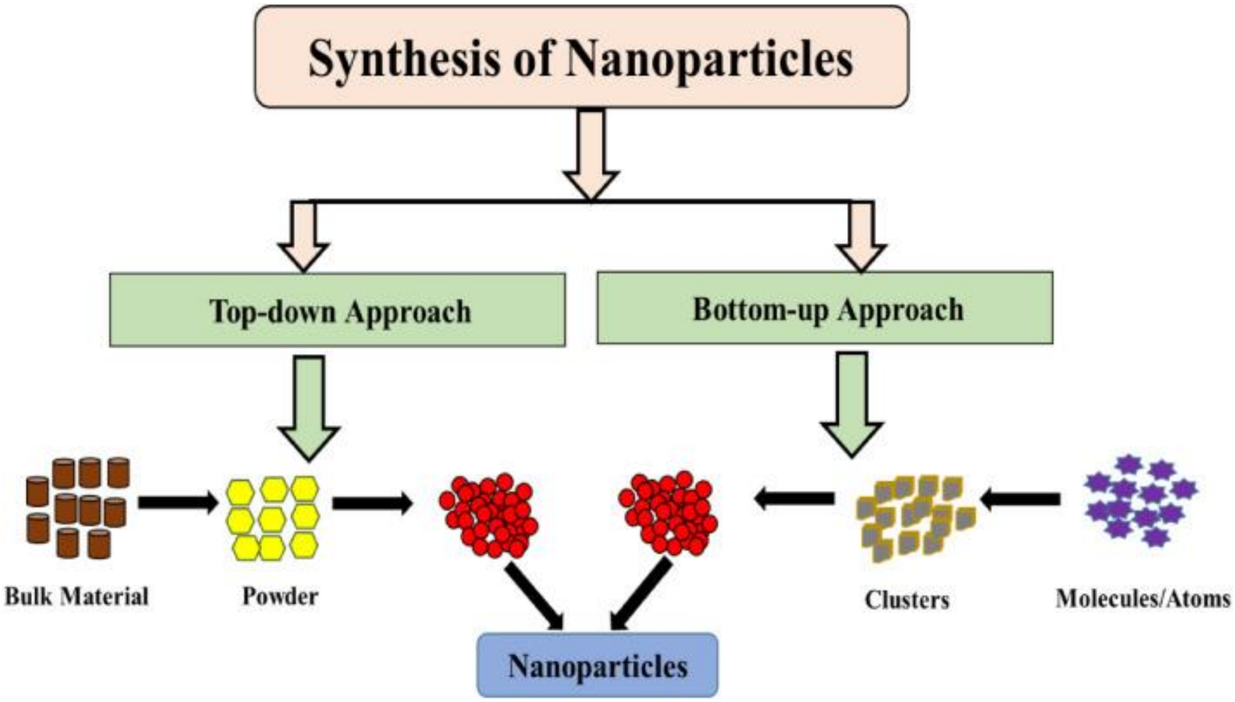

Figure 2: Synthesis of Nanomaterials [19]

Co-precipitation method: Another name for this wet chemical process is the solvent displacement technique. Ethanol non-solvent polymers are included. The polymer phase might be either natural or artificial [20]. When the polymer solvent mixes with the polymer solution, it instantly diffuses into a non-solvent polymer phase, producing nanoparticles. There are two steps involved in creating nanoparticles with interfacial tension. Co-precipitation, the most popular solution method for creating multi-component oxide ceramics, produces a "mixed" precipitate that are extracted from solution at the same time. In order to create a uniform solution with ion clusters, inorganic salts (such as nitrate, sulphate, etc.) are typically dissolved in distilled water. To make such salts precipitate as hydroxides, hydrous oxides, or oxalates, the solution is subsequently exposed to evaporation or pH change. Temperature, real pH, pH variation rate, and salt concentration each have an impact on crystal formation and accumulation. The solid mass is collected, cleaned, and heated to the medium's boiling point to gradually dry it out after precipitation. The washing and drying processes used for co-precipitated hydroxides must be taken into account when producing nano-sized powders since they influence the level of aggregation in the finished product. For the hydroxide to become a crystalline oxide, it must be calcined. This is frequently accomplished using calcinations or, more alluringly, a hydrothermal procedure [21].

Advantages of Co-precipitation Method includes consistency of constituent supply, cheap, High yield, High product purity, Slight reaction temperature, The deficiency of the requirement to use organic solvents and simply reproducible. Controlling the physical properties of the particles, such as their size, shape, and crystal structure, requires different techniques than controlling the preparation conditions that affect crystallization and growth [22]. For obtaining a high degree of homogenization, small particle size, and rapid reaction rates, co-precipitation has been observed as one of the finest production approaches. In addition to being cheap and easy to set up, the co-precipitation technique offers a large variety of precursors to choose from as starting materials, ranging from simple salts to intricate organic-inorganic combinations. In order to produce nanoparticles with a limited particle size dispersal, co-precipitation is one of the best means. The technique's primary advantage is the improved uniformity of the final nanoparticles, which results in comparatively high reactivity [23].

