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Effect of Doping on Structural and Dielectric Properties of Nife2o4 Nanoparticle

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Abstract

Original Research Article

Bare and calcium-doped Nickel ferrite ($NiFe_2O_4$) was prepared by sol-gel auto combustion method. This study investigates the structural and dielectric properties of bare and calcium doped $NiFe_2O_4$ nanoparticles. X-ray Diffraction (XRD) analysis confirmed the formation of a single-phase spinel structure, with slight shifts in peak positions indicating successful incorporation of Ca^{2+} into the $NiFe_2O_4$ lattice. The average crystallite size was found 25 nm and 20 nm for bare and doped nanoparticles. The slight shift in XRD showed due to the greater ionic radii of Ca as compared to Ni. Dielectric properties showed the increasing trend by Ca doping due to larger average crystallite size of doped as compared to bare nanoparticles. Overall, calcium doping in $NiFe_2O_4$ leads to enhanced dielectric properties, making it a viable candidate for advanced technological applications. Future studies should focus on optimizing the doping concentration and further exploring the mechanistic aspects of these enhancements to fully harness the potential of calcium-doped ($NiFe_2O_4$).

Keywords: Nickel ferrite ($NiFe_2O_4$), Calcium doping, Dielectric properties, Nanoparticles, Sol-gel auto combustion.

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1.1 INTRODUCTION

Nanotechnology is a multidisciplinary field of science and technology that involves the manipulation of matter on an atomic, molecular, and supramolecular scale to create new structures, materials and devices. It is an advanced and leading technology of the present age and emerging field of the 21st century [1-2]. It deals with nano size meterials and small structures and it is bringing revolutions in the numerous fields of life especially in the field of electronics, sensor technology, medical, information storage devices and industry etc. Norio Taniguchi is the founder of nanotechnology, the introduced the term “Nanotechnology”. In 1959 Richard Feynman gave the ideas of creating nano-scale machines [3].

Nanotechnology is the branch of science and technology in which the material has at least one dimension from sub nm to hundred nm or 1 nm to 100 nm. Nanotechnology is dealing with different branches like physics, chemistry, biology, medical and material science. The surface to volume ratio of the nanomaterial enhances due to particle size decreasing [4-5].

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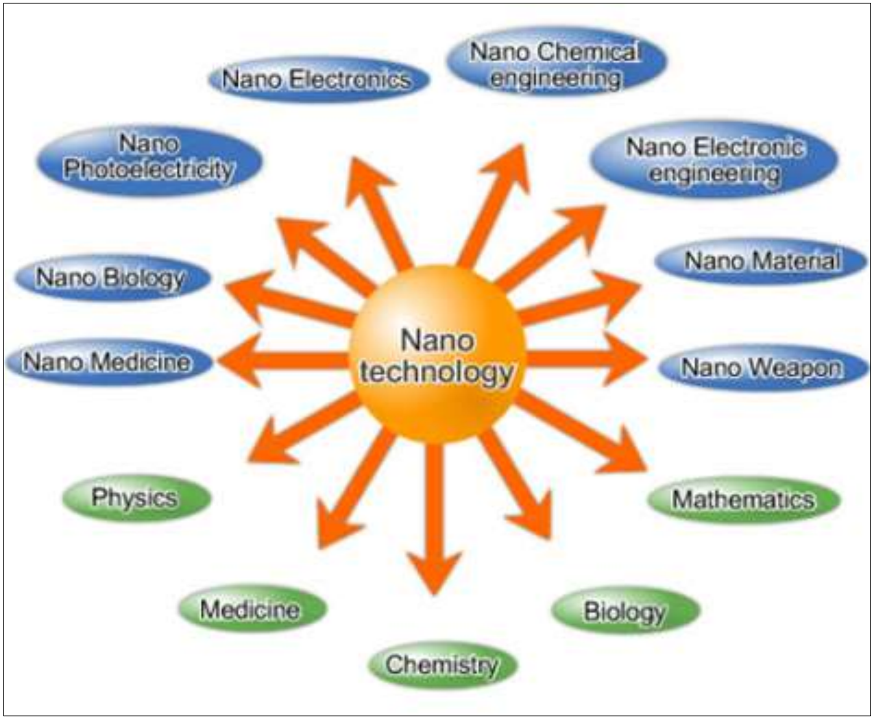


Fig. 1.1: Fields of nanotechnology & nanoscience [6].

1.2 What is Nano

Nano is a Greek word which means very small or tiny but in nanoscience it is used for a specific unit known as a nanometer (nm). It is defined as "a billionth part of a meter" i.e. 10^{-9}. It is very surprising if we define one inch in nanometers is that one inch contain twenty five millions of nanometers. One nanometer is a length that approximately equivalent to 5 silicon atoms or 10 hydrogen atoms aligned in a line. It is also measured that the diameter of weak human hair is round about 80,000 nm and diameter of human DNA is 2.5 nm. We can understand one nanometer is the growth of human nail or human hair at the rate of 1nm per sec [7].

1.3 Nanoparticles

Those particles whose particle size lies in the range of 1 to 100 nm are called nanoparticles [8]. These particles exist in nature naturally and can also be produced artificially. The research on nanoparticles shows that nanoparticles exist in atmosphere and these are created by human made processes such as industrial combustion processes and traffic emission. There are two types of nanoparticles in atmosphere. Those nanoparticles which are directly emitted by traffic exhaust or industrial combustion processes are called primary nanoparticles and those nanoparticles which are generated itself in the atmosphere from the condensation of low volatile vapours formed by the oxidation of atmospheric gases are called secondary nanoparticles. Nanomaterials and nanostructure has a large fraction of surface to volume ratio. When the size of the material decreases from bulk to nanoscale different properties like magnetic, electronic, optical, thermodynamic and mechanical properties are changed. When the size of the particle reduces such as less than 100 nm the material possess unique magnetic properties due to which material scientist take much interest in magnetic nanoparticles [9].

1.4 Bulk material and their Nanoscale Materials

At nanoscale material behave differently from their bulk material e.g. gold is very stable at bulk state and at nanoscale gold is very reactive and becomes a very highly efficient catalyst. Silver nanoparticles exhibit bioactive properties that are not found in larger particles. Ferroelectric materials and ferromagnetic materials change their properties at nanoscale. Melting point temperature of the materials also decreases due to decreasing the size of the particles [10].

1.5 Types of nanomaterials

There are many types of nanomaterials. These can be zero dimensional (0D), one dimensional (1D), two dimensional (2D) and three dimensional (3D).

In zero dimensional material, the motion of electron is confined in all dimensions such as the trapped electron in a box. Quantum dots are the example of zero dimensional material. In zero dimensional material most of the properties are size dependent properties. These materials can be crystalline or amorphous [11].

