THE COMPARATIVE STUDY OF MAGNETIC PROPERTIES OF ZINC DOPED NICKEL FERRITES AND COPPER DOPED NICKEL FERRITES PREPARED BY SOL-GEL METHOD

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LAHORE, PAKISTAN

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BY

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DEPARTMENT OF PHYSICS
KINNAIRD COLLEGE FOR WOMEN, LAHORE
2023

RESEARCH COMPLETION CERTIFICATE

It is certified that **Ms. Affaf Amir** of BS (session 2019 – 2023), Department of Physics has carried out research work entitled **"The Comparative Study of Magnetic Properties of Zinc Doped Nickel Ferrites and Copper Doped Nickel Ferrites prepared by Sol-Gel Method"** under my supervision.

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

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ABSTRACT

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 formed by the combination of iron oxide with one or more metals. Magnetization refers to the concentration of magnetic dipole moment that appears in a magnetic substance when it is in proximity to a magnet. This research work includes the study and comparison of magnetic properties of Zn doped Ni ferrites and Cu doped Ni ferrites prepared using sol-gel method. At first Zn doped Ni ferrite samples were considered. Magnetic traits using vibrating sample magnetometer were inspected. Zn content with x=0, 0.3, 0.5, 0.7 and 1 were reviewed for magnetic parameters. Later, Cu doped Ni ferrite with values ranging from x= 0, 0.5, 1 were plotted. The M-H hysteresis curves of the samples reveal substantial increase in saturation and coercivity. These ferrites are utilized in implementation in engineering to make systems and improve their function.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

Zn	Zinc
Cu	Copper
Ni	Nickel
$NiFe_2O_4$	Nickel ferrites
$MnFe_2O_4$	Manganese ferrites
Fe_2O_4	Iron Oxide
XRD	X-ray Diffraction
H	Magnetizing Force
H_c	Coercive Field
M	Magnetization
M_r	Remnant Magnetization
M_s	Saturation Magnetization
SEM	Scanning Electron Microscopy
VSM	Vibrating Sample Magnetometer
UV	Ultra Violet
UV-Vis	Ultra Violet-Visible Spectroscopy
EDAX	Energy Dispersive Spectroscopy
DC	Direct Current
AC	Alternating Current
FTIR	Fourier Transformed Infrared Spectroscopy
HRTEM	High Resolution Transmission Electron Microscopy

CHAPTER 1
INTRODUCTION

1.1 Introduction to 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 formed by the combination of iron oxide with one or more metals. Ferrite is used inside permanent magnets, computer memory elements and solid-state devices. Ferrites are also known as ferrate. Dominant properties of ferrite material contain proportionally minimal conductivity, small eddy current, drop in dielectric and permeability with high levels [1].

Figure 1.1 Ferrites [2]

1.1.1 Representation of Ferrites

Ferrites are denoted by a term which is M (Fe_xO_y). M refers to any metal that is adapted to form divalent bonds, including any of the metals mentioned accordingly. For example, nickel ferrite is $NiFe_2O_4$ and $MnFe_2O_4$ is manganese ferrite [1].

1.1.2 Formation of Ferrites

Production of ferrites consists of when iron oxide or rust reacts with various metals, considering aluminum, copper, magnesium, nickel, manganese, cobalt and

even iron. These ferrites incorporate pure iron with body-centered cubic crystal structure and are commonly found in low-carbon steel [1].

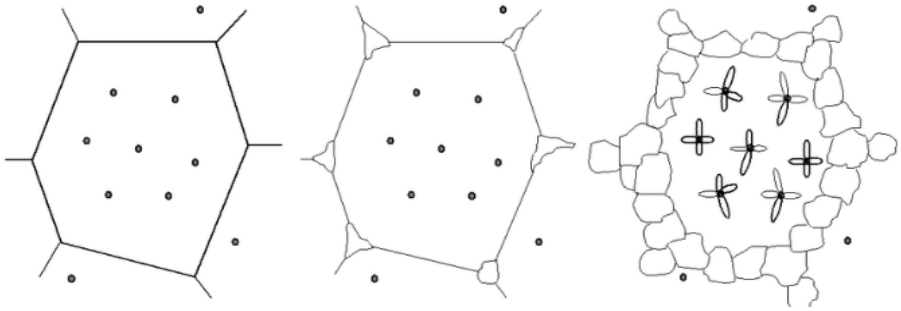

Figure 1.2 Formations of Ferrites [3]

1.1.3 Composition of Ferrites

Ferrites are ceramic compounds that express ferromagnetic properties and are attained out of iron oxides. Magnetite (Fe_3O_4) is a well-known example of a ferrite. Likewise most ceramics, ferrites are brittle, hard and poor conductors of electricity. Numerous ferrites have a spinel structure that is indicated by the formula AB_2O_4. In the formula different metal cations are presented by A and B together with iron (Fe). Typically, spinel ferrites have structural pattern of cubic close-packed (fcc) oxides (O^{2-}), with A cations occupying one-eighth of the tetrahedral holes and B cations occupying half of the octahedral holes, resulting in $A^{2+}B_2{}^{3+}O_4{}^{2-}$ [4][5].

1.2 Magnetic properties of Ferrites

Ferrites are categorized in the following ways dependent on their magnetic features:

- Soft Ferrites
- Semi-hard Ferrites
- Hard Ferrites

1.2.1 Soft Ferrites

Ferrites are commonly employed in the construction of electromagnetic or transformers cores and are composed of compounds containing zinc, nickel and/or manganese. These ferrites are classified as “soft” due to their low coercivity. This

property allows for the magnetization of the material to easily change position without eliminating excessive amounts of energy, described as hysteresis losses. Additionally, the high resistivity of the ferrite material serves to prevent eddy currents from forming within the core, which would otherwise result in energy loss. Owing to their minimal losses at higher frequencies, these subtle ferrites find immense use in RF transformers and inductor cores. Applications of such kind of ferrites contain loop stick and switched-mode power supplies antennas used in the AM radios [4].

Figure 1.3 Soft Ferrites [6]

1.2.2 Semi-hard Ferrites

$CoFe_2O_4$ ($CoOFe_2O_3$) also familiar by the name of cobalt ferrite, is a semi-hard magnetic material that manifest properties between those of soft and hard magnets. Its high saturation magnetostriction, which reaches 200ppm, makes it particularly useful in magnetostrictive applications such as sensors and actuators. Another advantage of cobalt ferrite is that it does not contain rare earth elements, making it a promising substitute Terfenol-D [5].

Figure 1.4 Semi-hard Ferrites [7]

1.2.3 Hard Ferrites

Permanent ferrite magnets, known for their high remanence and coercivity after magnetization, are typically composed of hard ferrites. The production of hard ferrite magnets involves utilizing materials such as barium and iron oxide or strontium carbonate. With their ability, to resist demagnetization, a permanent magnet must have this defining quality. Hard ferrite magnets have high coercivity. In addition, they exhibit a high magnetic permeability. Such apparently known ceramic magnets are affordable as well frequently employed at household items such as refrigerator magnets [4].

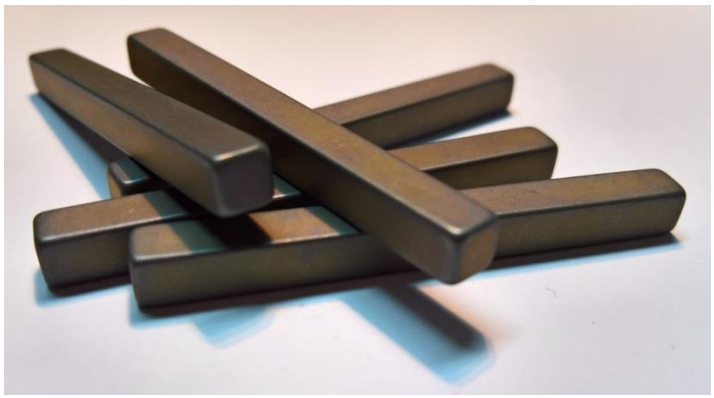

Figure 1.5 Hard Ferrites [8]

1.3 Types of Ferrites

Ferrites can be found in following varieties, based on their crystal structure:

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

1.3.1 Spinel Ferrites

Spinel ferrites are category of crystalline solids renowned due to their customizable magnetic characteristics. The name “spinel” originates from the naturally occurring oxide mineral $MgAl_2O_4$, but in material sciences, it is used more broadly to indicate a comparable crystalline structure found in other metallic

oxides. Spinel ferrites are described by the chemical formula MFe2O4 where M stands for other metallic cations (e.g. Co2+, Zn2+, Ni2+, Mn2+) [9].

