THE COMPARATIVE STUDY OF STRUCTURAL AND MAGNETIC PROPERTIES OF ZINC DOPED COBALT FERRITES

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DEPARTMENT OF PHYSICS

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THE COMPARATIVE STUDY OF STRUCTURAL AND MAGNETIC PROPERTIES OF ZINC DOPED COBALT FERRITES

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NOOR-UL-AIN

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RESEARCH COMPLETION CERTIFICATE

It is certified that **Ms. Noor-Ul-Ain** of BS (session 2018 – 2022), Department of Physics has carried out research work entitled **“The Comparative Study of Structural and Magnetic Properties of Zinc Doped Cobalt Ferrites”** under my supervision.

It is assured that research work is original and has not yet been published anywhere else.

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noor-ul-ain

Noor-Ul-Ain

ABSTRACT

Ferrite is a magnetic ceramic-like substance that can be used in a variety of electronic devices. Ferrites are polycrystalline and they are made up of many microscopic crystals they are tough, brittle, iron-rich, and often grey and black in color. They are made up of an iron oxide chemical combination and one or more other metals. Nanostructured cubic spinel ferrites with finely controlled nanostructures open up the possibility of developing nanomaterials with specialized characteristics for specific uses. In this research work the structural and magnetic properties of Zn doped Co ferrites were studied and compared. Firstly, the Zn doped Co ferrites prepared by sol-gel method were studied. X-ray diffraction of $Co_{1-x}Zn_xFe_2O_4$ ferrite nanoparticles with x = 0.0, 0.1, 0.2, and 0.3, confirms the single-phase cubic structure. Magnetic properties were investigated using the Vibrating Sample Magnetometer. The hysteresis curve of the samples reveals a considerable increase in magnetic properties. Also, the Zn-doped cobalt-ferrites with compositions of $CoFe_{2x}Zn_xO_4$ x=0.0, 0.1, 0.2, and 0.3, were synthesized by auto-combustion method. The XRD analysis of the prepared powders showed irregular shaped grains morphology, as well as small impurity phases. These ferrites are used in electronic industry in order to make components and it is also used in security systems.

“THE COMPARATIVE STUDY OF STRUCTURAL AND MAGNETIC PROPERTIES OF ZINC DOPED COBALT FERRITES”

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF ABBREVIATIONS

$CoFe_2O_4$	Cobalt Ferrites
Zn	Zinc
Co	Cobalt Ferrites
Fe_2O_4	Iron Oxide
XRD	X-ray Diffraction
EDX	Energy Dispersive X-Ray
HRSEM	High Resolution Scanning Electron Microscopy
FTIR	Fourier Transformed Infrared Spectroscopy
H	Magnetizing Force
H_c	Coercive Field
M	Magnetization
M_r	Remnant Magnetization
M_s	Saturation Magnetization
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
UV	Ultra Violet
VSM	Vibrating Sample Magnetometer
UV-Vis	Ultra Violet-Visible Spectroscopy
XRD	X-Ray Diffraction
MRI	Magnetic Resonance Imaging
EDAX	Energy Dispersive Spectroscopy
EDL	Electrical Double Layer
SiO_2	Silicon Dioxide
NPs	Nanoparticles

CHAPTER 1
INTRODUCTION

1.1 Introduction of Ferrites

Ferrite is a magnetic ceramic-like substance that can be used in a variety of electronic devices. Ferrites are polycrystalline that is, they are made up of many microscopic crystals they are tough, brittle, iron-rich, and often grey and black in color. They are made up of a chemical mixture of iron oxide and one or more additional metals. Ferrite is used in permanent magnets, computer memory elements and solid-state devices. Ferrites is also known as ferrate [1].

Figure 1.1 Ferrites [2]

1.1.1 Representation of Ferrites

The formula M(FexOy) defines a ferrite, where M is any metal that forms divalent bonds, such as any of the metals specified previously. For example, nickel ferrite is $NiFe_2O_4$ and $MnFe_2O_4$ is manganese ferrite [1].

1.1.2 Formation of Ferrites

A ferrite is formed by the reaction of iron oxide or rust reacts with a wide range of different metals to form ferrites, with any of the number of other metals, including aluminum, copper, magnesium, nickel, manganese, cobalt and even iron itself. A ferrite is a form of pure iron with a body-centered cubic crystal structure, occuring in low-carbon steel [1].

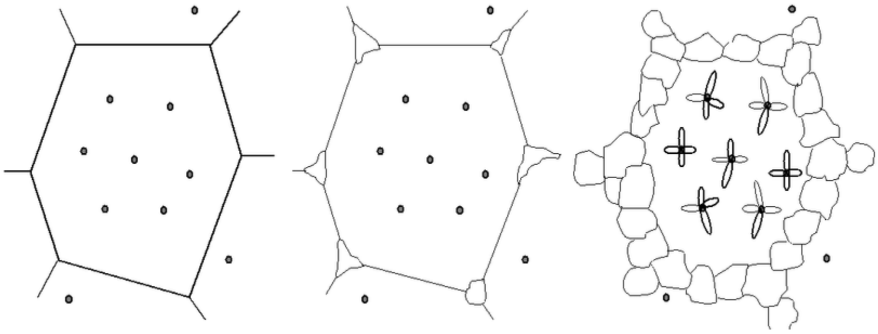

Figure 1.2 Formation of Ferrites [3]

1.1.3 Composition of Ferrites

Ferrites are usually the ferromagnetic ceramics compounds. Ferrites are derived from iron oxides. The most famous example of ferrites is magnetite (Fe_2O_4). Mostly ferrites are hard conductors of electricity.

The majority of ferrites have a spinel structure with the formula of AB_2O_4, in which A and B are distinct metal ionic bonds and iron is frequently present [4].

1.1.4 Structure of Ferrites

Spinel crystals have the inverted spinel structure instead of the regular spinel structure: B cations occupy 1/8th of the tetrahedral holes, 1/4th of the octahedral sites, and remaining fourth are engaged by B cations.

Only a few ferrites, such as strontium and barium ferrites, have a hexagonal crystal shape [4].

1.2 Magnetic properties of Ferrites

Magnetic characteristics of ferrites are categorized as follow:

- Soft Ferrites
- Semi Hard Ferrites
- Hard Ferrites

1.2.1 Soft Ferrites

Soft ferrites, which contain zinc, nickel, or manganese compounds, are commonly employed in electromagnetic or transformer cores. Coercivity is low in soft ferrites. The term low coercivity refers to the ability of a material's magnetization to quickly reverse without losing energy, but high resistivity refers to the material's ability to deal with eddy current in the core, which is another means of energy waste. Due to their small losses at higher frequencies, soft ferrites are commonly employed in RF transformers [4].

Figure 1.3 Soft Ferrites [5]

1.2.2 Semi-Hard Ferrites

Formula for a chemical $CoFe_2O_4$ ($CoO. Fe_2O_3$) is a semi-hard magnetic material that is in the middle between soft and hard magnetic materials. Majority of semi-hard materials are used in magneto strictive activitives i.e., actuators and sensors. The cobalt ferrites have the benefits of being infrequent that shows it is an excellent substitute for the Terfenol-D [4].

1.2.3 Hard Ferrites

Hard Ferrites is usually referred as permanent ferrites magnets, that contain high coercivity after magnetization. Hard ferrite magnets are generally made of barium and iron oxide or strontium carbonate. High coercivity is usually referred as the materials are more resistant to demagnetized. A permanent magnet must have a high coercivity as well as a high magnetic permeability [4].

Figure 1.4 Hard Ferrites [6]

1.3 Types of Ferrites

Ferrites are classified into four categories:

- Spinel Ferrites
- Hexagonal Ferrites
- Garnet Ferrites
- Ortho Ferrites

1.3.1 Spinel Ferrites

Ferrites of Spinel is a type of crystalline solid with magnetic characteristics that can be adjusted. $MgAl_2O_4$ is the oxide mineral that makes up spinel. In mineral sciences, the term "spinel" refers to a crystalline structure. And the word ‘ferrites’ are referred to as the occupancy of some sites in crystalline structure [7].