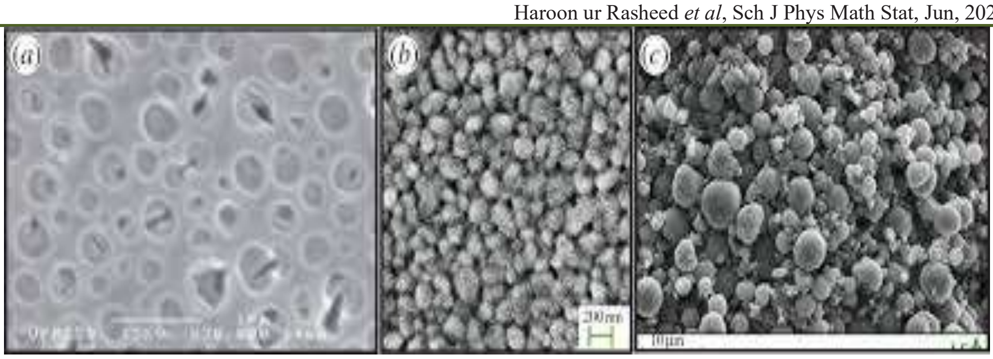


Fig. 1.2: Zero dimensional material[11].

In one dimensional material, electron motions are confined in two dimensions and canmove only in one dimension. Carbon nanotubes and nanowires are the examples of one dimensional material [11].

Fig. 1.3: One dimensional material [12]

In two dimensional material, the motion of electron is confined in one dimension and allow to move freely in other two dimensions. Quantum wells and thin films are the examples of two dimensional material[11].

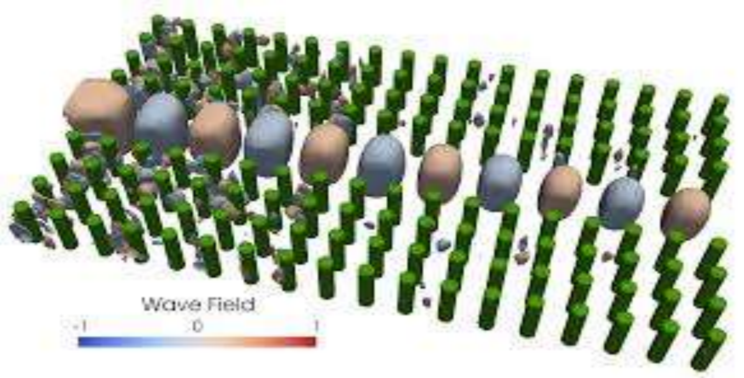


Fig. 1.4: Two dimensional material [13]

In three dimensional materials, the motiono of electron is not confined in any direction and can move in all directions. These materials are greater than 100nm.

1.6 Applications of nanotechnology in various fields

There are various applications of nanoparticles in different fields. Some are mentioned below,

Aerospace: Landing gear coatings, Thruster and body materials

Automotive: Corrosion-resistant coatings, Spark plugs

Consumers: Sport equipment, Computers, Hand held devices, Televisions, Cosmetics

Environmental: Pollution control, Water Purification, Ceramic membranes

Industrial Coatings: Environmental coatings (Chromium, Cadmium, Beryllium replacement) magnetic coatings

Medical: Implant coatings, Antimicrobials coatings, Sensors, Therapeutic drugs

Powers: Power transformers/transmissions, solar Panels

Fuel Cells (Microbial fuel cells)
Information Storage devices

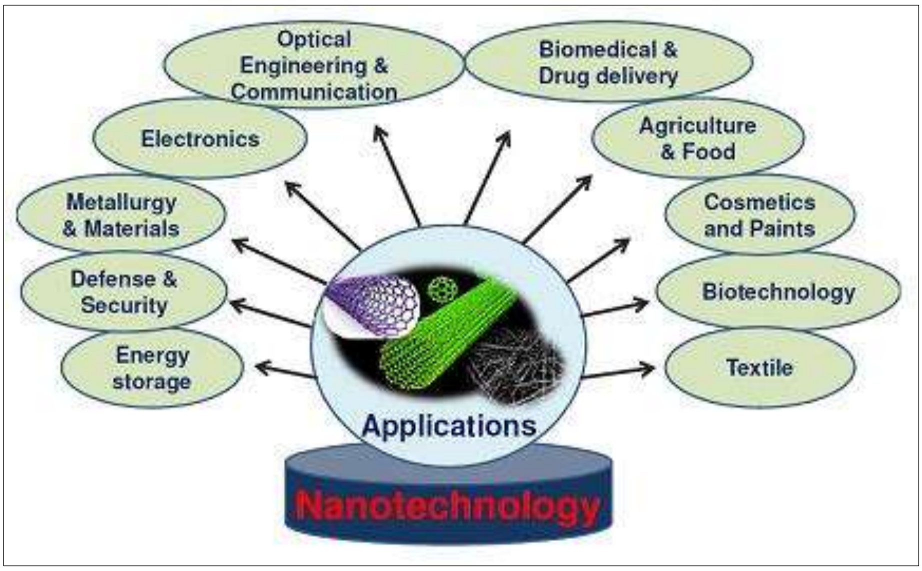


Fig. 1.5: Applications of nanotechnology [6]

1.7 Fabrication Techniques of Nanomaterials

There are two techniques or approaches for the fabrication of Nanomaterials.

1) Top down approach
2) Bottom up approach

In top down synthesis approach, nanoparticles can be prepared by mechanically grinding e.g. ball milling. These both approaches has some advantages and some disadvantages. The drawback of top down approach is that the ball milled nanoparticles contain some impurities such as imperfection of surface structure and significant crystallographic damage to the processed pattern. This drawback can make the top down approach difficult for fabrication. However, in the "bottom up" synthesis approach an ionic precursor is used to prepare nanoparticles e.g. by co-precipitation method, sol gel method and colloidal aggregation. By using this approach, we can control the particlesize distribution and can achieve a narrow particle size distribution. The illustration of Bottom up and Top Down approach is shown in Fig. 1.6.