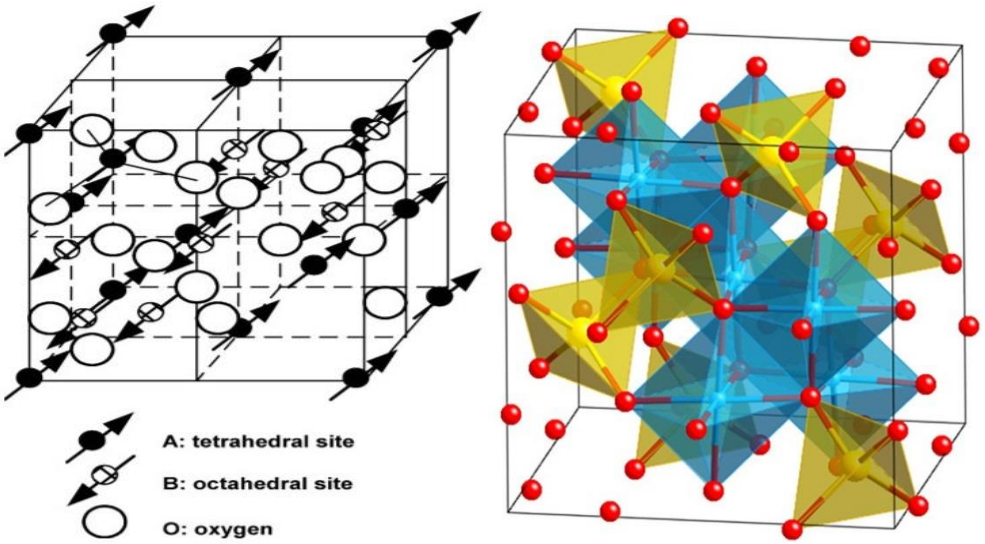

Figure 1.6 Spinel Ferrites [10]

1.3.2 Hexagonal Ferrites

Depending on group of permanent magnets with standard formula MFe_2O_{19} where M can be Ba, Sr, Ca, and Pb are hexagonal ferrites. Hexagonal ferrites have a crystal structure composed of oxygen ions arranged in a closed packed hexagonal pattern inside a unit cell that accommodates two $MFe_{12}O_{19}$ molecules. The high magneto-crystalline and coercivity exhibited by hexagonal ferrites enables them to be utilized in the formation of permanent magnets. These materials, which make up the majority of all magnetic materials produced globally and are now of enormous commercial and technological significance, possess plenty of various applications and functions [11].

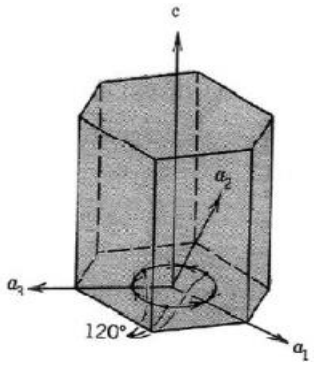

Figure 1.7 Structure of Hexagonal Ferrite [45].

1.3.3 Garnet Ferrites

Garnet ferrites have a crystal structure of garnet minerals $Mn_3Al_2Si_30_{12}$. Garnet ferrites are created through the substitution of Fe^{5+} ions for Al and Si, and the replacement of Mn with rare earth cations (R), resulting in the formation of a magnetic garnet with the general formula $R_3^{3+}Fe_5^{3+}0_{12}$. The garnet ferrite has a body-centered cubic structure with eight formula units. The garnet ferrites crystal has cubic symmetry and consists of three sub-lattices that are 24 tetrahedral (A), 16 octahedral (B) and 24 dodecahedral (C) sites [11].

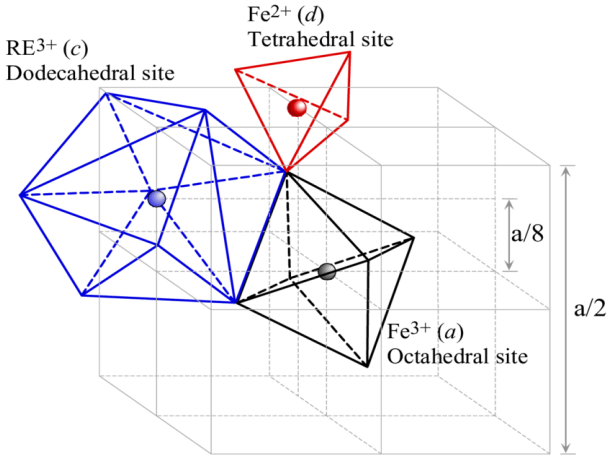

Figure 1.8 Garnet Ferrites [12]

1.3.4 Ortho Ferrites

Ortho-ferrites own a precise formula of $MeFeO_3$, where Me represents a trivalent metal ion, such as rare earth ion or Y. these compounds possess an orthorhombic unit cell with a distorted structure. Weak ferromagnetism is shown by these ortho-ferrites, which is thought to be due to slight canting in alignment of two anti-Ferro magnetically coupled lattices [11].

1.4 Applications of Ferrites

Ferromagnetic substances have an extensive range of applications, with the hysteresis curve playing a crucial and significant role. The phenomenon of ferromagnetism finds applications in various fields such as transformers, electromagnets and magnetic tape recording. Moreover, applicability of ferrites is in magnetic resonance imaging, radio frequency circuits, high pass filters and mount devices.

1.5 Magnetization

Magnetization refers to the concentration of magnetic dipole moment moments that emerge in a magnetic substance when it in proximity to a magnet. Additionally materials magnetic properties can be stimulated by the passage of electric current through it, which is then triggered by the movement of electrons in atoms or the spin of electrons or nuclei. Magnetization is sometimes referred to as magnet polarization [13].

1.5.1 Types of Magnetizations

- Diamagnetism
- Paramagnetism
- Ferromagnetism
- Anti-ferromagnetism
- Ferrimagnetism
- Super-paramagnetism

Diamagnetism

A diamagnetic substance has minimal or negligible magnetic properties because it lacks any unpaired electrons. Lenz's law also explains the meaning of diamagnetic. This law explains that diamagnetic substances develop induced dipoles when subjected to an external magnetic field, also the orientation of these dipoles is such that they oppose the magnetic field, resulting in repulsion. Diamagnetic materials have lower and negative magnetic susceptibility. In 1845, Michael Faraday discovered diamagnetism and its implications for materials. With the development of the modern periodic table, it was established that many elements, such as Gold, Silver, and Copper, exhibit diamagnetic properties. Additionally, semiconductors are considered among the most effective diamagnetic materials [14].

Paramagnetism

Paramagnetic materials exhibit a weak tendency to become magnetized along same direction as an external magnetic field when placed within it. They typically

possess a permanent magnetic or dipole moment, but lose their magnetism once the external field is removed. Upon application of magnetic field, atomic dipole within the material aligns with direction of the field, resulting in magnetization of material in that direction. This phenomenon is known as paramagnetism, which is primarily caused by the presence of unpaired electrons or partial alignment of randomly oriented atomic dipoles within material.

Magnetization of this kind relies on Curie's law, which states that the magnetic susceptibility χ of paramagnetic materials varies inversely with their temperature. It is represented as;

$M = XH = C/T \times H$

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 [15].

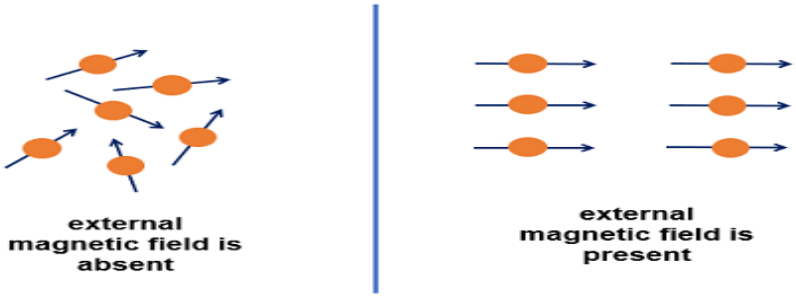

Figure 1.9 Paramagnetism [16]

Ferromagnetism

Ferromagnetic materials possess an inherent net magnetization at the atomic level, even without an external magnetic field. When subjected to an external magnetic field, these elements transform into highly magnetized in same direction as the field. Furthermore, ferromagnetic materials retain their magnetization for a period after the removal of the external magnetic field, a property known as hysteresis. Ferromagnetism is a distinctive magnetic behavior displayed by specific materials, including iron, cobalt, and various alloys [17].

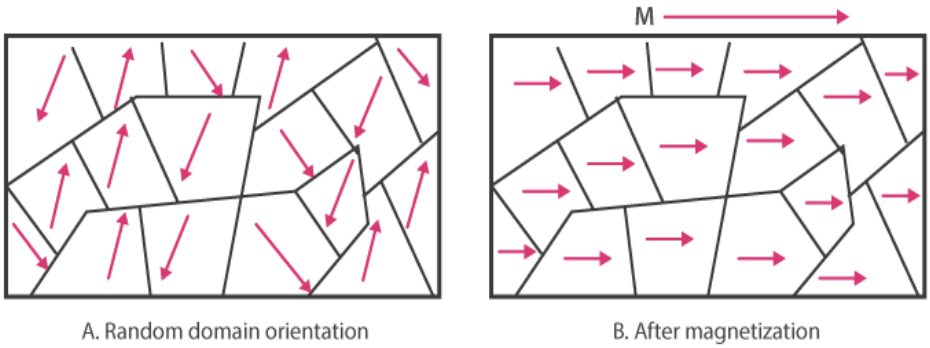

Figure 1.10 Ferromagnetism [17]

Anti-ferromagnetism

The hold of solids on magnetism such as manganese oxide is known as anti-ferromagnetism and the manganese ions are Mn2+. At low temperatures, ions within a material exhibiting magnetic behavior align themselves as tiny magnets into opposite or antiparallel arrangements. As a result, the material displays very little overall external magnetism. Antiferromagnetic materials, which consist of certain metals, alloys, and ionic solids, feature a cancellation of magnetic effects from atoms or ions that are oriented in one direction by those that are aligned in the opposite direction [18].