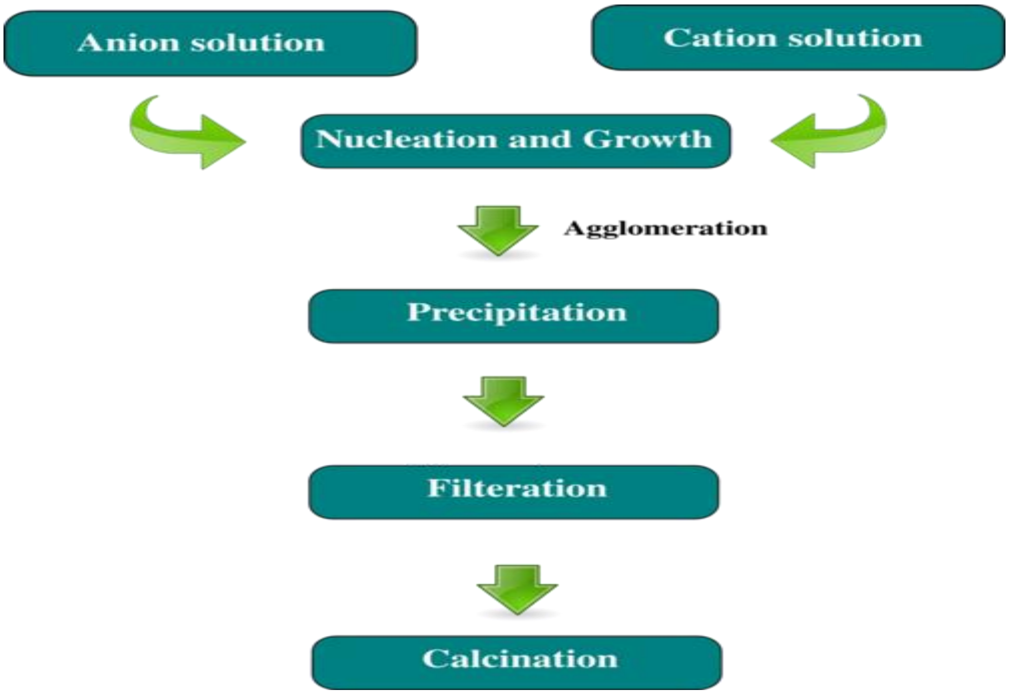

Figure 3: The co-precipitation method of nanoparticle synthesis [24]

Synthesis of Zn-doped $CoFe_2O_4$ nanomaterials: The synthesis of Zn-doped Cobalt Ferrites ($Co_{1-x}Zn_xFe_2O_4$) was carried out using the co-precipitation method to prepare samples with different Zn concentrations (x = 0, 0.1, 0.2, 0.3, 0.4, 0.5). To begin, stoichiometric amounts of cobalt nitrate ($Co(NO_3)_2 \cdot 6H_2O$), zinc nitrate ($Zn(NO_3)_2 \cdot 6H_2O$), and iron nitrate ($Fe(NO_3)_3 \cdot 9H_2O$) were separately dissolved in distilled water[14]. These solutions were then mixed and stirred intensely for one hour to achieve homogeneity. The mixed solution was heated to 80°C under constant stirring. Subsequently, a 4 M solution of NaOH was added dropwise in the required proportion, resulting in the formation of a black precipitate. This precipitate was washed several times with distilled water to remove any impurities [59]. After washing, the sample was heated at 100°C for 72 hours to dry the precipitate. The dried powder was then calcined at 800°C for 2 hours and allowed to cool slowly to room temperature [7].