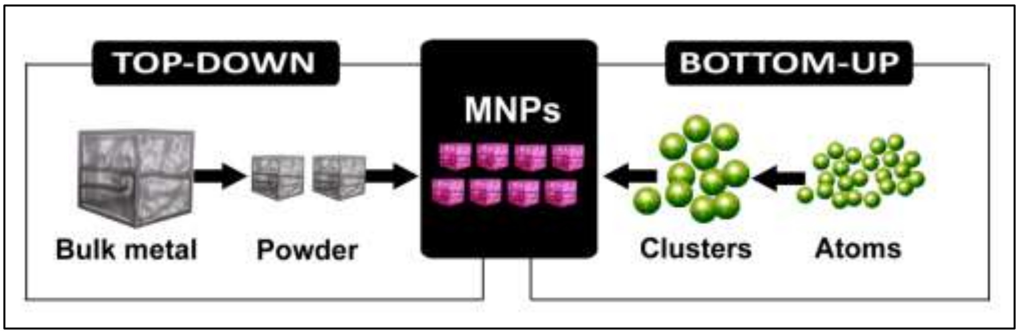


Fig. 1.6: Bottom up and top down approach [12]

1.8 Magnetic materials and Magnetism

Due to presence of electron orbits, the internal magnetic dipoles are produced in matter, which are oriented in random directions in the absence of external magnetic field but when external magnetic field is applied the internal magnetic dipoles align themselves along or against the direction of applied magnetic field which depends upon the type of magnetic material [15]. Therefore magnetization is qualitatively defined as production and alignment of internal magnetic dipoles along or against the direction of applied magnetic field [20]. Quantitatively magnetization is defined as "magnetic dipole moment per unit volume" of the magnetic materials. Mathematically

$M = m / V$………………………….…………. (1.1)

Where, "m" is magnetic dipole moment, "V" is the volume and M is the magnetization with unit A/m [16].

1.9 Classificationsof Magnetic Materials

On the basis of applied magnetic field and paired/unpaired electron, magnetic materials are classified into different classes. These classes are:

1) Diamagnetic

2) Paramagnetic
3) Ferromagnetic
4) Antiferromagnetic
5) Ferrimagnetic

1.9.1 Diamagnetism

Diamagnetism behaviour was discovered by Michael Faraday in 1847. He observed that there is some repulsion when the sample of bismuth is brought near the strong magnet [17].In diamagnetic material, the net magnetic moment due to spinning and orbital motions of electron in any atom is zero in the absence of an external magnetic field. When external magnetic field is applied on this material a force on the orbiting electrons is produced which causes the perturbation in the angular velocities. As a consequence, a net magnetic moment is created. This is a process of induced magnetization. According to Lens's law, the induced magnetic moment always opposes the applied field thus reducing the magnetic flux density. The macroscopic effect of this process is equivalent to that of a negative magnetization that can be described by negative magnetic susceptibility. Diamagnetism arises from the orbital motion of electrons within an atom and is present in all materials. In most materials it is too week for any practical application. Diamagnetic materials exhibit no permanent magnetism, and induced magnetic moment disappears when the applied field is withdrawn [16]. Examples of diamagnetic materials are Hydrogen (H), Sulphur (S), Helium (He), Neon (Ne) and Nitrogen etc. [14]

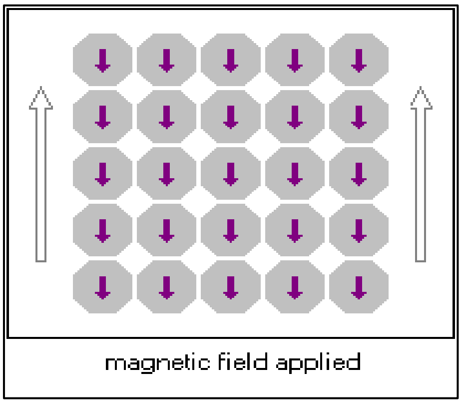
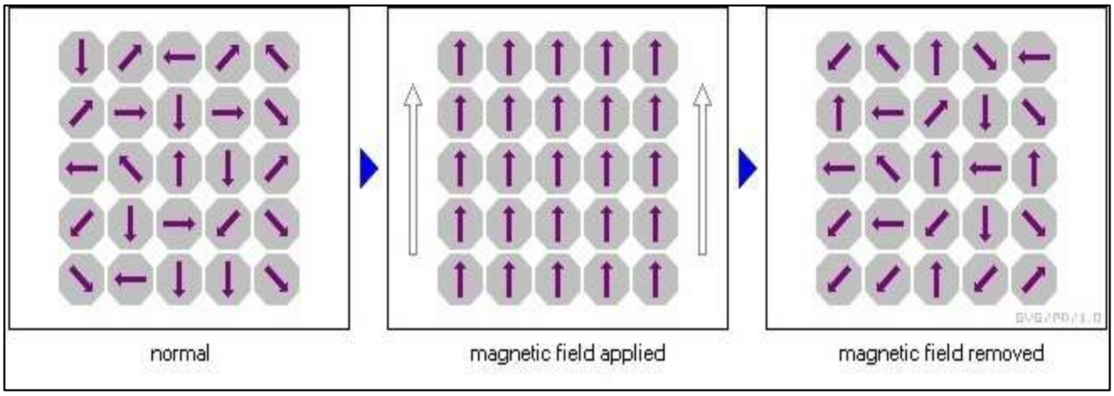


Fig. 1.7: Diamagnetic behaviour when magnetic field is applied [16]

1.9.2 Paramagnetism

Paramagnetism arises from the magnetic dipole moments of the spinning electrons. When magnetic field is applied the alignment forces acting upon the dipoles are counteracted by the deranging effect of thermal agitation. Paramagnetic effect is temperature dependent which is stronger at lower temperature where there is less thermal collisions [16]. In these type of materials the alignment of magnetic moment depends upon the strength of applied magnetic field [18]. Examples of paramagnetic materials are manganese, platinum, potassium, alkali and alkaline earth metals.



A B C

Fig. 1.8: Paramagnetic behaviour (a) absence of magnetic field (b) when magnetic field is applied (c) when magnetic field is removed [21]

1.9.3 Ferromagnetism

Ferromagnetism can be explained in term of magnetized domains. Ferromagnetic material is composed of many small domains ranging from few microns to about 1mm. In these domains, each domain containing about 10^{15} or 10^{16} atoms which are fully magnetized in the sense of that they contain aligned magnetic dipoles, resulting from spinning electrons even in the absence of an applied magnetic field. Quantum theory shows that there is strong coupling force exist between the magnetic dipole moments of the atom in a domain, which holds the magnetic dipole moments parallel. Between adjacent domains there is a transition region about 100 atoms called domain walls. In the absence of applied magnetic field the magnetic domains have different directions or oriented randomly. When external magnetic field is applied to a ferromagnetic material the walls of the magnetic domains having magnetic moment aligned with the applied magnetic field.

Ferromagnetism is due to strong coupling effects between magnetic dipole moments of the atoms in a domain. When the temperature of the ferromagnetic material is raised to such an extent that the thermal energy exceeds the coupling energy, the magnetized domain becomes disorganized. This temperature is known as "Curie Temperature" and represented by T_c [19]. At this temperature the ferromagnetic material behave as a paramagnetic substance. The curie temperature of iron is 770 ºC [17].