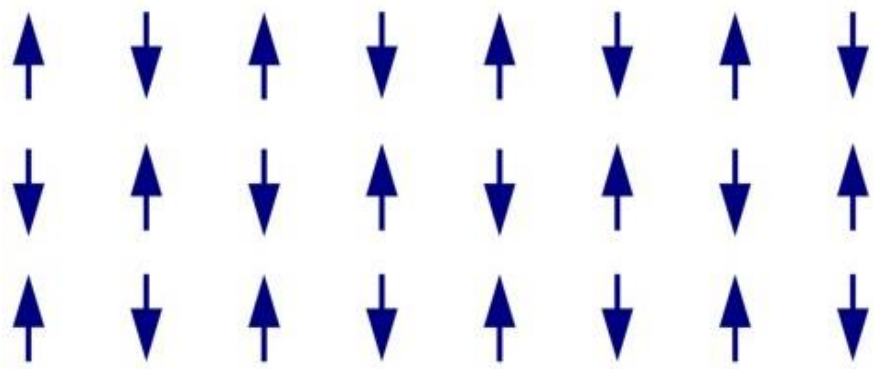

Figure 1.11 Anti-Ferromagnetism [19]

Antiferromagnetic materials display unique behavior when subjected to an external magnetic field, which varies based on temperature. At extremely low temperatures, the material remains unresponsive to the external field due to rigidly maintained antiparallel alignment of atomic magnets. However, as temperature increases, some atoms within the material break free of ordered arrangement and align with exterior magnetic field [18].

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. 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 sub lattice [20].

Super-paramagnetism

Super-paramagnetism is a type of magnetism observed in small ferromagnetic or ferri-magnetic nanoparticles. Such particles, magnetization may arbitrarily switch direction when temperature is taken into account, with time between two flips being termed Neel relaxation time. If measuring period for nanoparticles magnetization is much greater than Neel relaxation time and no external magnetic field is present, average magnetization appears be zero, indicating the nanoparticles are in the super-paramagnetic state. At this point an external magnetic field can magnetize the nanoparticles, corresponding to a paramagnet, but their magnetic susceptibility is much greater than that of paramagnets [21].

1.6 Zinc doped Nickel Ferrites

Nickel ferrites doped with Zinc, denoted by the chemical formula $Ni_{(1-x)}Zn_xFe_2O_4$, are considered highly versatile soft ferrites due to their ability to resist high levels of resistance and low eddy current losses. The composition of doping has an important impact on characteristics of nickel ferrite, as it alters both its chemical and physical properties. Zinc ferrites, due to their high opacity, are commonly utilized as pigments, particularly in applications that require heat stability. For instance, a Zinc ferrite manufactured from yellow iron oxide can serve as a substitute for temperatures above 350°F or 177°C. Additionally, Zinc ferrite is incorporated into high corrosion-resistant coatings, corrosion resistance improves as there is a rise in Zinc ferrite concentration. Recent research indicates that Zinc ferrite, which is paramagnetic in bulk, becomes ferromagnetic when a

Nano crystalline thin film forms. By controlling thin film growth conditions, significant ambient temperature magnetism and a narrow ferromagnetic resonance line width can be achieved.

These ferrites find use in a wide range of applications, including radio-frequency circuits, high-quality filters, rod antennas, and transformer cores. Preparation of these Zinc doped nickel ferrites involved utilizing sol-gel auto combustion method, where precursors Nickel nitrate hexa-hydrate ($Ni(NO_3)_2.6H_2O$), Iron nitrate Nano hydrate ($Fe(NO_3)_29H_2O$), and Zinc acetate di-hydrate ($(CH_3COOZn)_2.2\ H_2O$) were used as starting materials [22].

1.7 Copper doped Nickel Ferrites

Nickel-copper substituted nickel ferrites belong to an essential group of spinel ferrites. Nickel ferrite, which possesses a crystal structure of an inverse spinel ferrite, exhibits high electrical resistivity and low eddy current losses [24]. Introduction of copper into nickel ferrite alters its properties, rendering it useful in various device applications. Due to their high electrical resistivity, high saturation magnetization, and high magnetic permeability, Nickel-copper ferrites hold significant importance among magnetic materials. These $Ni_{1-x}CuxFe_2O_4$ ferrites are synthesized via the sol-gel method to achieve a homogeneous crystal structure, followed by sintering at high temperatures. The magnetic characteristics of samples of Cu doped Ni ferrites at x=0, 0.5 and 1were collected and investigated suitably [23].

1.8 Sol-Gel Method

The Sol-gel method is commonly employed technique for sample preparation that involves several steps. Initially, a solvent is used to prepare a precursor solution. The solution is then subjected to heat, resulting in the formation of a gel. The gel is subsequently heated to create solid particles, which are ultimately dried to form a powdered sample with the desired properties.

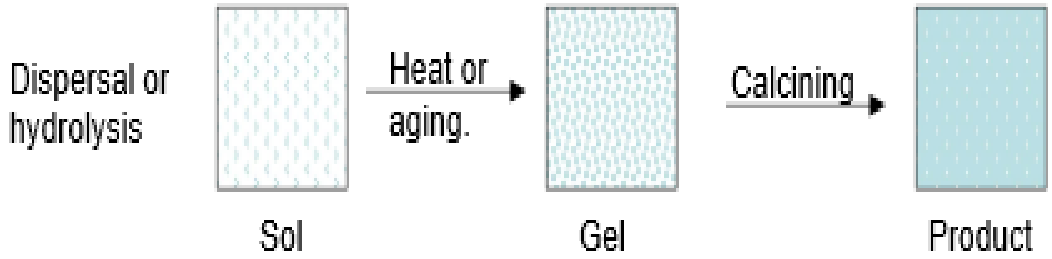

Figure 1.12 Stages of Sol-Gel Method [46]

The Sol-gel method is sensitive to a number of factors, including pH, solvents, time, temperature, and catalysts. Despite this sensitivity, it remains a popular choice for preparing large quantities of samples due to its cost-effectiveness and ability to be performed at room temperature. This versatile approach is particularly useful in the production of nanoparticles, powders, films, and fibers. Conversely, Sol-gel method is not without its shortcomings, including poor bonding, little resistance to wear, and elevated permeability [48].

RATIONALE

The scope of this study is to investigate and study the magnetic properties of Zinc doped Nickel Ferrites and Copper doped Nickel ferrites. Sol-gel method provides a simple and economic alternative technique ensuring good properties in sample under consideration. Since these ferrites are technologically advanced and used in various applications therefore their magnetic properties are studied.

OBJECTIVES

The objectives of the study are as follows:

- To study the magnetic properties of zinc doped nickel ferrites.
- To study the magnetic properties of copper doped nickel ferrites.
- Comparing the magnetic properties of zinc doped nickel and copper doped nickel ferrites.

CHAPTER 2

LITERATURE REVIEW

Sarwar Hasan et al. (2022) generated $Ni_{1-x}ZnxFe_2O_4$ (x 0.25 and 0.75) spinel ferrite Nano composites produced by sol-gel auto-combustion and treated between 250°C and 1000°C. Using X-ray diffraction, a single-phase spinel structure was discovered (XRD). The lattice parameters range from 8.351 to 8.434A, while the crystal size falls between 17.55 to 66.98 nm. When the zinc content increased from 0.25 to 0.75, X-ray study found a minor shift of peaks to shorter angles. XRD measurements were employed to determine distribution of metal ions in spinel ferrite system. Structural parameters lattice spacing, lattice constant, crystallite size, oxygen position parameter, tetrahedral and octahedral ionic radii, and bond lengths were calculated for each sample using the XRD data. Vibrational sample magnetometer (VSM) data were used to determine magnetic moment, remnant, coercivity, and saturation magnetization; the saturation magnetization magnitudes demonstrated impact of cations dispersal [26].

Abhishek Nigam et al. (2021) analyzed the physical and magnetic behavior of Zinc doped Nickel ferrites with chemical formula $Ni_{(1-x)}ZnxFe_2O_4$ (x = 0.2, 0.4, 0.6, and 0.8). Both Sol-gel auto-combustion and hydrothermal processes were used in the formation. The shape of the compound has been verified by SEM. The crystal size and lattice parameter have been examined by XRD. By utilization of a VSM, the magnetic characteristics of nanoparticles were determined at room temperature. Specimen has a single-phase cubic spinel ferrite structure, according to X-ray diffraction. According to the VSM analysis, Zn content significantly affects magnetic properties including magnetization (Ms), coercivity (Hc), and remanence (Rm) (Mr). Ms significantly increases from x=0.2 to 0.4 and subsequently decreases from x=0.6 to 0.8 due to the doping concentration of Zn increasing as x increases. Results demonstrated zinc-doped nickel ferrite Nano powder's permeable and fluffy nature. It also indicates that the smaller primary particles are grouped into the bigger secondary particles [27].