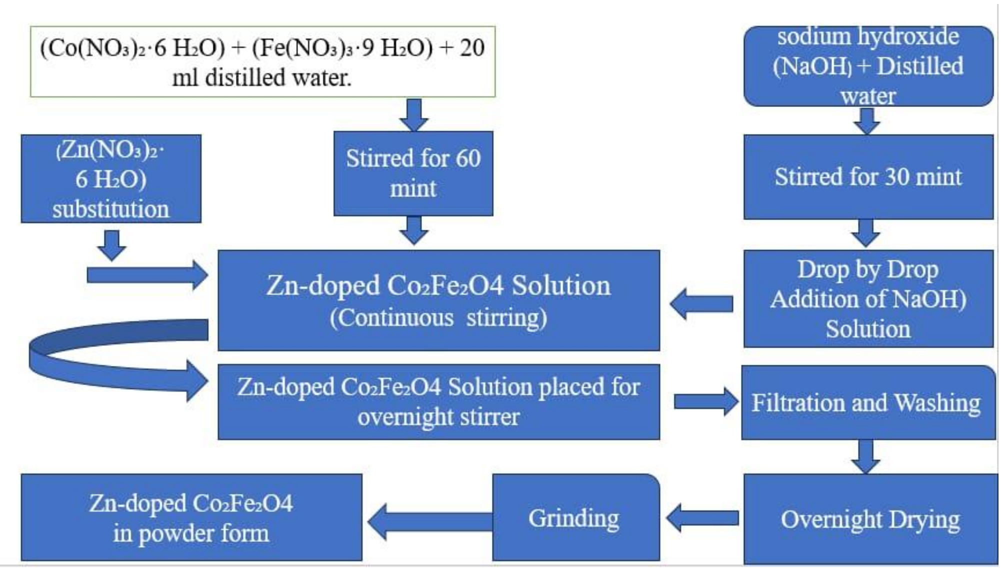

Figure 4: Flowchart of synthesis of Zn-doped $Co_2Fe_2O_4$ nanoparticles.

Characterization techniques for sample analysis: The resulting product was finely ground using the calcined material. To analyse structural, morphological and optical properties, they characterized the synthesized samples by XRD, SEM, FTIR, and UV-absorbance. Although their working principles differ, both techniques produce highly magnified images. Nanoparticle observation is made possible by these imaging techniques. Characteristics include composition, structure, and other characteristics like electrical, magnetic, chemical, and physical characteristics [25].

Results and discussion:

The FTIR spectrum of 1% Zn-doped $CoFe_2O_4$ nanoparticles shows absorption bands at 3740 and 3353 cm^{-1}, corresponding to O–H stretching vibrations of surface hydroxyl groups. The peak at 1623 cm^{-1} is attributed to the bending vibration of adsorbed water molecules. A band around 2361 cm^{-1} is assigned to atmospheric CO_2, while the absorption near 1051 cm^{-1} corresponds to metal–oxygen lattice vibrations associated with the ferrite structure. These results approve the formation of Zn-substituted cobalt ferrite with distinctive spinel characteristics. The characteristic absorption bands of the FTIR spectra are similar between pure $CoFe_2O_4$ and 1% Zn-doped $CoFe_2O_4$, suggesting that Zn substitution does not destroy spinel ferrite framework but varies certain lattice environments (which contribute to spectral shifts). For mutually samples, the broad band is observed at 3377–3353 cm^{-1} and less intense signals ranged between 3745 and 3739 cm^{-1} can be assigned to O–H stretching vibrations of surface hydroxyl groups signifying that ferrite nanoparticles also have adsorbed moisture. The absorption bands in the region of ~1621.5–1623.5 cm^{-1} are associated with bending vibrations of H–O–H groups of water molecules adsorbed to their intermolecular contacts [26].

Both samples display peaks at ~2363.5–2361.5, cm^{-1} which are often attributed to the adsorption of atmospheric CO_2 under these conditions. The Zn-doped sample shows an extra band at 2970 cm^{-1} which can be due to residual C–H stretching from organic type/precursors since they should have left after combining. This metal–oxygen band emerges at 1051 cm^{-1} (Zn-doped) and 1055 cm^{-1} (pure), and the small shift suggests that the introduction of Zn^{2+} changes the surrounding bonding between cations (Fe/Co) and O, supporting a redistribution of cations within the lattice as well as an exchange among octahedral versus tetrahedral sites in this framework. Overall, the striking similarity of spectra as well as intensity variation and a slight peak shift ensures a successful formation of Zn-doped cobalt ferrite with maintaining spinel structure.

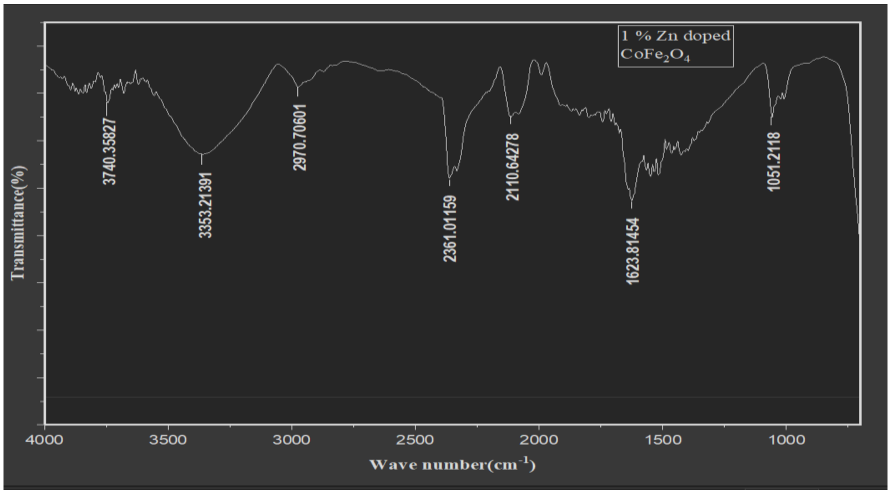

Figure 5: Zn-doped $CoFe_2O_4$

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