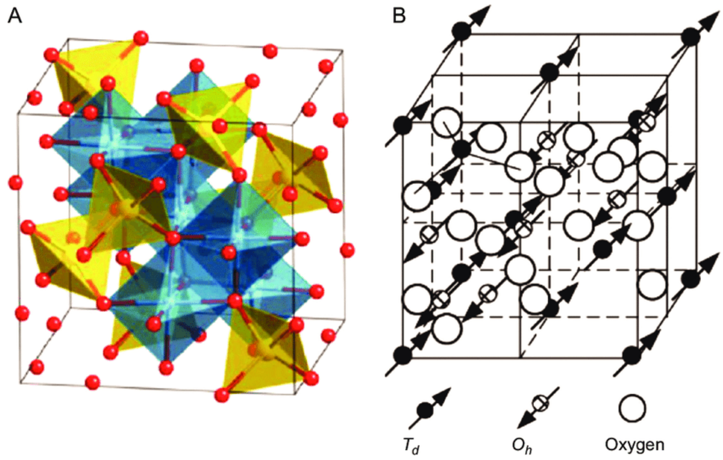

Figure 1.5 Spinel Ferrites [8]

A spinel ferrite has the general formula Me $^{[2+]}$ Fe $^{[3+]}{}_2$ O_4 where Me stands for other metallic cations (e.g., Co^{2+}, Zn^{2+}, Ni^{2+}, Mn^{2+}) [7].

1.3.2 Garnet Ferrites

Garnet ferrites have a crystal structure of garnet ferrites $Mn_3Al_2Si_3O_{12}$.

Garnet minerals become garnet ferrites when Al and Si are replaced with fe^{5+} ions and Mn is replaced with rare earth cation (R) then from this the garnet magnetic having general formula $R^{33+}Fe^{53+}O_{12}$. The Garnet ferrites have a body-centered cubic structure and it has eight formula units. Garnet ferrites crystal has cubic symmetry.

Garnet Ferrites consists of three sub-lattices in which 24 tetrahedral (A), 16 octahedral (B) and 24 dodecahedral (C) sites [9].

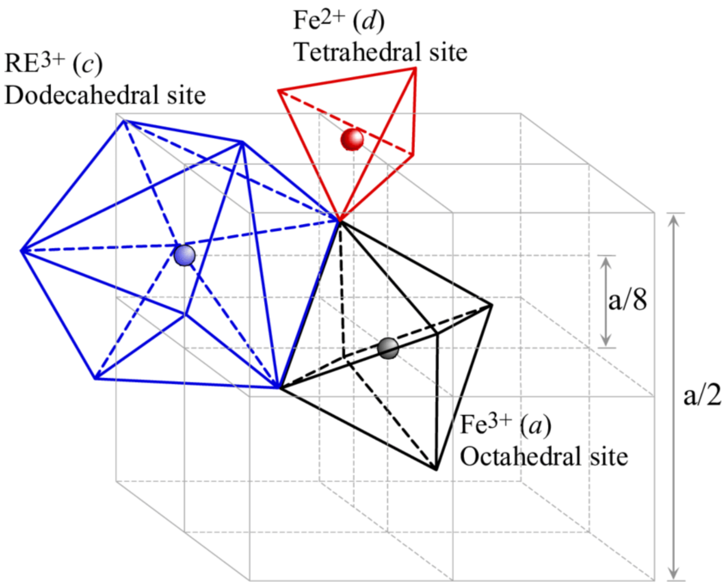

Figure 1.6 Garnet Ferrites [10]

1.3.3 Hexagonal Ferrites

Hexagonal ferrites are the type of permanent magnet with the general formula of $MF_{12}O_{19}$ where is the barium, strontium, calcium and lead. The hexagonal crystal structure is made up of oxygen ions having a close packing hexagonal arrangement unit cell, this unit cell contains two molecules of $MF_{12}O_{19}$. The coercivity of hexagonal ferrites and its magneto-crystalline anisotropy makes it permanent magnet. The crystal structure and chemical formula of hexagonal ferrites are divided into five different categories, i.e., M-type, W-type, Y-type, X-type, and Z-type with the chemical formula of $SrFe_{12}O_{19}$, $SrMe_2Fe_{16}O_{27}$, $SrMe_2Fe_{12}O_{22}$, Sr, Me, Fe_2sO_{46}, and $Sr_2Me_2Fe_{24}O_4$ respectively [9].

1.4 Applications of Ferrites

A ferromagnetic material has a wide range of applications. The hysteresis curve is quite important and plays an important role.

In transformers, electromagnets, and magnetic tape recording, ferromagnetism is used [11].

1.5 Magnetization

Magnetization is the density of magnetic dipole moments created in a magnetic material by proximity to a magnet [12].

1.5.1 Types of Magnetizations

- Diamagnetism
- Paramagnetism
- Ferromagnetism
- Antiferromagnetism
- Ferrimagnetism
- Superparamagnetism

Diamagnetism

The material that has very low or minimal magnetic effect is known as diamagnetism. It is occurred in the absence of any unpaired electrons [13].

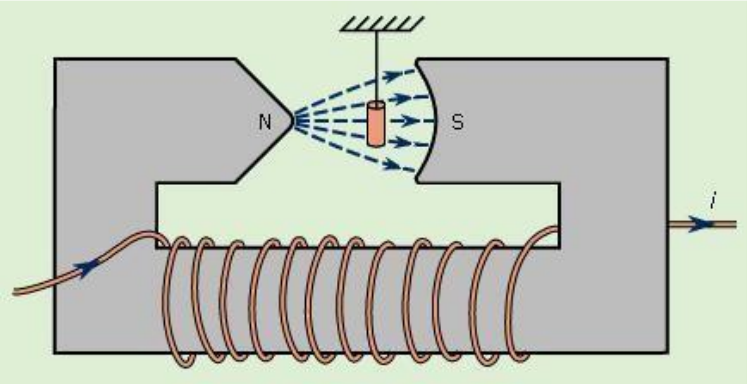

Figure 1.7 Diamagnetism [14]

Lenz's law also explains the meaning of diamagnetic. From this it is stated as "In the presence of external magnetic field the diamagnetic material gets induced dipoles. This is happened when the magnetic field and induced dipoles repel each other" [13].

Michael Faraday firstly discovered the diamagnets and the meaning of diamagnetic materials in 1845. He proved that the most of the elements in the modern-day periodic table are diamagnetic such as Gold, Silver and Copper etc. These di-magnetic elements cover majority of the elements in the periodic table, and the remaining have a lesser number of elements. Semi-conductor material are the best diamagnetic materials. Most of the diamagnetic materials have low and negative susceptibility [13].

Paramagnetism

Because of the presence of unpaired electrons in paramagnetic materials, the total magnetic moment of an atom's electrons does not equal zero. In this situation, the atomic dipole exists. The magnetization of paramagnetic materials is weak in the direction of the magnetising field this is due to by applying an atomic dipole and the external magnetic field that are arranged in the orientation of the external magnetic field [15].

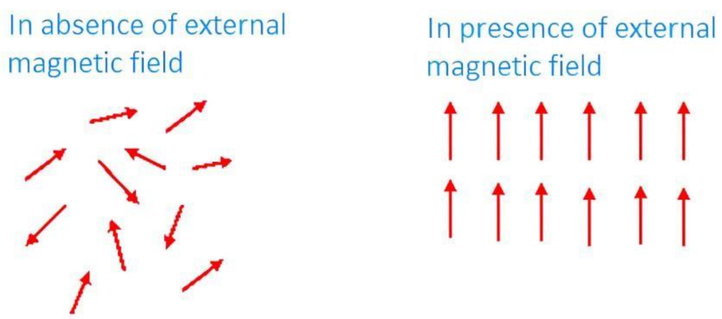

Figure 1.8 Paramagnetism [16]

The materials that have weak attraction of magnets is called paramagnetism. This mainly occurred because of an unpaired electron in the materials, or it is because of partially arrangement of uniformly aligned atomic dipole along the field. When the applied field is zero, no net magnetization is created as a result of the weak interaction between the magnetic moments. [15].

Curie's law governs this form of magnetism as, the susceptibility of magnetic X is inversely related to their temperature, that is mentioned [15].

It is represented as

M = XH = C/T x H

Where,

M is the magnetization, X is the magnetic susceptibility, C is the material-specific Curie constant, T is the absolute temperature in Kelvin and H is the auxiliary magnetic field.