MP Ghosh et al. (2021) prepared Cu2+ ions doped nickel ferrite crystals. The purity of materials' crystal phases can be seen in their X-ray diffraction patterns. The resultant samples' average crystallite diameters ranged from 12 nm to 39 nm. The copper doped nickel ferrite has a higher lattice parameter as the Cu level rises. Samples with a high Cu content indicated the presence of the super paramagnetic features. Every sample had a single spinel phase free from impurities. The reduction of the crystal symmetry of the octahedral sub lattices with increasing Cu concentration was observed by the Raman spectra of all samples. Every samples absorption spectra were put down using UV-Vis spectroscopy, which proved their opaque nature in the near-red and infrared (IR) area. Average blocking temperatures for all tests performed were kept below 300 K to observe magnetic conductivity. According to the study, it was concluded on a whole that a small amount of Cu2+ dopant can drastically alter the structural, optical, magnetic, and dielectric properties of nanostructure nickel ferrite [28].

S Khangembam et al. (2021) chose composition of polycrystalline zinc doped nickel ferrites prepared by sol-gel combustion method. Sol-gel offers a straightforward and affordable alternative technique that ensures precise stoichiometric control, production of particles with a narrow size distribution in a relatively shorter duration, high homogeneity, and excellent sintering ability at lower temperatures. By using XRD, structural studies were conducted. The XRD data was used to calculate the density, size, lattice parameter, and other parameters. Moreover, in order to determine basic composition of samples SEM and EDAX experiments were employed. With an increase in frequency, the dielectric constant and loss decreases and eventually reach a constant value. With more Zn substitution, DC resistivity becomes less effective. Nickel ferrites doped with Zn are considered to be useful in microwave and electronic devices [29].

MML Sonia et al. (2014) developed Copper doped Nickel ferrites by sol-gel process which was then sintered at 1000 °C for 24 hours. Non-magnetic additive Copper was added to nickel ferrite Nano particles, so that it could be seen how the samples' important attributes were changed correspondingly. In general, the doping produces a reorientation of the two pre-existing octahedral and tetrahedral

sites for Fe+3 ions. The size of particle is significantly influenced by the sintering temperature and duration. Using X-ray diffraction, scanning electron microscope, and vibrating sample magnetometer examinations, structural, morphological, and magnetic characteristics the spinal ferrite specimen were examined. The functional groups of the magnetic material were identified from FTIR spectrum. The X-ray diffraction investigation demonstrated creation of a single phase spinal structure revealing a significant size reduction when copper was substituted. According to magnetic studies, the coercivity has increased while the saturation magnetization and retentivity have decreased [30].

GR Kumar et al. (2012) investigated how copper doping affected the magnetic and structural characteristics of nickel ferrites sintered at high temperatures. These ferrites were created using the sol-gel process. The production of single phase spinel structures and particle sizes larger than the Nano scale were clearly demonstrated by X-ray diffraction experiments. It has been discovered this lattice parameter grows as the copper content does. The structural and magnetic characteristics were found to be significantly impacted by the copper substitution. At x = 0.0 to 0.5 and x = 0.5 to 0.9, the saturation magnetization results show a non-collinear ferromagnetic form and a Neel's collinear ferromagnetic structure, respectively, implying a change in magnetic ordering [31].

F Shahbaz Tehrani et al. (2012) made zinc doped nickel ferrites and copper doped nickel ferrites by sol-gel auto combustion method. $Ni_{1-x}MxFe_2O_4$ (0x1, M, Cu, Zn) nickel ferrite Nano crystals were created by adding Zn and Cu dopants, and the resulting samples' distinct and specific properties were examined. Due to their diverse physical characteristics, ferrite Nano crystals are an appealing material. It was demonstrated that this doping process actually causes the samples to acquire important magnetic characteristics. The Jahn-Teller effect also appears in Cu doped sample, which we pinpoint using the materials' FTIR Spectroscopy. Additionally, we demonstrate a rise in lattice parameters of the doped samples along with super paramagnetic behavior, whereas Jahn-Teller effect prevents the $CuFe_2O_4$ Nano crystals from exhibiting a comparable conduct. By measuring photoluminescence at room temperature, results of Zn and Cu replacements on

optical characteristics of nickel ferrite Nano crystals are examined. Vibrating Sample Magnetometry (VSM) magnetic measurements showed that saturation magnetization (Ms) increased along content of Zn/Ni and Cu/Ni up to x=0.5, and subsequently decreased for x>0.5 [32].

AT Raghavender et al. (2011) successfully prepared and investigated the magnetic properties of Nano crystalline $Ni_{1-x}ZnxFe_2O_4$ (x=0.0, 0.2, 0.4, 0.6, 0.8, and 1.0) ferrites. Formation of ferrites was exclusively done by sol-gel process, which is thought to be efficient and useful for changing the surface of substrates and highlighted various properties contained in sample. It was observed that at temperatures between 500 and 1000 °C, the resulting powder was annealed. The structural changes occurring with variable Zn concentration were investigated using X-ray diffraction (XRD) and infrared spectroscopy (IR). Along a growth in Zn concentration, grain size was reduced while the lattice parameter increased. It was noticed that the particle size decreases as the Zn content rises. Since Zn had been attributed with larger ionic radius contrast to Ni, the lattice parameter increased as Zn content increased. A thorough investigation was conducted utilising IR measurements, and it was found that the doping concentration and annealing temperature have a significant impact on structural alterations [33].

SA Saafan et al. (2010) investigated zinc doped nickel ferrites. X-ray diffraction analysis (XRD) was applied to analyze nanoparticles in order to confirm the Nano scale production of the ferrite. The samples' infrared (IR) spectroscopy supports the occupancy of water and the distinctive ferrite absorption bands. The samples' ac and dc conductivity was examined right away after sample preparation. The materials were then reexamined after drying at 200°C for roughly 12 hours. In the two situations, conductivity behaves very differently, clearly demonstrating how humidity has an impact. Also, the vibrating sample magnetometer (VSM) availed regarded magnetic induction of manufactured samples. Small particle size clearly has an impact on the saturation magnetization values and hysteresis loops. A super paramagnetic phenomenon is present in the materials with a wide range of practical uses [34].

D Gao et al. (2010) fabricated and studied nanowires of $ZnFe_2O_4$ having a diameter of approximately 16 nm were synthesized, possessing a body-centered cubic (bcc) structure where the texture direction along the nanowire axis is [110], according to results of XRD and HRTEM. Due to preferred orientation and shape anisotropy of the nanowire arrays, $ZnFe_2O_4$ nanowire arrays exhibit uniaxial magnetic anisotropy, with the easiest direction for magnetization being along nanowire axis. Furthermore, $ZnFe_2O_4$ nanowire arrays emit blue-violet light, as shown by the optical characteristics, and the optical band gap E_g is calculated from the absorption coefficient. The magnetic and optical characteristics of $ZnFe_2O_4$ nanoparticles are currently the subject of future research [35].

M Sultan et al. (2009) deposited thin films of zinc doped nickel ferrites at room temperature (RT) and a pure oxygen environment; deposition on glass substrates was carried out using rf-magnetron sputtering. X-ray diffraction analysis of as-deposited films revealed zinc ferrite is source of the single phase Nano crystalline spinel patterns. The behavior of the magnetization exhibits ferromagnetic properties and is highly dependent on the oxygen working pressure. The film that was deposited at a pressure of 27 mTorr of oxygen had a maximum magnetization of 230emu/cm3 and 42emu/g at room temperature. High magnetism that results from the speedy cooling of sprayed vapors to form solid state layer may be caused by the concentration of oxygen vacancies and the random distribution of Zn^{2+} and Fe^{3+} on both tetrahedral and octahedral sites. Above 550 nm wavelength, the films' optical characteristics exhibit 85% transparency. Direct and indirect band gaps are expected to be 2.5 and 1.9 eV, respectively [36].

A Costa et al. (2008) investigated structural, morphological, and magnetic characteristics of $NiFe_2O_4$ ferrite samples to see by what means replacement of nickel ions for zinc ions influences such features. Powders of $Ni_{1-x}Zn_xFe_2O_4$ (x 14 0.0, 0.3, 0.5, 0.7) were produced using combustion reaction and structure analyzed applying X-ray diffraction. Nitrogen adsorption method (BET) was used to determine the powders' specific surface area. An alternate gradient magnetometer (AGM) was used to assess magnetization of grind produced by the combustion reaction, and the results showed that the powders had Nano sized particles with a

particle size range of 18–27 nm. Production of primary phase Ni-Zn ferrite and traces of secondary phase hematite were seen in all of compositions (a-Fe_2O_3). The size of the crystallites and the lattice parameter grew as Zn concentration raised. Moreover, saturation magnetization and coercive field were improved by increasing Zn content in the $NiFe_2O_4$ ferrite [37].