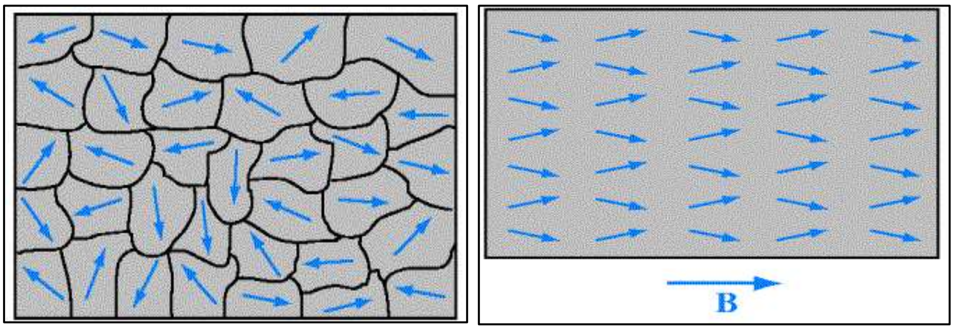


Fig. 1.9: Ferromagnetisn behaviour in the absence of magnetic field and when magnetic field is applied [10old]

1.9.4Antiferromagnetism

Antiferromagnetic materials belonging to that class of materials in which their magnetic moments are aligned in opposite direction and their net magnetization is zero. Antiferromagnetic materials exhibit antiferromagnetism at low temperature and become disordered above a specific temperature is called Neel temperature [10old]. Above this temperature antiferromagnetic material behaves like a paramagnetic substance. Manganese oxide is the antiferromagnetic material which changes in paramagnetic material at Neel temperature.

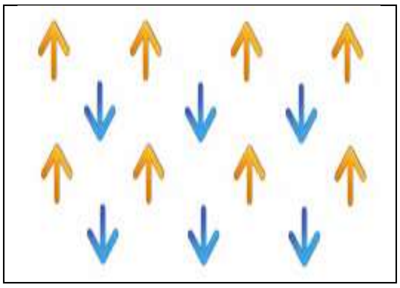


Fig. 1.10: Antiferromagnetic behaviour in the absence of magnetic field [22]

1.9.5 Ferrimagnetism

Ferrimagnetic materials correspond to that group of materials in which their magnetic moments are aligned in opposite direction and possess unequal magnitude of magnetic moment due to which there is a spontaneous magnetization. Below the Curie temperature Ferrimagnetism possesses Spontaneous magnetization and above that temperature these materials having no magnetic ordering [23].

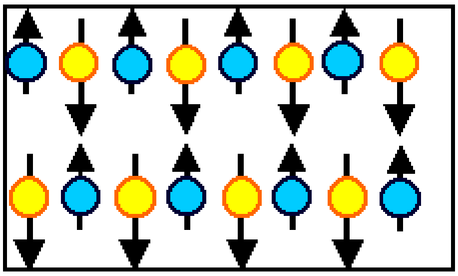


Fig. 1.11: Antiparallel alignment of magnetic moment in ferrimagnetic material [24]

1.10 Linear magnetic materials

Linear magnetic materials are those magnetic materials whose magnetization “M” is directly proportional to the magnetic field intensity “H”[16].

$M\ \alpha\ H$ …………………………………………...(1.2)

$M = \chi_m H$ ……………….…………………………(1.3)

Where χ_m is the magnetic susceptibility.

1.11 Bound Current and Free Current

The current produced in a magnetic material due to magnetization M is called bound current and current produced in magnetic materials not as a consequences of magnetization is called free current [15].

1.12 Hysteresis Loop

The word hysteresis is a Greek word which means lagging behind or deficiency. The ferromagnetic material contains a numbers of magnetic domains which is oriented randomly in the absence of externally applied magnetic field and net magnetization is zero. But when an external magnetic field is applied the magnetic domains start rotating with increasing in their size in case that the direction of domains is favourable with the applied field, and when the direction of domains is not favourable then size of domains decreases. As the applied field increases, the magnetic domains continue to grow until the material becomes a single domain which is oriented in the direction of applied field. At this state material has reached at saturation. If the applied field is reduced from saturation with reverse direction, the magnetization curve does not retrace in its original direction, it will retrace in another direction and form a closed loop which is called hysteresis loop [32]. When the applied field reaches to zero, the material may remain magnetized and this residual magnetization is called remanence M_r [21-23].

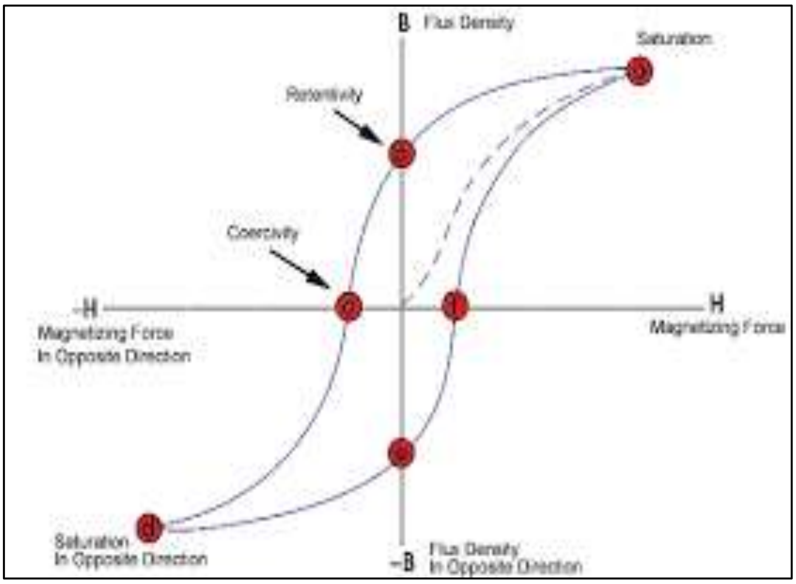


Fig. 1.12: Demonstration of hysteresis loop [22]

1.13 Ferrites

Ferrites are chemical compounds with a number of applications in engineering and material science. They are used for magnetic applications because they have ability to magnetize permanently. They have many types which depend upon the different properties. Ferrites are invented by two Japanese scientists Takeshi Takei and Yogoro Kato in Japan in 1930 during working in Tokyo Institute of Technology.

Ferrites have very fine dielectric properties and basically they are sand ceramic insulators. The general formula of ferrites is Fe_2O_3 which shows that they are combination of oxygen and iron. Ceramics are inorganic non-conducting materials. Sometimes material engineers add another element in ferrites for the enhancement of desired properties and then ferrites become NFe_2O_3, where N is the divalent ion added in the chemical composition of ferrites and divalent elements may be Co, Ni, Mn, Zn etc. Ferrites have many applications in our daily life due to their dielectric properties, electronic properties and magnetic properties[24]. As we are dealing with nanotechnology and nanoscience we should study ferrites at nanoscale. Ferrites have very outstanding dielectric properties at nano scale which depends upon the following factors[26].