Ferromagnetism

The materials in which certain materials like iron strongly attract each other in known as ferromagnetism. It is commonly used phenomena that is responsible for magnetism in magnets. The rare earth materials and gadolinium are mostly used in ferromagnets. Ferromagnetism occurred in ferromagnets and this ferromagnets need the net angular moment that is obtain from the orbital component of the spin component [17].

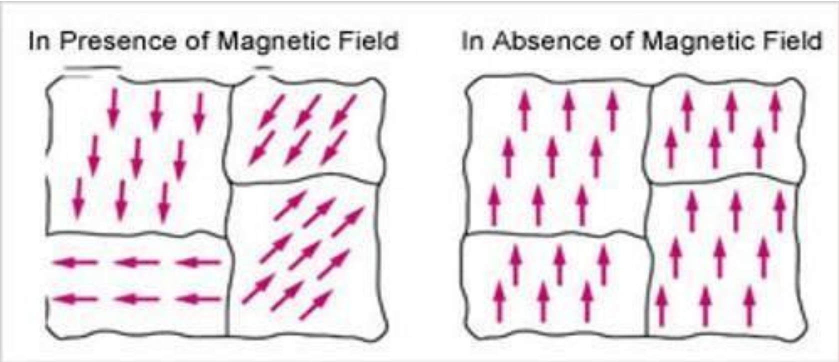

Figure 1.9 Ferromagnetism [18]

Ferromagnetism is used in electromagnets, transformers and magnetic tape recording. The hysteresis loop plays an important role in ferromagnetism [17].

Antiferromagnetism

The Magnetism in solids such as manganese oxide is known as antiferromagnetism and the manganese ions are Mn^{2+}. It will be spontaneously aligned themselves at relatively low temperature in to the direction opposed or arranged antiparallel. In antiferromagnetic materials, the magnetism of magnetic atoms or ions organised in one direction is wiped off by a set of magnetic atoms or ions arranged in the opposite direction [19].

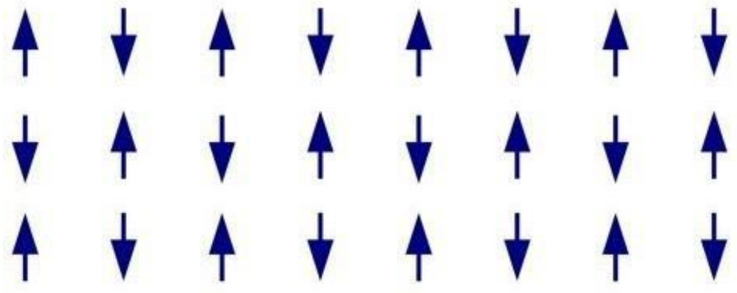

Figure 1.10 Antiferromagnetism [20]

The antiferromagnetic solids behave differently in the presence of magnetic field that depends on their temperature. At very low temperature no response exhibits to the external field of antiferromagnetic solids this is because of the antiparallel arrangements of atomic magnets in a rigid or solid material. This weak magnetism or alignment produce in rigid material where it reaches their highest point at Neel temperature. The term Neel temperature is defined as the atomic magnets' antiparallel coupling [19].

Ferrimagnetism

Ferrimagnetism is one of the types of permanent magnetism, mostly it is used in solids in which the magnetic field relate with the atoms on their own that arrange themselves spontaneously. Some of them are in same direction or in parallel [21].

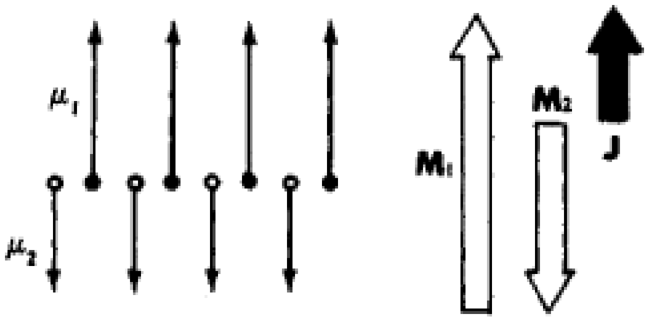

Figure 1.11 Ferrimagnetism [22]

Ferrimagnets and antiferromagnets are similar in that the exchange of magnetic ion bonding between adjacent magnetic ions results in an antiparallel arrangement of the confined moments. Because of this magnetization process, the magnetism of one lattice is larger than that of the oppositely orientated sublattice [21].

Superparamagnetism

A sort of magnetism that occurs between small ferromagnetic or ferrimagnetic nanoparticles is known as superparamagnetism. Under the effect of temperature, the magnetization of tiny nanoparticles can randomly shift or flip their direction. Neel relaxation time is the period between two flips. In the absence of an external magnetic field the time required to quantify nanoparticle magnetization is substantially longer than the Neel relaxation period. Average magnetization is zero in superparamagnetic state. The susceptibility to magnetic fields of super paramagnets is much greater than the paramagnets [23].

1.6 Zinc doped Cobalt Ferrites

The doping composition has a big impact on the qualities of Cobalt ferrite since it changes its chemical and physical properties. Zinc ferrites are a group of inorganic zinc and iron compounds ferrites with the universal formula $Zn_xFe_{3-x}O_4$. Spinel (Zn, Fe) Fe_2O_4 is a tan-colored solid that cannot be dissolved in acids, diluted alkali, or water. Because of their high opacity, zinc ferrites used as pigments, particularly in applications requiring heat stability. A Zinc ferrite produced from yellow iron oxide, for example, can be used as a substitute for usage above 350°F or 177°C, when zinc ferrite is put on high corrosion-resistant coatings, the corrosion resistance improves as the zinc ferrite concentration increases. According to a recent study, zinc ferrite, the paramagnetic in bulk, becomes ferrimagnetic in nanocrystalline in a thin film form. Controlling thin film growth conditions produced considerable ambient temperature magnetism and a narrow ferromagnetic resonance linewidth [24]. The size of the particles, as well as cation substitution and distribution, have a significant impact on the magnetic characteristics of $CoFe_2O_4$. The sol-gel auto combustion technique is the straightforward approach for synthesizing nano ferrites that speeds up the synthesis of complicated materials and improves crystal uniformity. The magnetic and structural properties of Zn^{2+} ions replaced cobalt ferrite, $Zn_xCo_{1-x}Fe_2O_4$ x = 0.0, 0.2, and 0.4, synthesized by sol gel auto-combustion technique are investigated in this paper [25].

1.7 Sol-Gel Method

The sol-gel method is used for making solid things out of tiny molecules. Metal oxide is manufactured using this process. A sol is a colloidal particle or polymer dispersion in a fluid that is stable. Amorphous or crystalline particles are possible. A gel is made up of a three-dimensional solid network that surrounds a liquid phase. In general, van der Waals forces or hydrogen bonds may be used to interact between the sol particles. However, most gel systems utilized for materials synthesis have a covalent connection, thus the gel process is irreversible [26].

Sol-gel is a method of forming a three-dimensional network from solid nanoparticles distributed in a liquid (a sol) that runs the length of the liquid (a gel) [26].

To make sol-gel, you simply combine components containing molecules capable of joining to produce bigger molecules and, finally, nanoparticles. After that, the nanoparticles connect to form a gel network. When nanoparticles are created and then spread in a liquid, gels become more advanced. Quantum dots and carbon nanotubes, for example, are created by a procedure and then dissolved in liquid or distributed with the use of a surfactant [26].

When the rigid nanoparticles scattered in a sol combine to create a particle network that spans the liquid, it becomes a gel. This necessitates the Temperature changes cause solid nanoparticles in liquid to bounce about in random ways. Because some nanoparticles possess reactive surface groups that condense together to create bonds, this is simple, nearly automated. The viscosity of a sol approaches infinity as it becomes a gel, and it eventually becomes immovable. Gelation is the process of transitioning from a sol to a gel [26].

The sol-gel process is a revolutionary approach to the development of new materials. This approach allows for more exact control of all solid-state synthesis processes [26].