J Azadmanjiri et al. (2007) utilized sol-gel combustion method for combination of Nano-sized Cu–Ni ferrite ($Ni_{1-x}Cu_xFe_2O_4$) Nano powders. After being ignited in air using this manner, gel displays a self-propagating characteristic. High sintering activity was shown by the produced powders, which may be sintered at temperatures lower than 950 °C. Obtained low-temperature sintered Ni-Cu ferrites have fine-grained microstructures and good electromagnetic characteristics, which make them suitable materials for high-performance and inexpensive electronic applications. There was discussion of the potential causes of the composition dependence of the primary electromagnetic characteristics. For the Ni-Cu ferrites, the Cu concentration significantly affects the electromagnetic properties, including initial permeability, resistivity, and dielectric loss tangent [38].

GP Josh et al. (2003) modified Nano composites of Ni-Zn at regular pressure and room temperature, which were then examined in the presence of various amounts of zinc ions. At room temperature, reflection spectra in the wavelength range of 400–850 nm by a spectrophotometer are used to measure the energy band gap of these materials. Direct band gaps of Nano composites of aniline and formaldehyde copolymer with $Ni_{1-x}Zn_xFe_2O_4$ (x = 00, 02, 04, 06, 08 and 10) range from 150-166 eV, according to the examination of reflection spectra. The increase in band gap observed experimentally as the concentration of Zn increases implies that the change in structure within the composite is the likely cause of this variation [39].

SM Hoque et al. (2002) studied $Ni_{1-x}Cu_xFe_2O_4$ using scanning electron microscopy (SEM) to examine the crystal structure, X-ray density, porosity, and compressive strength so as to determine the impact of composition and microstructure on magnetic alongside electrical properties. X-ray diffraction proves that single-phase ferrite is derived. With the sample of composition x 14 1, or for pure $CuFe_2O_4$, tetragonal deformation is seen. For samples of other

components, the crystal structure is face-centered cubic (FCC). According to SEM micrographs, copper concentration increases together with the size of the grains. With more Cu present, the compressive strength drops. Initial magnetic permeability and saturation magnetization are at their highest for $Ni_{0.8}Cu_{0.2}Fe_2O_4$ (x 14 0:2), which can be assigned to composition's highest sintered density. The initial permeability shows an increase from x=0 to x=0.5, along with a shift in the dispersion towards lower frequencies. As for the saturation magnetization, it increases with increasing Cu content up to x=0.2 and then gets lesser. On other hand, resistivity decreases as the Cu content increases [40].

CHAPTER 3
MATERIALS AND METHODS

3.1 Zinc Doped Nickel Ferrites Prepared by Sol-Gel Method

F Shahbaz Tehrani et al. used the raw materials i.e., citric acid $C_6H_8O_7$, ferric nitrate Fe $(NO_3)_3.9H_2O$ (98%), nickel nitrate Ni $(NO_3)_2.6H_2O$ (99%), zinc nitrate Zn $(NO_3)_2.6H_2O$ (99%) and copper nitrate Cu $(NO_3)_2.3H_2O$ (99%) to prepare the Zn doped Ni ferrites nanoparticles. To create 0.5M solutions, the balanced amounts of nitrates and acid citric were separately diffused in deionized water. Metal nitrates and citric acid were considered to have a mole ratio of 1:1. A magnetic stirrer was used to continuously stir the combined solution at 80ºC. Auto-combustion was used to produce dark-brown granules from the dried gel. Ethylenediamine was added to the solution until its pH reached 1 in order to produce smaller Nano crystals. This gel was baked for 24 hours at 135°C before being grounded into powder [22, 32].

3.2 Copper Doped Nickel Ferrites Prepared by Sol-Gel Method

MML Sonia et al. prepared copper doped nickel ferrite powders using sol-gel method. Citric acid ($C_6H_8O_7$), Ferric nitrate (Fe $(NO_3)_2.9\ H_2O$), Nickel nitrate (Ni $(NO_3)_2.6\ H_2O$), and Copper nitrate (Cu $(NO_3)_2.3\ H_2O$ served as the initial components. To create 0.5 M solutions, proper quantities of nitrates and acid citric were separately liquefied in deionized water. Metal nitrates and citric acid were taken to have a mole ratio of 1:1. The produced sol was then heated continuously at 700C while being stirred constantly until a dry brown gel formed. The gel created in this way was baked at 1000 o C for 24 hours before being finely powdered. The powder was calcined at 300–600 °C for 4 hours to produce different Cu^{+2} replacements [30].

3.3 Sol-Gel Method

The Sol-gel auto combustion technique is rapid and economical method used to synthesize particulate products. This technique has been extensively employed to

produce a diverse range of metal and alloy nanoparticles, resulting in the formation of Nano-sized, homogeneous, and highly reactive powders through the amalgamation of different elements at atomic level. Precursors used in process were Cobalt Nitrate, Iron Nitrate, Zinc Nitrate, and glycine. To begin, stoichiometric amounts of all metal nitrates in a glass beaker were dissolved alongside minimal quantity of double-distilled water. Glycine was then added to metal solution mixture in a molar ratio of nitrates to glycine of 1:4, and mixture was stirred using a magnetic stirrer at approximately 1500°C until uniform stirring and evaporation resulted in the formation of a highly viscous gel. As the process progressed, solution became increasingly viscous also eventually formed a very homogeneous, viscous brown gel. Once the auto-ignition was completed, resultant product resembled structure of a branched tree, with black-colored ashes. Finally, the powders of all the samples were calcined separately at 6000°C for three hours to obtain complete results and end product [24].

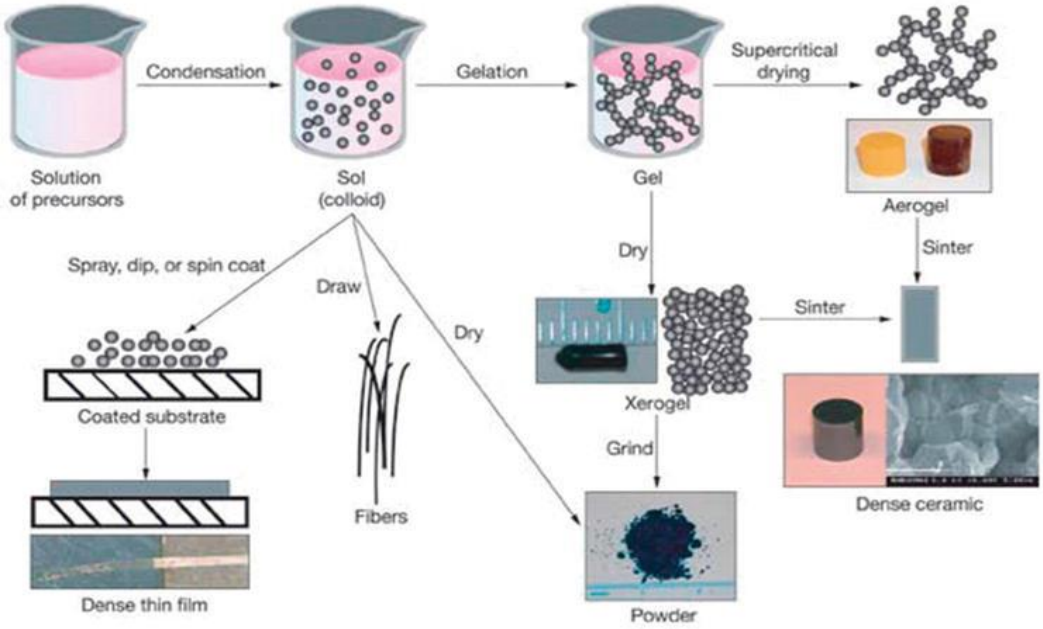

Figure 3.1 Systematic levels of Sol-Gel Method [48].

The sol-gel coating process typically involves four distinct phases. Firstly, to create a sol colloidal particles are disseminated in a liquid. Next, the sol solution is applied to substrates using techniques such as dipping, spinning or spraying. Stabilizing agents are removed from the sol, and the particles are then

polymerized to create a continuous network within the gel. Finally, any remaining organic or inorganic components are pyrolyzed, resulting in the formation of an amorphous or crystalline coating [46]. The sol-gel process is revolutionary approach towards development of novel resources. This approach allows for more exact control of all solid-state synthesis processes [24].