1. Chemical composition
2. Method of preparation
3. Sintering process
4. Sintering time

1.14 Types of ferrites

On the basis of chemical and physical properties ferrites are divided into many types. On the basis of physical properties i.e. magnetic properties, ferrites have two types

1. Hard Ferrites
2. Soft Ferrites

1.14.1 Hard Ferrites

Hard ferrites cannot be easily magnetized and cannot be easily demagnetized as compared to soft ferrites. They have large hysteresis loop with large coercive force in contrast with soft ferrites. Permanent magnet is a best example of hard ferrites. If we apply a large amount of magnetic field to the material it becomes magnet and behaves like a permanent magnet because it cannot be easily demagnetized. The coercivity region of hard ferrites is greater than10 KA/m. Hard ferrites have a number of applications in our daily life such as communications devices, refrigerators, phase shifters, speakers and tunable devices like radios etc.[27]. Examples of hard ferrites are Barium ferrite, Cobalt ferrites and cobalt alloys.

Fig. 1.13: Hard ferrite as a permanent magnet [28]

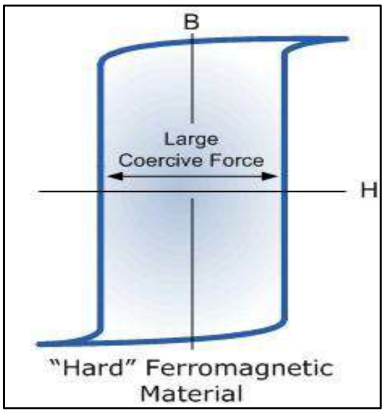


Fig. 1.14: Hysteresis loop for hard ferrites [29]

1.14.2 Soft Ferrites

When we apply a magnetic field on the material the magnetic dipole moments align in the direction of applied magnetic field. By gradually removing the applied magnetic field the magnetic dipole moments cannot regain the initial position and the material become a magnet itself. To remove this magnetism we need to apply magnetic field in opposite direction. The amount of magnetic energy required to make a material permanent magnet and demagnetize it again determine the type of ferrites.

To observe the relation between applied magnetic field 'B' and magnetization 'H' we draw a graph between B and H. A closed loop is formed which is called Hysteresis loop. Those ferrites which have small hysteresis loop called soft ferrites, which shows that they can be easily magnetized and demagnetised. They have small coercive force and energy loss will also be small. Their coercivity region is less than 1 KA/m. Soft ferrites are used for making iron cores for transformers and also used for making electromagnetic coils [30]. Examples of soft ferrites are Nickel-Zinc Ferrite and Magnesium ferrites.

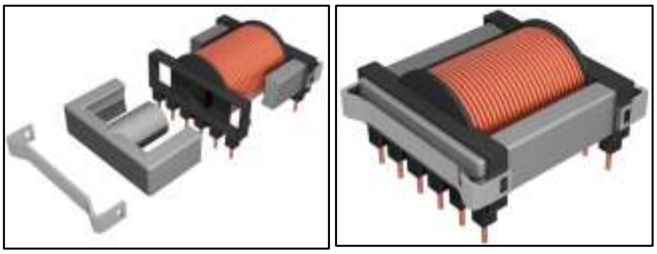


Fig. 1.15: Soft ferrites cores [30]

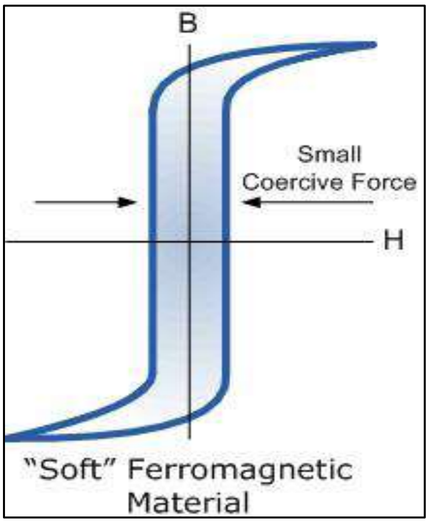


Fig.1.16: Hysteresis loop of soft ferrites [29]

1.15 Nickel Ferrite:

Nickel ferrite ($NiFe_2O_4$) is a type of soft ferrite, a class of magnetic materials with a spinel crystal structure. It is a ceramic compound made up of Nickel, iron, and oxygen, with the chemical formula ($NiFe_2O_4$)

Motivation: Ferrite nanoparticles are important for different applications such as targeted Drug delivery, Transformer core, Data storage, Hyperthermia, Ferrite beads, etc.

- High electrical resistivity
- Wide range of coercivity
- Tc lies above room temperature

Structure: - Nickel ferrite has a cubic structure belong to the inverse spinel family.

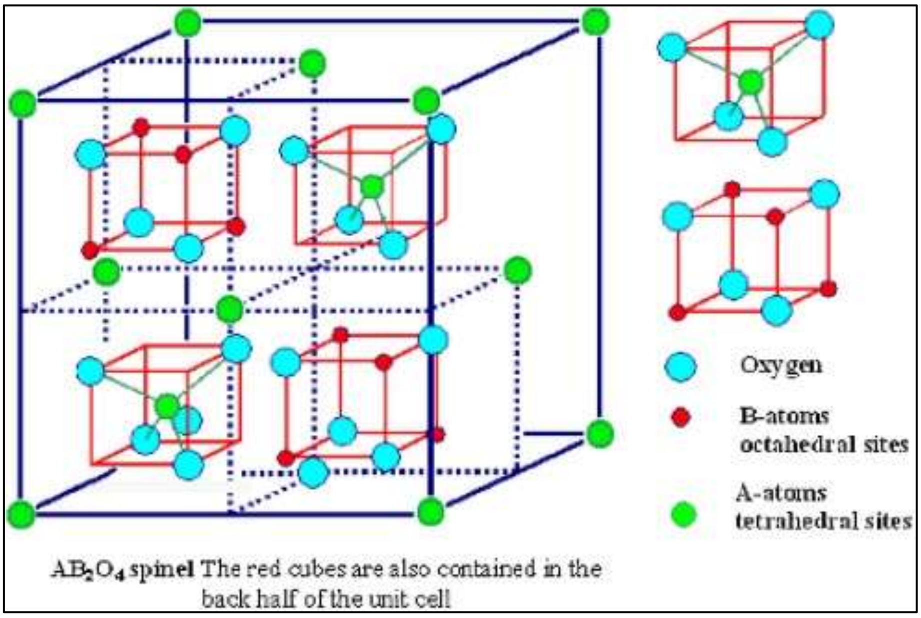


Fig. 1.17: Spinel structure of ferrites with tetrahedral structure &octahedral structure

Calcium: Calcium is a chemical element with the atomic number 20. It is a soft, silvery-white alkaline earth metal that is essential for many biologically and industrial process.