Four phases are commonly involved in the sol-gel coating process. To make a sol, the appropriate colloidal particles are first distributed in a liquid. The coatings are then applied to the substrates by dipping, spinning the sol solution or spraying. After eliminating the stabilizing components, the particles in the sol are polymerized, resulting in a gel with a continuous network. Following that, the residual organic or inorganic elements were pyrolyzed, resulting in an amorphous or crystalline coating [26].

1.7.1 Advantages of Sol-Gel Method

- It operates at low temperature.
- It is very cheap.
- It obtained a very thin layer of metal oxide.
- It is capable of readily shaping materials in a gel condition.

1.7.2 Disadvantages of Sol-Gel Method

- Sol-Gel techniques have some limitations because of this it can't arrives at its full industrial potential.
- It has weak bonding
- It has low-wear resistance
- It has high permeability

RATIONALE

The purpose of this research is to study the effects of zinc doped cobalt ferrites. Structural and magnetic characteristics of zinc doping on cobalt ferrites will be explored in this work.

OBJECTIVES

Following are the objectives of this study:

- To understand the methods used for preparation of Zn doped Co ferrites.
- To investigate the structural properties of Zn doped Cobalt ferrites.
- To investigate the magnetic properties of Zn doped Co ferrites.

CHAPTER 2

LITERATURE REVIEW

C. Joseph Prabagar et al. (2021) investigated the sol-gel auto combustion process used for making metal substituted cobalt ferrites nano crystallites with the configuration of $Co_{1-x}M_xFe_2O_4$ (M = Zn, Cu, Ag; x = 0.2) the crystal structure was determined using X-ray diffraction. morphology, magnetic characteristics of elemental composition of the samples. XRD findings validate the material's single phase cubic spinel structure. X-ray, EDX, FTIR, HRSEM were used for analysis of the sample. The particles ranging in size from 13nm to 19nm. The two different techniques of Williamson-Hall and Scherer were used to determine the size of crystallites and the amount of strain in a sample. Metal-oxygen stretching brands were discovered in the FTIR spectra. Transition metal substituted cobalt's antibacterial properties. The effectiveness of nano ferrites against the bacteria was evaluated qualitatively using well diffusion test. Antibacterial activity compared to substituents like Zn and Ag that could be used in drug delivery system in a variety of different applications of biotechnology and biomedical [27].

R. Alange et al. (2021) the sol-gel auto combustion method was utilized to create Zn doped spinel Co ferrites nanoparticles with formula $Zn_xCo_{1-x}Fe_2O_4$, where x = 0.2, 0.4. X-ray diffraction and a pulse field hysteresis loop tracer were used to explore the structural and magnetic characteristics. When Zn^{2+} levels rose, the average crystallite size (D) decreases from 32.15nm to 24.31nm. Magnetization field loops showed ferromagnetic activity, with a smaller hysteresis loop as the Zn content increased. Although coercivity (H_C) and remanent magnetization (M_r) decrease, that was linked to preferred site occupancy and a change in average crystallite size, saturation magnetization (M_s) declines, then increases as Zn^{2+} concentration increases [28].

M. Jameel et al. (2021) investigated that the nanoparticles made of cobalt ferrites were suitable for optical, electrical and magnetic applications in electrical appliances. Using zinc and Co-nitrate as precursors, a co-precipitation approach was employed to create Zinc doped Cobalt ferrites with the composition of $CoFe_{2-x}Zn_xO_4$ (x = 0.1-0.5) at varied percentages. XRD was used to examine the crystal phase configuration, hkl planes, volume, average crystalline size, and lattice parameters of the unit cell of $CoFe_{2-x}Zn_xO_4$ doped nanoparticles (XRD). The as-synthesized powders contained nano crystallite size of 42nm to 53nm a cubic spinel phase with irregularly structure, according to X-ray spectra analysis, UV visible spectra, FTIR spectra and SEM investigations. Calcination of the precursor at a low temperature 80 degree Celsius followed by sintering 500 degree Celsius for 3 hours, in a single-phase cubic spinel structure with the particle size of 42nm to 53nm. An increase in zinc concentration in cobalt ferrites increased the crystalline behaviour of the cubic spinel phase, as evidenced by sharp peaks and intense diffraction characteristics in the XRD pattern. The absorption band at low frequency is visible in cubical structure ferrites crystal lattices above 400cm-1 of the Fourier transform. The UV spectra absorbance data revealed that the Zn doped Co ferrite specimen has significant absorbance in the 600 nm to 900 nm wavelength range of visible zone. The high strain derivative of zinc doped cobalt ferrite suggested that it could be a promising material for electrical devices [29].

Deepali D. Andhare et al. (2020) investigated that the Co-precipitation method was used to successfully manufacture zinc doped cobalt ferrites nanoparticles. XRD analysis of Zn doped Cobalt ferrites showed the single-phase cubic structure and studied the structural parameter and Zn concentration. Structural parameter increased with increasing Zinc concentration. FTIR spectra showed the ferrite phase, SEM showed the effect of spherical grains, EDX spectra showed the process of Cobalt Zinc ferrites. UV-Vis spectroscopy revealed a graph showing the fluctuation of the energy band gap in Cobalt ferrite with increasing Zinc content. M-H curve showed the value of magnetic parameter. Hysteresis loop also gives the comparison of Zinc ferrites and Cobalt ferrites. From this it was concluded that the magnetic properties go on decrease with increase in the substitution of zinc ferrites [30].

P. Vinosha et al. (2020) studied the characterization and synthesizing the Zinc doped Cobalt ferrite nanoparticles and studied the applications of Zn doped Co ferrites in technological and industrial field. This paper reported the characteristics of spinel ferrites and several procedures for producing Zinc doped Cobalt ferrites High magnetic, optical, electrical, and dielectric characteristics are found in zinc doped Cobalt ferrites. The characteristics of Zn doped Co ferrites involved a low power loss, permittivity, coreactivity, permeability and resistivity. In biomedical field, like MRI and cancer treatment are also used these ferrites. Zn doped Co ferrites also used in electronics field such as in transducers, bio sensors, sensors, and transformer. Zinc doped Cobalt ferrites have some advantages that are used in our daily life such as in electronics or electrical appliances [31].

P. Imanipour et al. (2020) after doping cobalt ferrites with divalent Sr^{2+} and Zn^{2+}, the structural and magnetic properties were investigated. For this aim, samples with the configuration $Co_{1-x-y}Sr_xZn_yFe_2O_4$ where x=0.01, 0.05, 0.3 and y=0.0, 0.05, 0.1, 0.4, 0.5, 0.7 were manufactured using the spontaneous gel auto combustion technique. Methodology (Pechini). The effect of dopants on average lattice size and crystallite/grain size This study examined the density of the generated phase, as well as the purity and form of the synthesized nanoparticles. The secondary phase of spinel ferrites was not validated, however spinel ferrites formation was confirmed in all samples. $SrFeO_{2.086}$ was discovered in samples containing a high quantity of Sr. There is also thermal DTA-TG analysis available [32].

M. Khan et al. (2020) investigated that the magnetic nanoparticles have garnered a lot of attention in the field of biomedicine. When the size of particles is reduced to a few nanometers, it can attain a single magnetic domain and showed excellent coercivity and magnetization. With a diameter of less than 30nm, it shows superparamagnetism, making it an appealing option for magnetic resonance imaging, biosensing, and targeted drug delivery applications. Furthermore, colloidal instability restricts its use. The introduction of bi-functional linkers not only stabilizes nanoparticles but also allows for one-step production. To further understand how the addition of bivalent cations impacts the chemisorption capabilities, we employed dopamine to functionalize magnetite, cobalt, and zinc ferrite nanoparticles in this work. Theoretical parameters and interaction spacing were initially calculated using the Fourier Transform Infrared. The addition of Zn^{2+}

alters its reactivity to amine groups. Various combinations of pseudopotential and relativistic treatment approaches were used to investigate the interfacial reactivity and electromagnetic characteristics of doped iron oxides [33].