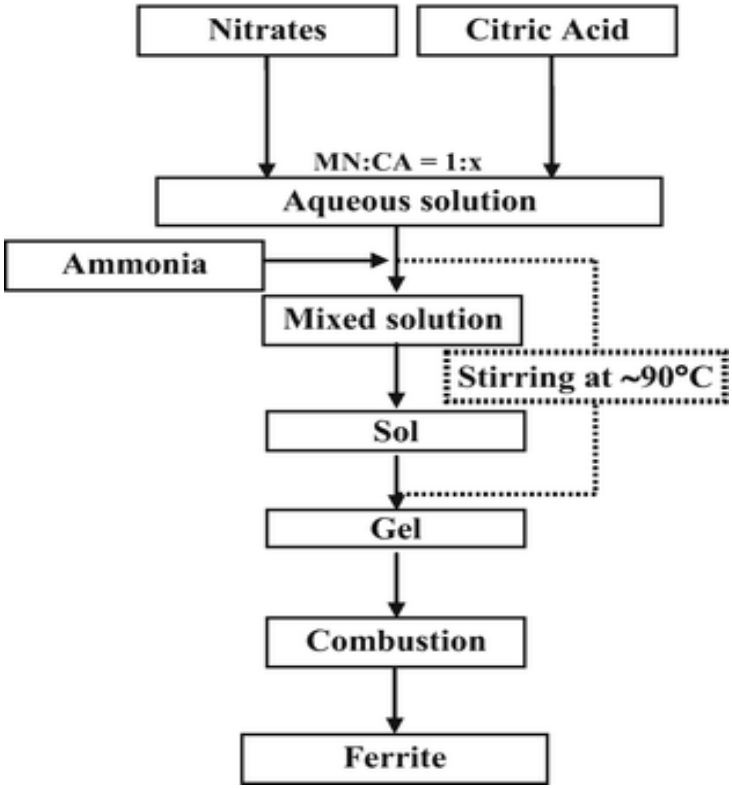

Figure 3.2 Flow Chart for Sol-Gel method of producing ferrites [24]

3.4 Techniques for Characterization

3.4.1 Vibrating Sample Magnetometer

A vibrating sample magnetometer (VSM) is laboratory tool that used to evaluate the magnetic properties of a substance. Vibrating component, which creates an electrical field in a coil, causes alternation in magnetic field of sample. Vibrating sample magnetometer additionally contains a mechanism to hold sample at the proper angle, a vibrating element, control unit, and a meter [40]. VSMs also enable researchers to examine a sample's magnetization from various angles, reducing the impact of extraneous domination. On the contrary, VSMs are not

ideal for determining the magnetization loop because of the demagnetizing effects of the sample. Temperature dependence is another issue with VSMs [41].

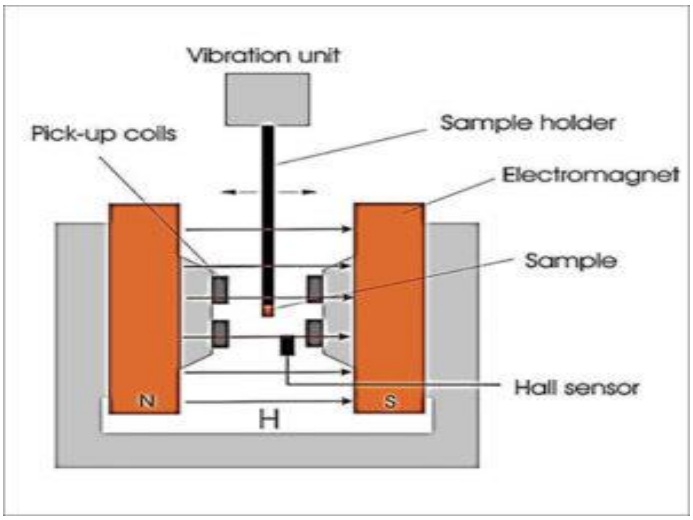

Figure 3.3 Constructive diagram of vibrating sample magnetometer [42]

The VSM technique can detect changes within the range of 10^{-5} to 10^{-6} emu. It operates on the principle of Faraday's law of magnetic induction, which states that changing magnetic field generates an electric field. The VSM measures this electric field to extract information about magnetic field [47]. Constructive diagram of VSM as illustrated in figure 3.1 comprises of several components, including a sample holder, water-cooled electromagnets, a vibrating head module, pickup coils, lock-in amplifier, control chassis, and computer system. The vibrating head module produces vibrations and up-down movements, with a frequency of 85Hz, which is monitored and regulated by the control chassis. The sample to be examined is placed on the sample holder, which can be moved and positioned in any desired direction. The sample is located between the electromagnets, and various sample holders are utilized to measure the magnetic moments in-plane and out-of-plane, or in-plane only [41].

Primarily, a sample is placed on the sample holder and subjected to a constant magnetic field. Sample becomes magnetized and the spins align with the applied magnetic field, with strength of magnetization depending on the intensity of the magnetic field. When position of the sample is altered, such as by moving it up or down, the induced magnetic field also changes, and this change is detected by the

pickup coils in form of an electric field. Varying magnetic field generates current, which is amplified by the amplifier and any unwanted signals, such as noise, are reduced. A computer system is integrated with the VSM to monitor changes in the magnetization of the sample [40].

3.4.2 M-H Curve

Magnetic hysteresis arises when ferromagnetic material, such as iron, is exposed to an external magnetic field, causing its atomic dipoles to align with the field. When 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 displayed an applied changing magnetic field. Hysteresis is term for repeated occurrence of magnetization and demagnetization. H is the magnetizing force represented in Oersted (Oe = $(4\pi)-1\times103$ A/m) and M is the mass magnetization expressed in magnetic moment per gramme (emu/g) [43]. Because substance had been magnetized, portion of alignment will retained after it is brought out of the field. Once the magnet is magnetized, then magnet 27 will stay magnetized forever. Demagnetization is required either when heat is applied or a magnetic field in opposite direction occurs. And this is the effect that provides a hard disc drive with its memory component [43]. These elements relation 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, and then progressively slows as it approaches magnetic saturation; M takes on a new shape, conditionally 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 primary hysteresis loop is obtained by plotting the H-M relationship for all magnetic field strengths. The middle part's width along the H axis is doubled [43].

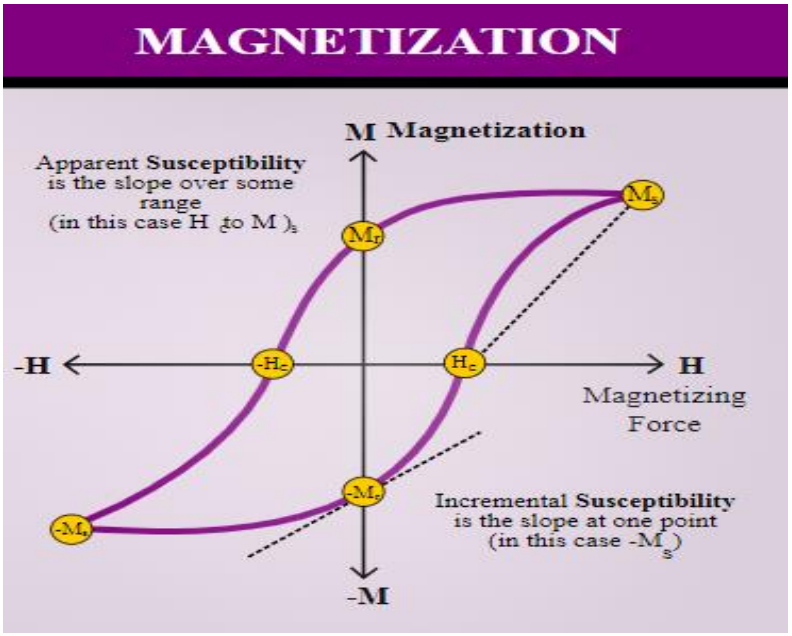

Figure 3.4 The M-H hysteresis curves [44]

CHAPTER 4
RESULTS AND DISCUSSION

Magnetic materials can be designed and categorized by relevant and defined use of M-H curve. Magnetic hysteresis loops are responsible for spin ordering in magnetic materials. When external varying magnetic field is applied on matter, it presents magnetization. M-H hysteresis loop is then used in order to measure resulting magnetization. Hysteresis is defined as the repeated process of magnetization and demagnetization. Mass magnetization is represented by M having units of magnetic moment per gram (emu/g) and H is the magnetizing force expressed in Oersted (Oe = $(4\pi)^{-1}\times10^{3}$ A/m).

Firstly, Zinc doped Nickel ferrites are studied and evaluated accordingly.