Structure:

- Face-cantered cubic (fcc) lattice structure
- Form compounds with other such as(CaO) ($CaCo_3$)
- Compounds form molecular structure like (ionic, covalent)

Use:

- It is essential for many biological and physiological process like:
- It is also use in industries including:
- Construction (cement, concert).
- Food process.

- Pharmaceutical.
- Cosmetic and skin care products.

Aims: -

- To examine the structure and dielectric properties of material.
- What are the effects of doped on its magnetic and dielectric properties.
- To examine their structure and how it's may helpful in different purpose.

Objectives: -

- We prefer Nickel ferrites because they are insulators and their Tc temperature and our basic need is that material have to be on room temperature
- To tune the dielectric properties by using dopant it enhances and modify the dielectric properties.

Characterization Techniques

We analyzed our material using various characterization techniques. The crystal structure may be ascertained using XRD, and the creation of nanoparticles can be verified using TEM. SQUID is utilized to characterize the sample magnetically. Below is a thorough discussion of various strategies.

3.1 X-Ray diffraction

3.1.1 Introduction:

The crystalline structure and lattice constants can be ascertained using X-ray diffraction, a non-destructive method. When X-ray beams are intact with the material, they scatter in X-ray diffraction. Their effect is amplified in multiple directions by these dispersed beams. These beams strengthen one another because of constructive interference. We call this procedure X-ray diffraction. A diagram of XRD is presented in Fig. 2.1.

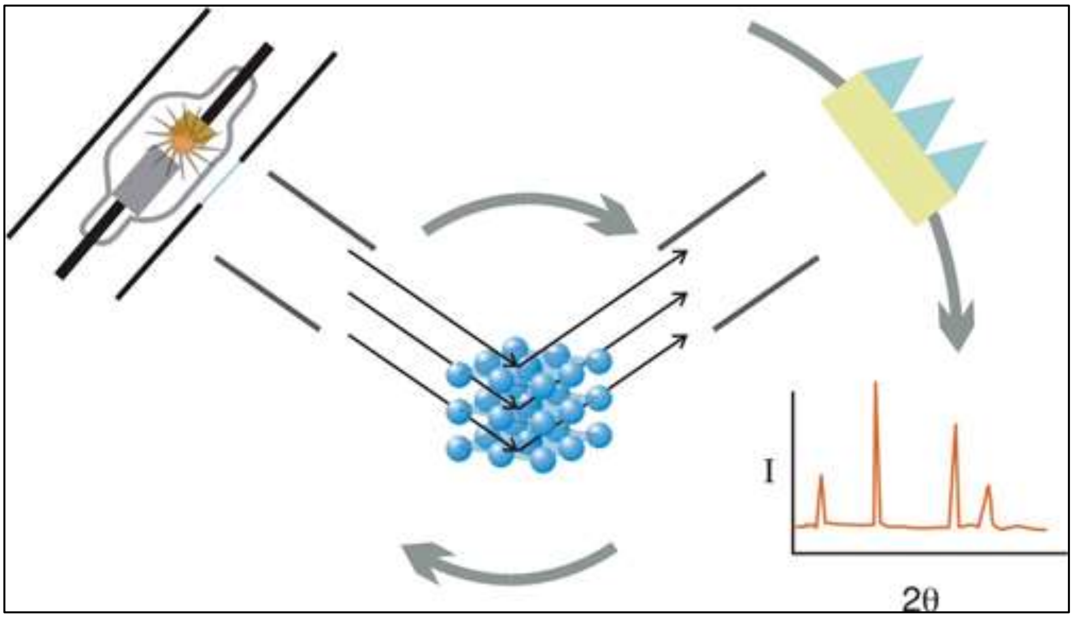


Fig. 3.1:Schematic diagram of X-ray diffraction[31]

The material's crystal is made up of atomic planes, which are the result of incident X-rays interfering with one another as they exit the crystal. We refer to this as X-ray diffraction. X-ray diffraction provides information about the internal arrangement of atoms in a crystal and defines its structural pattern.

3.1.2 Basic Principle:

As seen in Fig. 2.2, an X-ray beam impacting on the crystal and striking the atom will cause the electrons to oscillate. Both the incident beam frequency and the oscillation electron frequency will be the same. Destructive interference in certain directions indicates that the combining rays are out of phase. Contrary to this there will be constructive interference in few directions due to regular and periodic arrangement of atoms [32]. This is because the rays are in phase, which significantly affects the sample. Bragg's law provides an explanation for the XRD phenomenon of constructive interference.

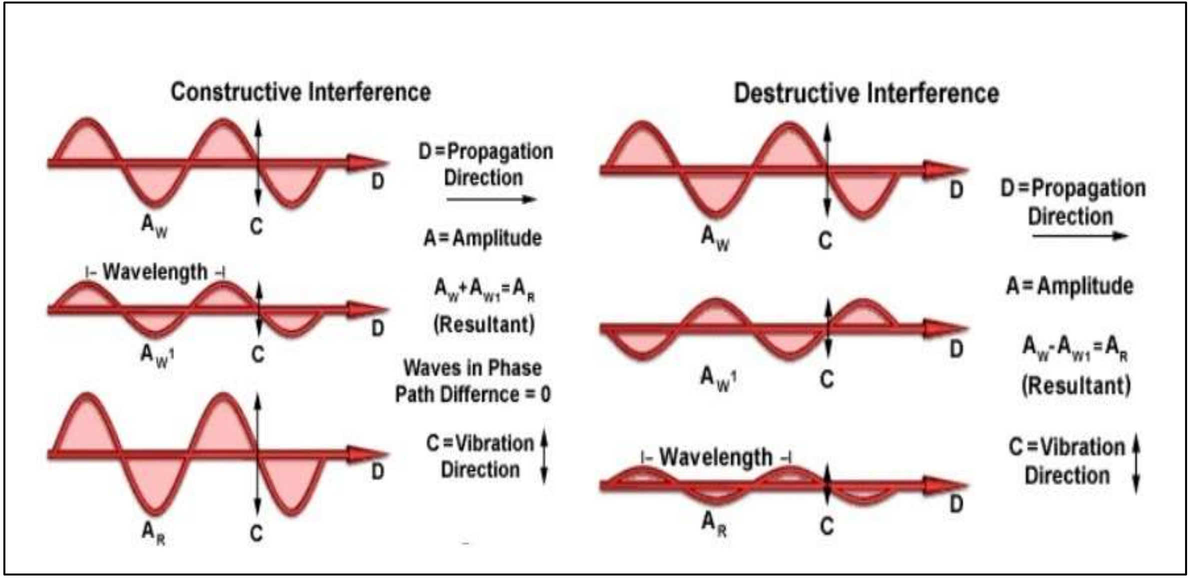


Fig. 3.2: Destructive interference (out of phase) & Constructive interference (in-phase)[33]

3.1.3 Bragg's Law:

Bragg's law was presented by W.H. Bragg and his son W.L. Bragg in 1913 and illustrates how X-ray interference results from beam scattering in a crystal. The given form of Bragg's law is,

$2d\sin\theta = n\lambda$……………………………..…………..(2.1)

Where,
λ = X-ray wavelength
θ = Angle of scattering
n = integer multiple 1,2,3…….