M.Madhukara Naik et al. (2019) published a report on zinc doped cobalt ferrites generated by utilizing curd as a fuel by combustion process. nanoparticles were characterized by using XRD, SEM, with EDAX, TEM, FTIR, UV-DRS, and luminescence spectroscopy. The X-ray diffraction features reflected resulted in the creation of a cubic-spinel structure. Scanning Electron Microscopy and TEM techniques revealed the spherical shape of nanoparticles. Characteristics reflected in FTIR indicated the vibrational stretching modes. The decreasing intensity of cobalt ferrites indicates that the recombination rate of electron hole pairs that may decrease with increased Zn doping. The efficiency of Zn doping of cobalt ferrite photocatalytic activity may enhance up to x=0.4 and decrease at x=0.6 mole fraction, as evidenced by the luminescence spectrum, which displayed highest value corresponding to violet, blue, green, yellow, and orange emission. Zn doped Co ferrites nanoparticles are suited for optoelectronic, photocatalytic, and therapeutic applications [34].

G. Muscas et al. (2019) investigated the effect of Zn doped Co ferrites, the bulk properties at nanoscale showed the size and shape of each particle and its physical and chemical structure. This paper showed the substitution of Zinc to particles with same size, shape, and structure but different magnetic anisotropy. Also studied the comparison of strong chemical and physical stability of nanoparticles and tunable magnetic properties. Found the balance between magnetic behavior of each element of nanoparticles and its strong interaction. It provided an advance analysis of magnetic properties. The magnetic properties of Cobalt ferrites nanoparticles have several applications from electronics to biomedical ones. Magnetic anisotropy showed the effect of interparticle interaction. Zinc doped ferrites developed the material of specific magnetic properties [35].

K. Anu et al. (2019) investigated that the two-step approach is used to make steady nano fluids of zinc doped cobalt ferrites with the doped concentrations of x = 0, 0.1, 0.2, 0.3, and 0.4. Impurity phase of the ferrite was confirmed by using an XRD pattern, while the chemical composition as determined by using EDAX analysis. Magnetic examination indicated that the samples had a mono domain structure. Because of the creation of an electrical double layer (EDL) in the dispersion, the electrical conductivity of all the nanofluids was greater than that of water and the base fluid. At ambient temperature, $Zn_{0.2}Co_{0.8}Fe_2O_4$ displayed a 90% improvement in electrical conductivity over the base fluid due to greater surface charge and ionic potential. A percolation action of nanoparticles of ferrites in the base fluid was used to investigate the influence of temperature electrical conductivity of nano fluids. It should also be emphasized that the magnetic field had no effect on the creation of EDL, so the electrical conductivity had no change as a result of applied field. Experimental results were compared to Maxwell and Shen's models and then they determined that compactible within Shen's framework [36].

M. Mahmood et al. (2018) investigated that the erbium (Er^{3+}) was used to replace cobalt zinc ferrite with the general formula. The micro-emulsion technique was used to create $Co_{0.70}Zn_{0.30}Er_xFe_{2-x}O_4$(x = 0, 0.01, 0.02, 0.03, 0.04, 0.05). The Er substitution was carried out in order to tune the optical band gap. Nano composites material with low graphene oxide (rGO) content was created using $Co_{0.70}Zn_{0.30}Er_xFe_{2-x}O_4$ which was created through a simple chemical process. The primary goal of graphene was to improve ferrite particle conductivity. For structural analysis, X-ray diffraction, scanning electron microscopy, UV-Visible spectroscopy was used. The XRD analysis confirmed the single-phase spinel structure and by using Scherer formula crystallized size could be calculated and it varying between 35nm to 60nm. The presence of well-distributed ferrite particles among graphene sheets was confirmed b SEM. It also investigated the current-voltage measurements of nanoparticles ferrites and their graphene composites. These nanoparticles have a wide range of potential applications [37].

M. Atif et al. (2018) investigated that the use of sol-gel process, a series of Zn substituted Co ferrite materials, $Co_{1-x}Zn_xFe_2O_4$ where x = 0.006, were synthesized. The production of a pure phase cubic spinel structure in the produced samples was confirmed by XRD patterns. The distribution of cations between the Rietveld method refined the XRD patterns to estimate tetrahedral and octahedral sites. The saturation magnetization is greatest at x = 0.4, according to magnetic experiments. In compared to other replaced samples, sensitivity is attained for the x = 0.4. An observed changes in magnetic findings are explained in terms of cation site occupancies and decreased anisotropy of the processed materials, with microstructure playing a minor effect. Furthermore, the dielectric measurements confirmed that the value was reasonable. The dielectric constant is low at low frequencies, and the ac conductivity is low related to electron-hole compensation. The x = 0.4 composition is thought to have the best magnetic and dielectric characteristics [38].

F. Nayeem et al. (2017) studied that the auto combustion method was used to create zinc doped cobalt ferrites nano particles with the nominal composition of $Co_{1-x}Zn_xFe_2O_4$ x = 0, 0.1, 0.2, 0.3, 0.4, 0.5. Samples were also investigated at 9000C. The X-ray diffraction patterns were captured by using an X-ray diffractometer. The XRD results indicate material's nanocrystalline nature. The samples had a single-phase spinel cubic structure, according to the XRD spectrum. The dielectric loss and dielectric constant of nanocrystalline cobalt ferrites were examined as a function of frequency and temperature. Hioki manufacturing LCR Hi- Tester 3250 with a frequency range of 100Hz to 1 MHz was used to test Zn^{2+} content at room temperature. The current $Co_{1-x}Zn_xFe_2O_4$ nano ferrites' conduction mechanism has been explored [39].

M. Sundararajan et al. (2016) investigated that the photocatalytic destruction of rhodamine B under the visible light was achieved by using a nanostructured catalyst that is produced by microwave combustion. The doping of Zn^{2+} ions in the cobalt ferrite matrix was verified by the diffraction peak-shift towards lower angles. Crystallite size was determined using the Williamson-Hall technique. All of samples' Conduction band and valence band edges were calculated using optical exams. The Photoluminescence analysis at 330nm indicated the rate of electron-hole recombination as well as the presence of defects. A Fenton-like photocatalytic degradation of RhB using a pure cobalt ferrite system was validated at pH 2.0 by varying the period, hydrogen peroxide addition, and starting concentration. Furthermore, all zinc doped cobalt ferrite photodegradation was performed at the optimal RhB concentration of 6 mg/l under similar working conditions. Based on the parameters of, possible RhB deterioration routes are suggested [40].

M. Sundararajan et al. (2015) investigated the microwave combustion method was used to make zinc doped cobalt ferrites nanoparticles. The samples were analyzed by using XRD technique, X-ray analysis, FTIR, SEM, reflectance spectroscopy, vibrating spectroscopy and UV-visible diffuse. XRD patterns were used to show formation of the single phase. $CoFe_2O_4$ reverses the structure of spine in the absence of contaminants. Lattice parameter rising the Zn^{2+} proportion from 8.380 to 8.396. Size of average crystallize calculated using a Scherrer method with the wavelength ranging from 46.22nm to 30.79nm. The Composition of Zn, Co, and Fe acquired from (EDX) analysis [41].

C. Singh et al. (2015) investigated that the reverse micelle approach was used to successfully synthesis nano particles of visible light that responsive Zinc doped Cobalt ferrites with the formula of $Zn_xCo_{1-x}Fe_2O_4$ where x = 0.0, 0.2, 0.4, 0.6, 0.8, and 1.0. The Sodium dodecyl sulphate was used as a surfactant and a templating agent. Spectroscopy of powders employing E-ray Diffraction and FTIR was used to establish formation of ferrites. One of most distinguishing qualities of this product is its spherical form. The size of ferrites particles was determined using a high-resolution transmission electron microscope. Magnetic investigations led to the discovery of $CoFe_2O_4$'s ferromagnetic property. When the zinc concentration of nanoparticles was increased, they became superparamagnetic. When the zinc concentration of nanoparticles was increased, they became superparamagnetic. A number of studies were conducted to explore the photo-Fenton activity of ferrites. The Rhodamine B dye fades when exposed to visible light. The catalytic activity of enzyme increased as the zinc levels rise [42].