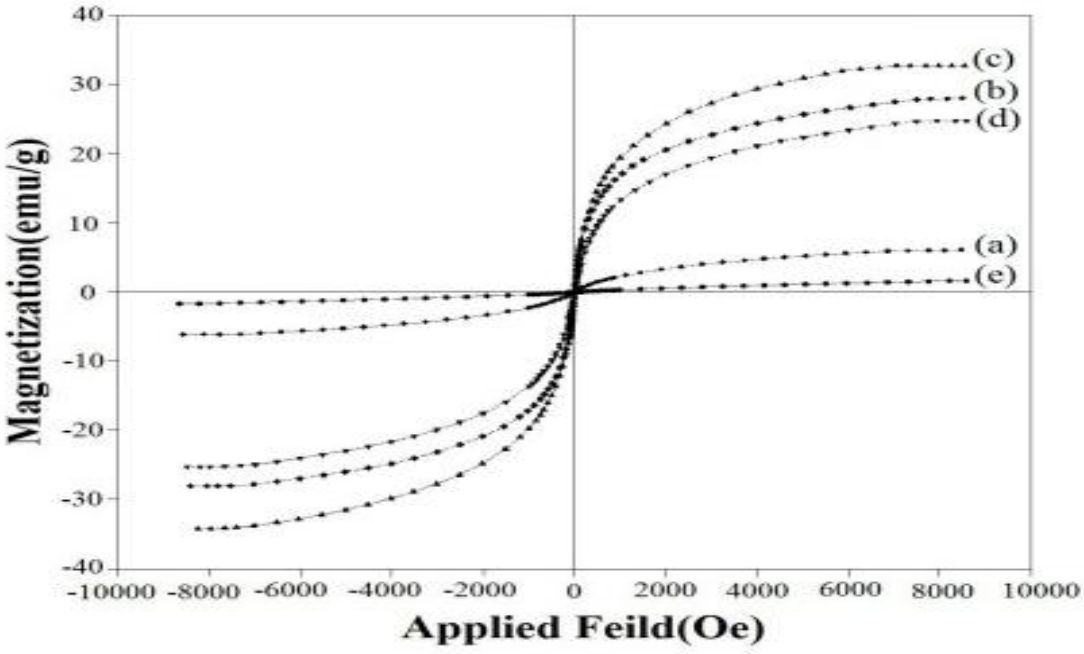

Figure 4.1 M-H loop of Zinc doped Nickel Ferrites nanoparticles prepared by Sol-Gel Method [32]

Magnetometer (VSM) was used to analyze the magnetic behavior Nano crystals used in this study at room temperature. The S shaped and inclined curves present various magnetic parameters when read on graph according to corresponding

labeled axis. Magnetic properties such saturation magnetization (Ms) and coercivity (Hc) values were obtained from curves and noted in the tables.

Table 4.1 Observed values of magnetic parameters for Zn Ni ferrite [32]

Zn content (x)	**Composition**	**Saturation Magnetization (emu/g)**	**Coercivity (O_e)**
0	$NiFe_2O_4$	6.4	0.6
0.3	$Ni_{0.7}Zn_{0.3}Fe_2O_4$	25.6	0.7
0.5	$Ni_{0.5}Zn_{0.5}Fe_2O_4$	34.8	0.9
0.7	$Ni_{0.3}Zn_{0.7}Fe_2O_4$	28.4	1.0
1	$ZnFe_2O_4$	1.7	3.3

Values up to five were taken for different Zn contents increasing periodically from x=0 to x=1. Corresponding composition, saturation and Oe values were recorded and explored. According to observation, Ms increases while increasing the Zn/Ni contents up to x=0.5 [32]. Maximum value of saturation is 34.8 emu/g at x=0.5. At this maximum point the composition is $Ni_{0.5}Zn_{0.5}Fe_2O_4$. After this M_S decreases gradually. Drop in values are measured as 28.4 and 1.7 [32]. Super paramagnetic Nano crystals have a quality where they have small particles which have almost low coercivity and are without any remanence. Thus it poses to be with only one domain along with small anisotropy energy. According to the table the coercivity rises at a moderate rate from 0.6 to 3.3.

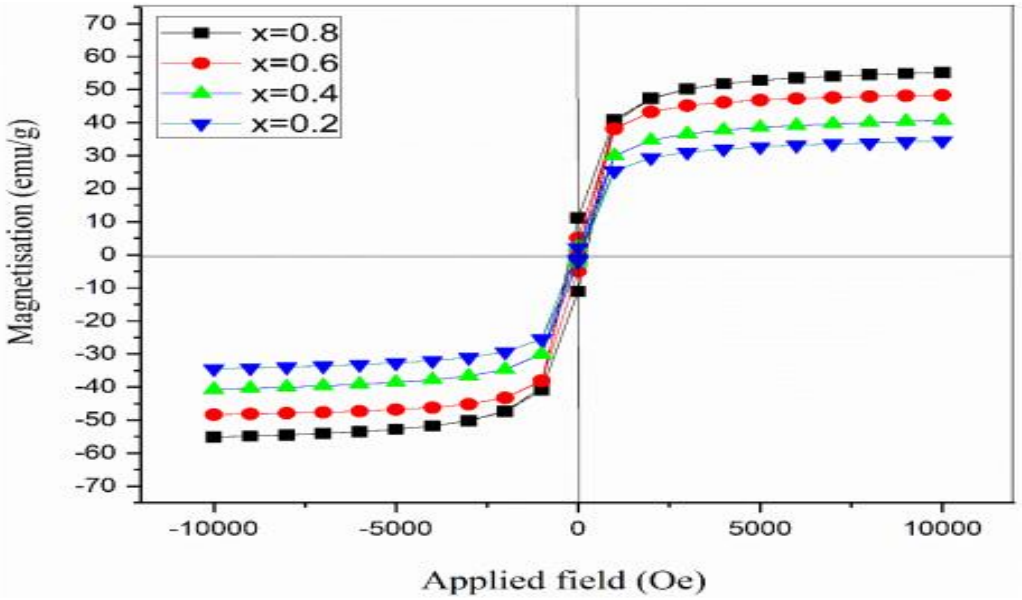

Figure 4.2 VSM Hysteresis for Zn Ni nanoparticles [22]

Table 4.2 Values listed for saturation and coercivity for Zn doped Ni [22]

Zn doping	M_s (emu/g)	H_c (Oe)
X=0.2	54.8	216
X=0.4	48.4	118
X=0.6	40.07	77.7
X=0.8	34.5	77.23

The VSM plot and the table listed with magnetic values shows that saturation level increase till x=0.2 and then moderately decreases [22]. This is particularly due to ion distribution and their respective locations. The magnetization increases. From x=0.4 onwards till x=0.8, the structural bonds become weak so magnetization is low. Coercivity appears to have a decreasing trend with increase in doping shown by values of x [22]. Minimum amount of coercivity was at x=0.8. Decrease in H_c values are because of variation in particle and size of ferrite during doping process. Zn doped Ni ferrite with comparatively large particle size may easily lose magnetization. Temperature is also an important character resulting in decline of H_c values as the annealing temperature becomes high.

Now, Copper doped Nickel ferrites are studied and examined.

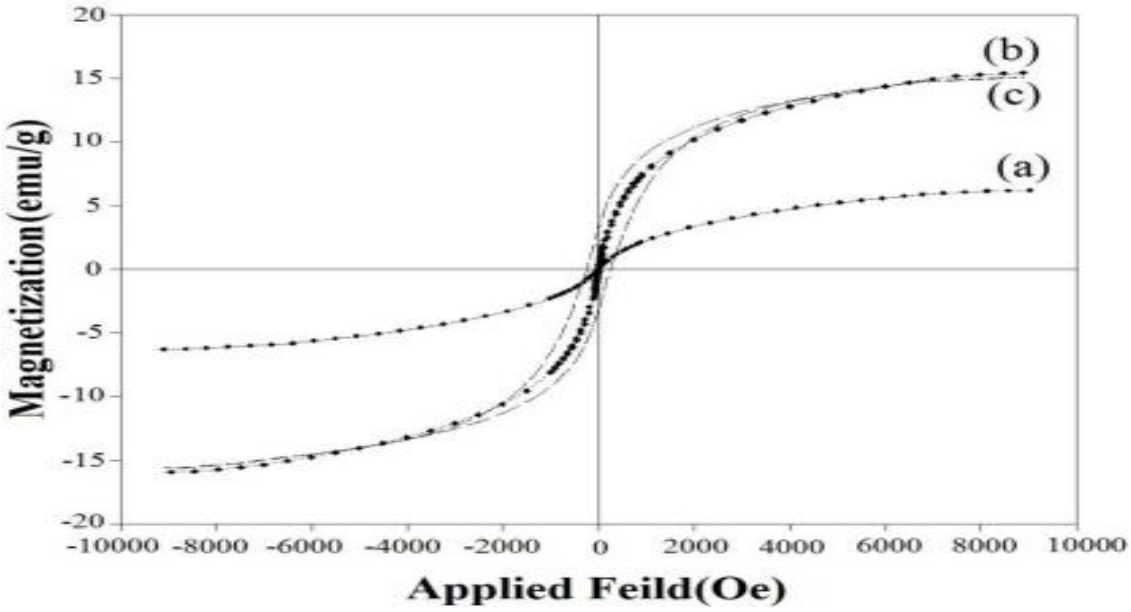

Figure 4.3 M-H loop for Copper doped Nickel Ferrites nanoparticles developed using Sol-Gel Method [32]

As the case of Zn doping and its illustration, similarly sol- gel method was employed to form Cu doped Ni ferrites which were then closely investigated for magnetic qualities and their effect on the sample.