Figure 2.3 provides an illustration of Bragg's rule by demonstrating the existence of two atomic layers separated by a distance of d. When a pair of X-ray beams strike these two layers, they scatter at an angle θ and reflect by traveling an extra distance, as depicted in Figure 2.3.

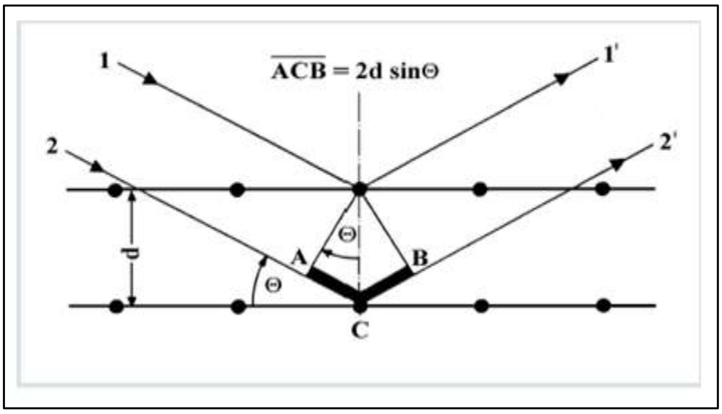


Fig. 3.3: Bragg's law representation [34]

Bragg's law requires that the incidence angle equal the reflecting angle, the extra distance be an integral multiple of the wavelength, and the two reflected beams be in phase. Diffraction will happen when these requirements are all met, and the phenomenon can be mathematically described as,

$n\lambda = 2d\sin\theta$…………………………..…….(2.2)

If we have a simple cubic crystal (a=b=c=a) then we can find lattice parameter as
$a = d / [h^2+k^2+l^2]^{1/2}$…………………….…….(2.3)

where,
h, k and l are miller indices
a is the lattice parameter
d is the distance between the planes

XRD has many applications in material science. The most important applications are

1) Atomic arrangement can be determined by XRD.
2) Average spacing between two layers can be measured.
3) Crystalline phases can be identified by XRD.
4) Integrated intensities can be obtained by XRD.

5) Spacing between the layers can be measured.
6) Crystal structure of unknown materials can be measured.

3.2 Dielectrics:

Dielectric Measurements: During experiment we used Impedance analyser 6500B for measuring the dielectric properties of the sample. The frequency range of this instrument is about 1 kHz – 2 MHz. Its accuracy is about ± 0.05%. The sample pellet is placed between the two metal plates and a desire frequency is applied on it. We set the desired parameters which are available on the screen and we get the data in the form of digit. Different parameters can be calculated through this instrument such as capacitance, resistance, inductance etc. For storing the measuring data USB was used.

Fig. 3.4: Impedance analyser 6500B for measuring the dielectric properties of the sample

METHODOLOGY

4.1 Sol gel synthesis:

Very first step two beakers, one filled with Citric Acid ($C_6H_8O_7$) 5.88g and Distilled Water 20ml.Second one filled with Ethanol 20ml and fill it with 2.09g of Nickel Nitrate,0.467g of Calcium Nitrate and 8g of Iron Nitrate. Stir both the beakers and add the first beaker solution into second one dropwise. By adding Ammonia we fix its PH value by 7. The citric acid ratio is 1:1. The solution is then heated at 80°C with continuous stirring until a clear gel formed. The gel is a colloidal suspension of metal ions and citric acid.

Fig 4.1 Formation of gel.

The mixture is then heated to room temperature, allowing it to form a gel. The gel is a three-dimensional network of metal ions and citric acid. The gel is dried in at room temperature at 130°C for 2 hours to remove excess water. The resulting material is a dry gel. The dry gel is then subjected to auto-combustion in air at 300°C.During this process, the gel undergoes a rapid and self-sustaining combustion reaction, resulting in the formation of nanoparticles. Grand this crystal solution in Mortar by the help of Pestle after grinding solution in the form of powder and ready for calcination. The resulting powder of Ca doped $NiFe_2O_4$ nanoparticles are then calcinied at 950°C for 24 hours to remove any remaining organic material and to crystallize the nanoparticles. The nanoparticles are then characterized using X-ray diffraction (XRD). The resulting Calcium-doped Nickel ferrite nanoparticles have a spinel structure and exhibit excellent magnetic and electrical properties, making them suitable for various applications such as magnetic resonance imaging, drug delivery, and electronic components.