K. Nadeem et al. (2014) studied the utilization of the sol-gel process to produce silica-coated 30 wt% cobalt zinc ferrite nanoparticles $Co_{1-x}Zn_xFe_2O_4$, x = 0, 0.2, 0.3, 0.4, 0.5, and 1. A Silica acts as a spacer between nanoparticles, preventing them from clumping together. X-ray diffraction reveals the cubic spinel ferrite structure of nanoparticles with crystallite sizes ranging from 37 nm to 45 nm. Transform of Fourier the production of SiO_2 and spinel ferrites was detected by infrared spectroscopy. Scanning electron microscopy images of SiO_2 and spinel ferrite indicate that NPs are virtually spherical and non-agglomerated because of the presence of nanomagnetic Silicon dioxide surface coating, all of the investigations revealed that $Co_{1-x}Zn_xFe_2O_4$ had good structural and magnetic properties. Ferrite nanoparticles are highly impacted by in different Zn concentration regimes, the concentration of Zn and average crystallite size of NPs were measured [43].

A. Raut et al. (2014) investigated that the magnetic properties and structural morphology of the spinel ferrite system synthesized by sol-gel auto combustion method. Metal ion nitrates also investigated. The ratio 1:3 was set for metal nitrates to citric acid. Ferrite was sintered at 600 °C for 12 hours. Cubic spinel in a single phase XRD data confirmed the structure of Co-Zn nanoparticles. X-ray Diffraction, followed by SEM and FTIR, was used to calculate the average lattice constant (a), crystallite size (t), and other structural properties of Zn substituted Co ferrite nanoparticles. Sol-gel auto combustion method has been discovered to offer various benefits for the production of technologically important Cobalt-Zinc ferrite NPs. The current investigation clearly reveals the influence of the synthesis technique as well as the probable association between magnetic properties and sample microstructure. When non-magnetic Zn^{2+} content of cobalt ferrite nanoparticles increases, the n_B and Ms, and other magnetic properties fall. At room temperature, the squareness ratio of Co-ferrite was 1.096 [44].

V. Reddy et al. (2013) investigated that the unique auto combustion approach was used to make Zn doped Co ferrites with the composition of $Co_{1-x}Zn_xFe_2O_4$. Structural characteristics of Zn substitution ferrites were investigated using XRD. The powders were calcined for 3 hours at 800 degrees Celsius before being compacted into cylindrical pellets. The XRD spectrum indicated that solid-state sintering of the green pellets at 1300°C for 12 hrs that showed the result in a single phase cubic-spinel structure. A VSM used to examine the magnetic properties at ambient temperature (VSM). In a pulse field magnetometer, magnetoelastic characteristics were measured by using the strain gauge technique [45].

N. Somaiah et al. (2012) investigated that the materials that are based on cobalt ferrites are the excellent candidates for magneto chemical sensor because of the high sensitivity of applied magnetic field. The fractional composition of Zn doped Co ferrites were synthesized via auto synthesis. Co-nitrate, Fe-nitrate and Zn-nitrate are used as a catalyst in combustion technique. Nano crystalline were comprised with analysis of X-ray spectra or TEM. Minor impurity phases are present in the cubic-spinel phase which has asymmetrically shaped grains. Oxidation process 800 °C for 3 hours of catalyst or followed by sintered metal 1300 °C for 12 hours then the resulted in a single-phase spinel structure with mean particle size, in the range 0.0-0.3, the highest value of magnetostriction monotonically decreases with increasing zinc concentration. Fluctuation in piezomagnetic coefficient observed in the Zn substituted, that limited the anisotropy of the system is related to cobalt ferrites. The material with a high strain derivative could be promising material for stress sensor applications [46].

CHAPTER 3
MATERIALS AND METHODS

3.1 Zinc Doped Cobalt Ferrites Prepared by Sol-Gel Method

Mujasam Batoo, K. et al. used the materials i.e., cobalt nitrate Co $(NO_3)_2.4H_2O$ (99%), zinc nitrate Zn $(NO_3)_2.4H_2O$ (99%), ferric nitrate Fe $(NO_3)_3.9H_2O$ (99%), urea CH_4N_2O (99%) to prepare the Zn doped Co ferrites nanoparticles. In double distilled water, metal nitrates were dissolved. The colored solutions were then thoroughly combined on a magnetic stirrer while maintaining a constant temperature of 70 degrees Celsius until a thick gel was formed. Samples were then cooked for 36 hours at 200 degrees Celsius in an air oven before being ground for 1 hour with a mortar and pestle. The powder materials were then thermally treated in a furnace at 600 degrees Celsius for 6 hrs that maintain a heating and cooling rate of five degrees Celsius/min, proceeded 1 hour of grinding with mortar and pestle [47].

3.2 Zinc doped Cobalt Ferrites Prepared by Auto-Combustion Method

Somaiah, N. et al. prepared the power of Zinc doped Cobalt ferrites, glycine was used as the fuel to the materials Co $(NO_3)_2.6H_2O$, Fe $(NO_3).9H_2O$, and Zn $(NO_3)_2.6H_2O$. The powders were calcined for 3 hours at 800 °C to produce the requisite single phase of cubic-spinel. At a pressure of 200MPa, calcined powder was crushed into pellets with dimensions of 10 mm x 9 mm (length x diameter). The green pellets were sintering for 12 hours at 1300 degrees Fahrenheit. The X-ray Diffractometer was used to examine the as-prepared, calcined, and sintered ferrites. The sintered pellets were subjected to magnetic characterization. At ambient temperature, a vibrating sample magnetometer (VSM) in fields up to 2T is used (~298K) [46].

3.3 Techniques for Characterization

3.3.1 X-ray Diffraction Analysis

In materials research, X-ray diffraction analysis is a technique that determining the crystallographic structure of a substance. The XRD is used to measure X-ray intensity and scattering angles that exit the material. The principal use of X-ray diffraction analysis is to identify materials based on their diffraction patterns. In phase identification, the x-ray diffraction method provides information on how the ideal structure differs from the exact one. XRD is a non-destructive method for determining structural characteristics such as lattice parameter, strain, grain size, and phase composition, as well as detecting crystalline phases and orientation [48]. The XRD peaks are created by constructive participation of a mono chromatic beam of X-rays distributed at varied angles from each collection of lattice planes in a material. The atomic sites inside the lattice planes define the peak intensities. X-rays are electromagnetic energy waves, whereas crystals are periodic arrays of atoms. When incident X-rays collide with the electrons of crystal atoms, they disperse. The electron is the scatterer in this process, which is known as elastic scattering. A regular array of scatterers always creates the same orbicular waves. Because of destructive interference, these waves cancel each other out in most directions; but, in less obvious directions, they combine constructively [49]. Bragg's law is defined as,

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

Here, d is the interval between diffracting planes, θ is the incident angle, a n is the integer, a λ is the wavelength of beam.

The exact orientations resemble reflections, which are diffraction pattern locations. Electromagnetic waves impinging on a smooth array of scatterers produce X-ray diffraction patterns. X-ray diffraction pattern is created using X-rays because their wavelength is almost equivalent to gap, d, between the crystal surfaces of 1-100 angstroms [49].

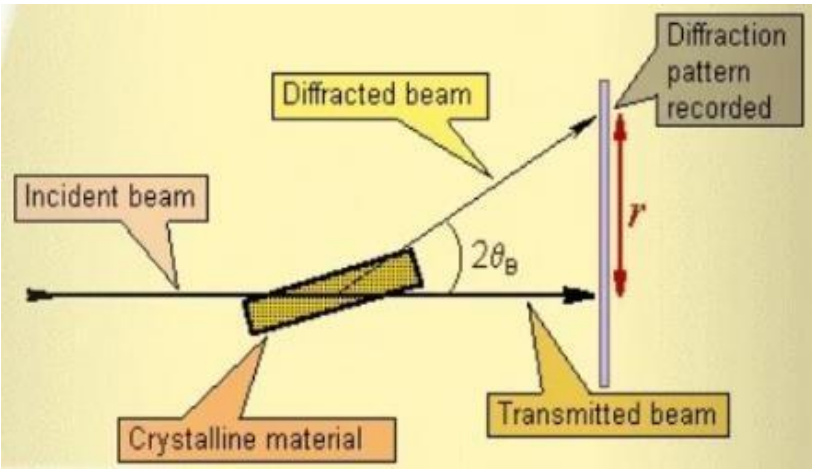

Figure 3.1 X-ray Diffraction [50]

3.3.2 Vibrating Sample Magnetometer

A vibrating sample magnetometer (VSM) is a laboratory tool that used to evaluate the magnetic properties of a substance. The vibrating component, which creates an electrical field in a coil, causes a change in the magnetic field of the sample. Vibrating sample magnetometer additionally contain a mechanism to hold the sample at the proper angle, a vibrating element, control unit, and a metre [51]. VSMs also enable researchers to examine a sample's magnetization from various angles, reducing the impact of extraneous influences. VSMs, on the other hand, are not well suited for determining the magnetization loop due to the sample's demagnetizing effects. Temperature dependence is another issue with VSMs [52].