Table 4.3 Noted values magnetic parameters for Cu Ni ferrite [32]

Cu content (x)	**Composition**	**Saturation Magnetization (emu/g)**	**Coercivity (O_e)**
0	$NiFe_2O_4$	6.4	0.6
0.5	$Ni_{0.5}Cu_{0.5}Fe_2O_4$	16.2	2.3
1	$CuFe_2O_4$	14.9	168.2

Various compositions were selected and three values of x= 0, 0.5 and 1 were checked for related magnetization and coercivity. Saturation magnetization increases till x= 0.5 and then drops to 14.9 emu/g. Highest values of M_s is 16.2 at

x=0.5 whereas the composition at that certain point is $Ni_{0.5}Cu_{0.5}Fe_2O_4$. The fall in M_s is not much significant in Cu doped Ni as compared to Zn/Ni compounds [32]. Increment takes place in Coercivity (Hc) of acquired Nano crystals fluctuating from 0.6 to 168.2. Hc values of all prepared Nano crystals are close to zero, except CuFe2O4 Nano crystals (Table 4.1 plus 4.3) [32]. Therefore at room temperature they exhibit super paramagnetic behavior. The Hc of the samples increases with the Zn/Ni and Cu/Ni substitutions. This increase is small for Zn/Ni ferrite Nano crystals, while it is significant for Cu/Ni ferrite Nano crystals.

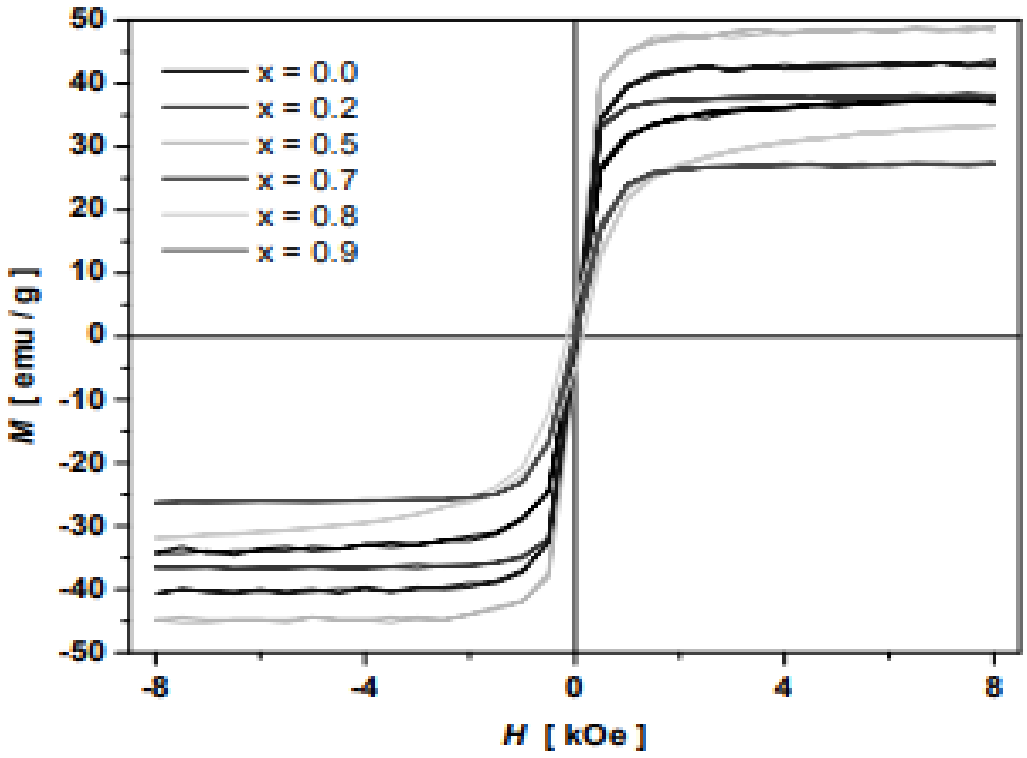

Figure 4.4 Hysteresis loops of $Ni_{1-x}Cu_xFe_2O_4$ ($0 \leq x \leq 1.0$) ferrites [31]

The magnetization of $Ni_{1-x}Cu_xFe_2O_4$ samples with varying compositions ($0 \leq x \leq 1.0$) was measured at room temperature using a vibrating sample magnetometer (VSM) [31]. Maximum applied field used was 8 KOe, and hysteresis loops showed complete saturation with the applied field.

Table 4.4 Saturation magnetization (M_s) with Cu content x [31]

Cu content (x)	**Saturation Magnetization (emu/g)**
0	35.8
0.2	37.7
0.4	44.1
0.5	46.9
0.7	41.9
0.8	32.5
0.9	26.9

Upon increasing the Cu content x, the saturation magnetization (MS) of the samples was observed to initially increase until x = 0.5, after which it gradually decreased [31]. The samples with compositions ranging from x = 0.0 to 1.0 displayed ferrimagnetic behavior. The magnetic behavior increased originally from x = 0.0 to 0.5, but then decreased against x = 0.5 to 0.9, indicating transition in the magnetic ordering.

MP Ghosh et al. observed Cu doped Ni samples were evaluated at 300K and plotted using magnetometer and the results were illustrated by several curves. It was noted that magnetic response of ferrites is affected by distribution of cations and concentration of doping. Saturation and coercivity values decrease after a certain point [23]. At higher copper content net magnetic moment also decreases. As copper doping increases nanoparticles gradually lose magnetic properties and slowly become paramagnetic. At reaching near 300K the nanoparticles sample obtained from ferrite have low magnetization values.

CONCLUSION

The magnetic properties of zinc doped nickel ferrites and copper doped nickel ferrites prepared by sol-gel method were studied and compared. Magnetic measurements through VSM indicated an increase in the saturation magnetization (MS) with the Zn/Ni and Cu/Ni content up to certain value and then decrease gradually. The increase in coercivity is higher in the Zn/Ni substitution as compared to Cu/Ni substitution. The drop in saturation is significant for Zn doped Ni compound and is majorly due to spin on the surface of nanoparticles. It is observed that preparation technique has significant impact in altering magnetic properties of ferrites. Coercivity of both the elements under exploration shows an increase. However, as the temperature raises coercivity values also begins to decline at a specific rate.

LIMITATIONS

- It was difficult to collect all the information online as some of the articles had no public access.
- The behavior of certain compounds was found to be irrelevant under different temperature conditions, likely due to the temperature dependence of doping.

RECOMMENDATIONS

- Ferrites in Nano scale exhibit magnetic properties that make them suitable for various practical applications that involve data storage through magnetism.
- Advancement in field of magnetic devices could be made on altering doping concentrations of ferrites.
- Hard magnets can be used in devices which require storing data for longer period of time such as magnetic recording devices.
- Soft magnets can be used in practical applications such as transformers, motors and electromagnets.

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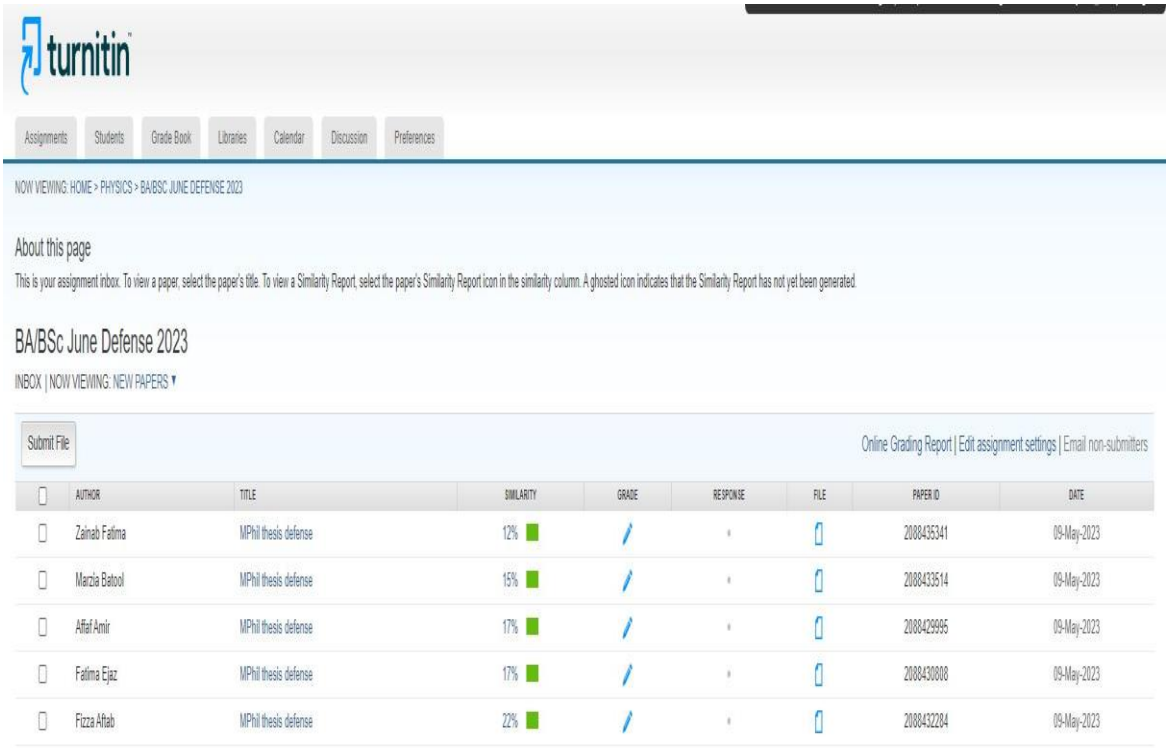
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