3.3.3 M-H Curve

Magnetic hysteresis occurs when a ferromagnet, such as iron, is subjected to an external magnetic field and its atomic dipoles align with it. When it comes to creating and categorizing magnetic materials, the M-H curve is crucial. Magnetization is detected using the M-H hysteresis loop when a material is exposed to an applied changing magnetic field. Hysteresis is the term for the repeated occurrence of magnetization and demagnetization. H is the magnetizing force represented in Oersted (Oe = $(4\pi)^{-1}\times10^{3}$ A/m) and M is the mass magnetization expressed in magnetic moment per gramme (emu/g) [53].

Because the material has gotten magnetized, portion of the alignment will be retained after the field is withdrawn. Once the magnet is magnetized, then magnet will stay magnetized forever. Demagnetization is required either when heat is applied or a magnetic field in the opposite direction occurs. And this is the effect that provides a hard disc drive with its memory component [53].

In such materials, the relationship is not linear between field strength H and magnetization M. When a magnet is demagnetized then H = M = 0. And the relationship between H and M for increasing field strengths is displayed in a demagnetized magnet. M will return to its previous magnetization curve. This curve begins quickly, then progressively slows as it approaches magnetic saturation, M takes on a new shape, if the magnetic field falls monotonically. The magnetization is offset from the origin by an amount known as retentivity or remnant magnetization at zero field strength. The main loop is a hysteresis loop that arises from plotting the H-M relationship for all magnetic field strengths. The middle part's width along the H axis is doubled [53].

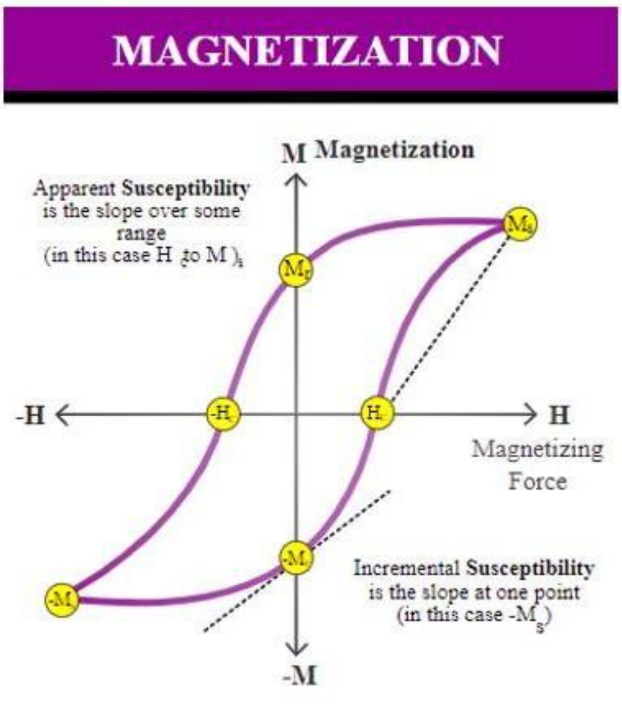

Figure 3.2 The M-H hysteresis curve [54]

CHAPTER 4

RESULTS AND DICUSSION

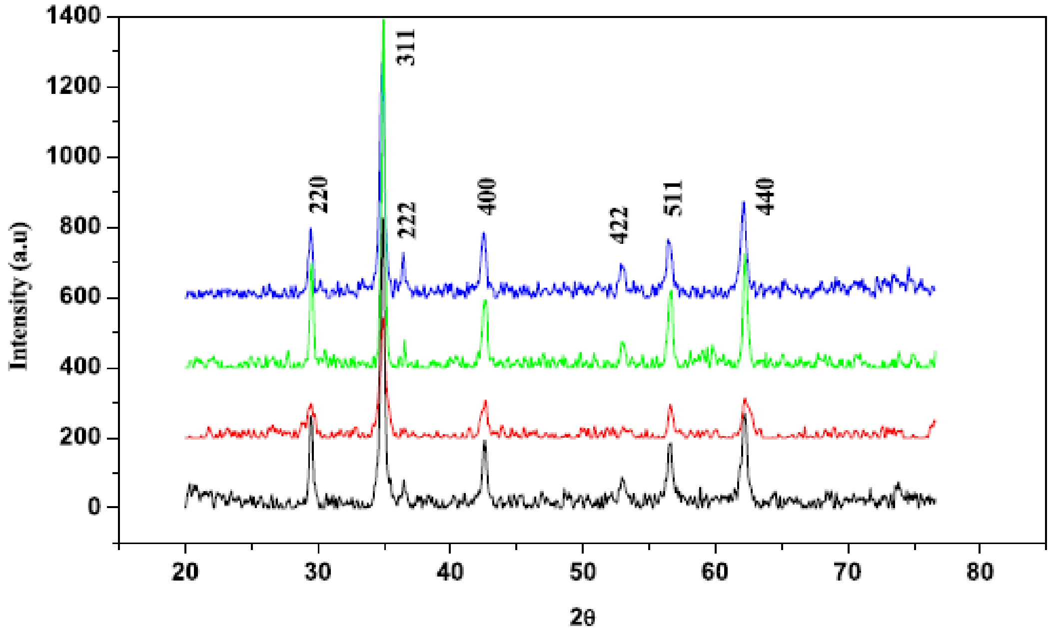

Figure 4.1 XRD Patterns of Zn doped Co Ferrites Nanoparticles Prepared by Sol-Gel Technique [47]

XRD patterns were shown in NPs sintered at 600 degrees Celsius. Powder X-software was used to evaluate the X-ray patterns, which confirmed the cubic spinel samples' nanocrystalline structure. Peak strength fell as Zn doping concentration increase, whereas peak broadening increased. The crystallite sizes for all compositions were approximated using the assumption that NPs were spherical in form (311). In the studies, the nanoparticle's average crystalline size was determined between the 55.38 and 32.87 nm. The structural and magnetic properties of Zn doped Co ferrites prepared by sol-gel method showed better results [47].

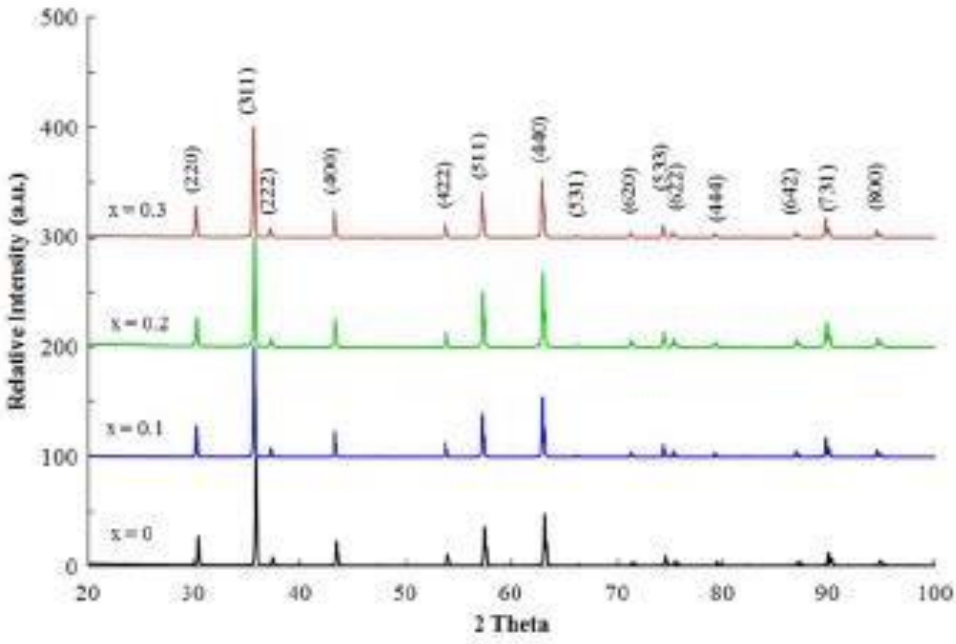

Figure 4.2 XRD Patterns of Zinc Doped Cobalt Ferrites Nanoparticles Prepared by Auto-Combustion Method [46]

The XRD spectra of sintered Zn doped Co-ferrites were sintered at 1300°C for 12 hours. There was no confusing reflections in the spectra, revealing a cubic spinel structure. Peak (311) depicted the crystalline size peak. The sharpest peaks were observed at (311). This particular plane represented dominancy of that plane in crystal structure sample. The size, shape, and internal stress of tiny crystalline regions were measured using XRD [46].

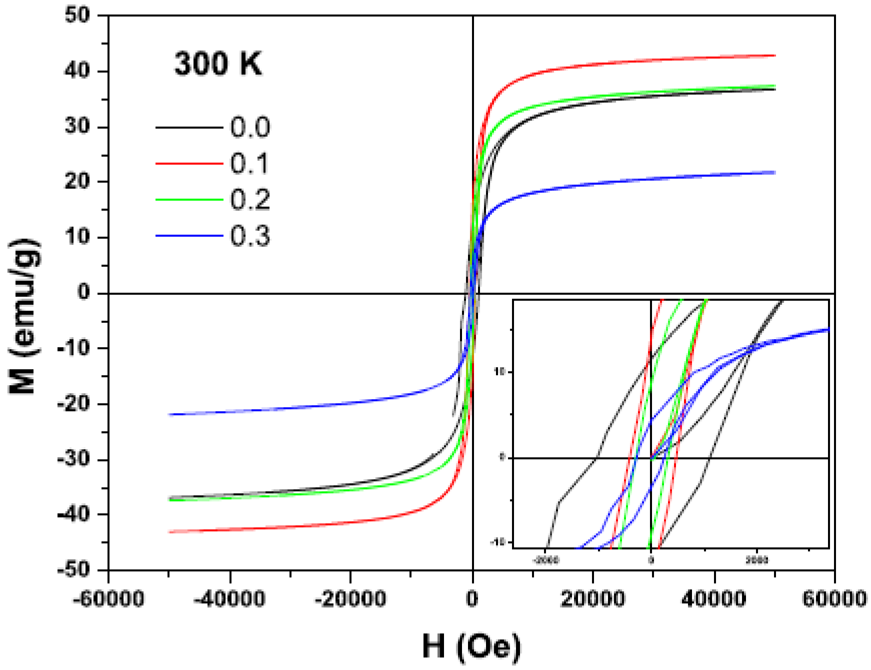

Figure 4.3 M-H loop of Zinc Doped Cobalt Ferrites Nanoparticles Prepared by Sol-Gel Method [47]

Magnetic hysteresis loops are responsible for spin ordering in magnetic materials. Magnetic hysteresis investigations were performed on the generated samples at 300K temperatures, confirming the similar hysteresis loops at the origin and emphasizing coercivity. Magnetic properties such saturation magnetization (M_S), remanence magnetization (M_r), coercivity (H_C), remanent ratio (R), and magnetic moment (B) were identified in all compositions at 300K. Because all of the $Co_{1-x}Zn_xFe_2O_4$ ferrites have 'S' shaped magnetic hysteresis loops, the magnetic measurements demonstrate that they are ferromagnetic. At 300 K, the sample showed narrow hysteresis loops, indicating modest ferromagnetism. M_S values decreased with increasing Zn content, from 36.8 emu/g for x = 0.0 to 21.9 emu/g for x = 0.3 at 300 K, with the exception of composition x = 0.1 (M_S = 43.12 emu/g). The magnetic moment, which increases initially before decreasing as the doping level increases by more than 10%, can explain the increase in saturation magnetization as Zn doping increases. At room temperature, size effects are expected to dominate nanomaterial magnetism, but size confinement, quantum effects, spin glass transition, and thermal dependence all contribute to magnetism at low temperatures. At the nanoscale, a nanoparticle should have quantized spin wave excitation, but at high temperatures, it should have greater energy levels with the continuous thermally excited spectrum [47].

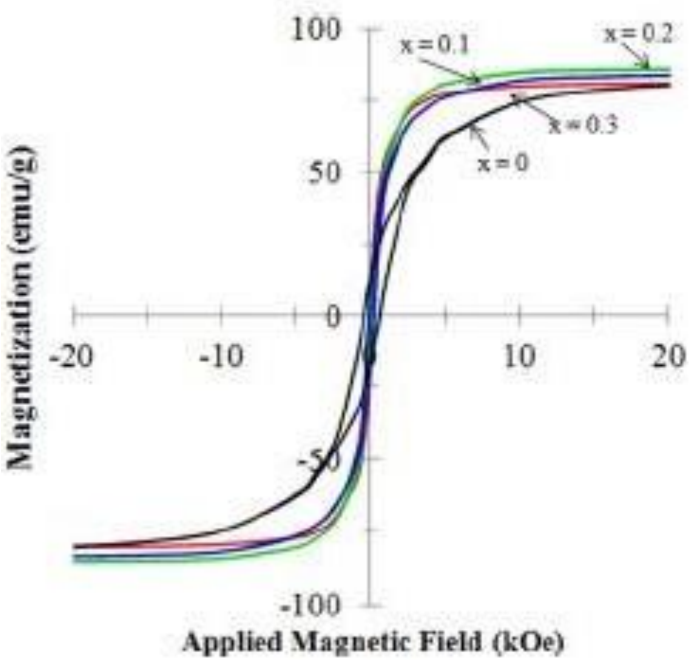

Figure 4.4 M-H Curve of Zinc Doped Cobalt Ferrites Nanoparticles Prepared by Auto-Combustion Method [46]

At ambient temperature (298 K), it illustrates the field dependence of magnetization M–H for Zn doped Co ferrites. The M_S value of the Cobalt ferrites sample is 82 emu/g, which is near to the saturation magnetization for Co ferrites. The M_S first increases with increasing Zn substitution from ~82 emu/g(x=0) to ~87 emu/g(x=0.2), but thereafter decreases with increasing Zinc concentration. Mixed ferrites exhibit a same shift in saturation magnetization when nonmagnetic Zn^{2+} added. The M_S grows by 3.3 percent from x=0-0.1, but only by 2.7 percent from x=0.1-0.2. The decrease in M_S percentage rise at x=0.1 suggests that Zn^{2+} preferentially goes to octahedral sites rather than tetrahedral sites. Coercivity (H_C) of cobalt ferrite is 525Oe in the present study, but it quickly drops to 165Oe for $CoFe_{1.9}Zn_{0.1}O_4$ followed by a constant decrease in H_C with each Zn concentration. Increased zinc concentration in cobalt ferrite is expected to reduce anisotropy while increasing coercivity [46].

CONCLUSION

The structural and magnetic properties of Zn doped Co ferrites prepared by sol-gel and auto-combustion method were studied and compared. The XRD patterns confirmed the cubic spinel structure of zinc doped cobalt ferrites which were prepared by sol-gel method. Structure of the sample was analyzed with XRD pattern and the magnetization was analyzed with M-H curve. The saturation magnetization, retentivity and coercivity decrease by increasing the Zn concentration. The sharpest peaks were observed at (311). This particular plane represents dominancy of this plane in crystal structure sample. Whereas in auto-combustion method, minor impurity phases of nanostructured cubic spinel phase were obtained from XRD analysis and the saturation magnetization, retentivity and coercivity decrease with the increase of Zn concentration. The structural and magnetic properties of Zn doped Co ferrites prepared by sol-gel method showed better results.

LIMITATIONS

- The lab resources were limited.
- It was difficult to collect all the information online as some of the articles had no public access.

RECOMMENDATIONS

- At the nanoscale, ferrites act as magnetic materials, which can be used in a variety of practical applications where data storage requires magnetism.
- Hard magnets can be employed in equipment like magnetic recording devices that need to store data for a long time. Soft magnets can be found in everyday items like transformers, motors, and electromagnets.
- Changing the doping concentrations of ferrites can help advance the field of magnetic devices.

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