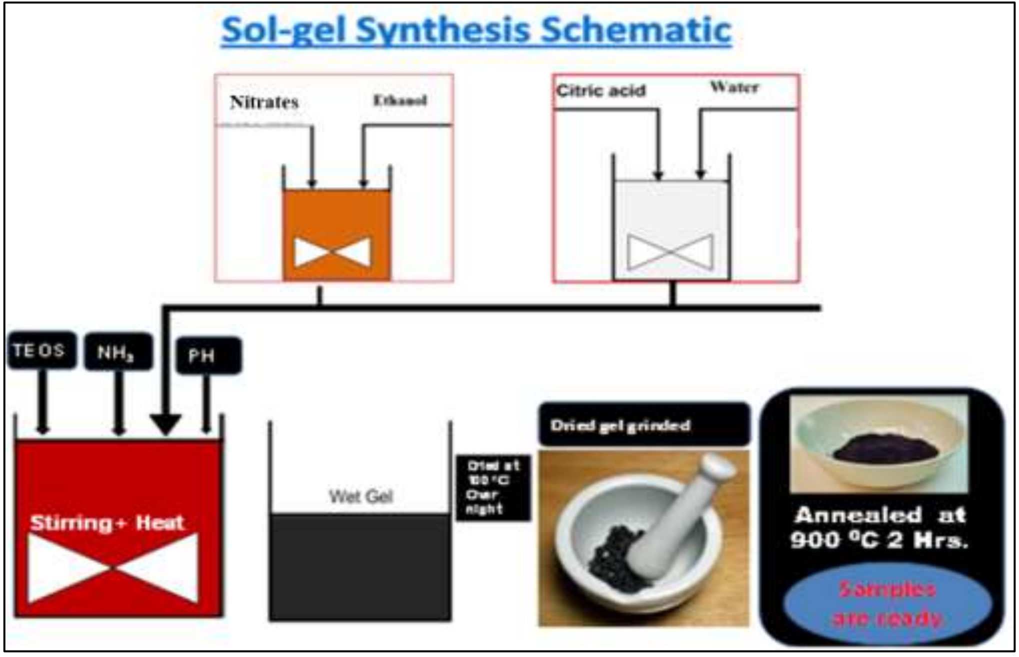


Fig. 4.2: Sol gel synthesis technique of the sample

RESULTS AND DISCUSSIONS

We have prepared two samples Nickel ferrite and calcium doped Nickel ferrite having general formula $Ca_xNi_{1-x}Fe_2O_4$ (x= 0, 0.2,) via sol-gel synthesis method and were subjected to different characterization techniques to investigate their structural and dielectric properties. The structural properties of $NiFe_2O_4$and $Ca_xNi_{1-x}Fe_2O_4$ (x= 0, 0.2,) were examined by XRD. The frequency dependent dielectric measurements were done by using Impedance analyser. The brief discussion of all the obtained results of measurements are given below.

XRD Analysis

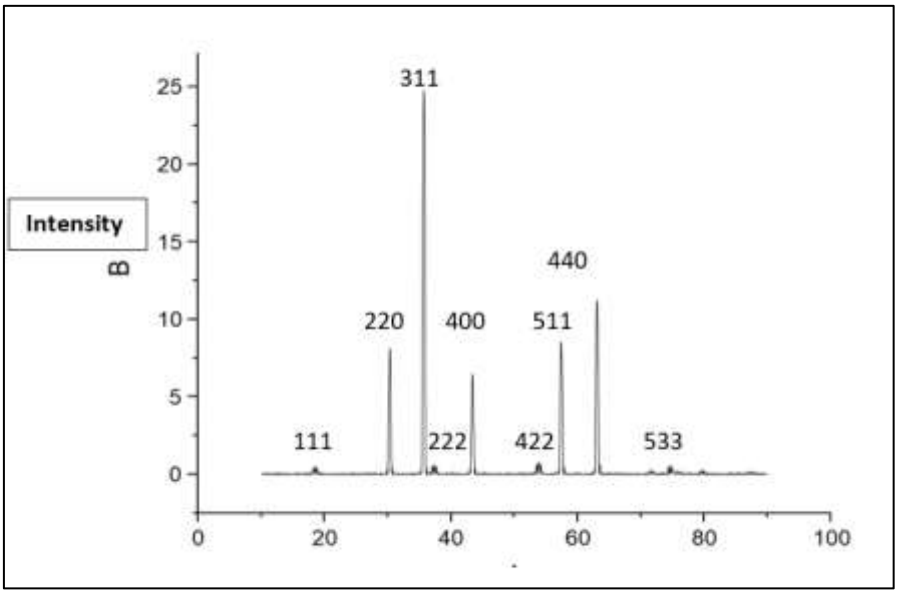


Fig. 5.1 :X-ray diffraction pattern of Pure $NiFe_2O_4$ nanoparticles

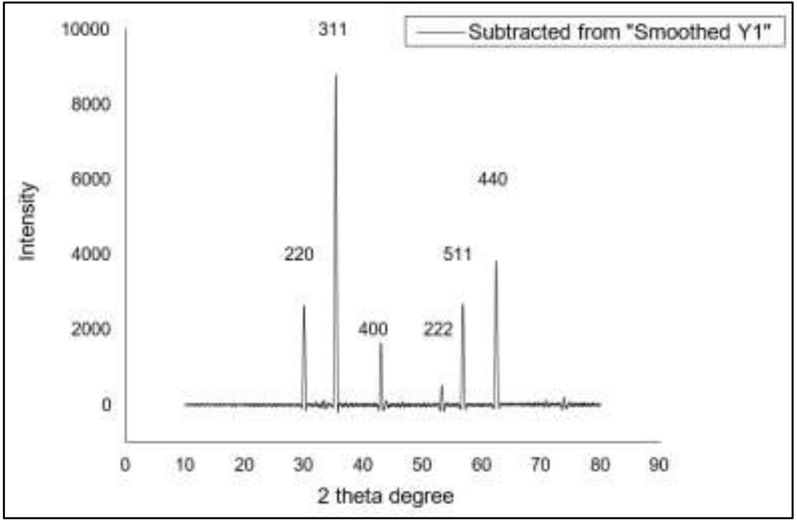


Fig. 5.2:X-ray diffraction pattern of doped $NiFe_2O_4$ nanoparticles (theta =1.14nm)

XRD is a well-known technique used to study the crystal structure and to find the average crystallite size of nanoparticles. The XRD pattern of $NiFe_2O_4$ and Ca doped $NiFe_2O_4$ nanoparticles shown in the Fig. 3.2 and 3.3

The XRD pattern of $NiFe_2O_4$ resulted the peak values (111), (220), (311), (222),(400) (422),(511),(440) and (533) at the angle of 2θ=19.1^{0},30.2^{0}, 37.7^{0}, 38.20,42.2^{0}, 55.2^{0},58.9^{0},62.4^{0} and 85.3^{0}. Similarly $Ca_xNi_{1-x}Fe_2O_4$ (x=0.2) (1-x=0.8) nanoparticle. The indexed peaks (220), (311), (400), (222), (511) and (440) with the miller indices of various planes of Calcium dopped Nickel Ferrite at the diffraction angles 2θ = 30.64^{0}, 35.69^{0}, 41.2^{0}, 53.6^{0} „57.66^{0} and 62.4^{0}.

X-ray diffraction peaks were confirmed by match with JCPD card number of 06-0545 NiFe2O4 phases. The XRD peaks of Nickel ferrite nanoparticles were found to be matched with the pattern of the cubic spinel structure. The Higher peaks in XRD pattern describe that the diffraction takes place from those planes having greater concentration of lattice atoms.

Average Crystallite size Calcium doped Nickel ferrite nanoparticles is measured by using Debye-Scherrer's formula,

$$Dp = k\,\lambda\,/\,B\cos\theta$$

Where Dp = Average crystallite size (nm)=16.47(nm), K = Scherrer constant=0.94, λ = X-ray wavelength=1.54059 Å, *B*= FWHM (Full Width at Half Maximum) of XRD peak, θ =XRD peak position, Average crystallite size (nm) of NiFe2O4 = 25.28nm. Average crystallite size (nm) of Ca dopped NiFe2O4 = 20.79nm. There is a small shifting in peaks arises due to the difference in atomic radii of the Nickel and Calcium 0.128 nm.

5.2 Dielectric Analysis:

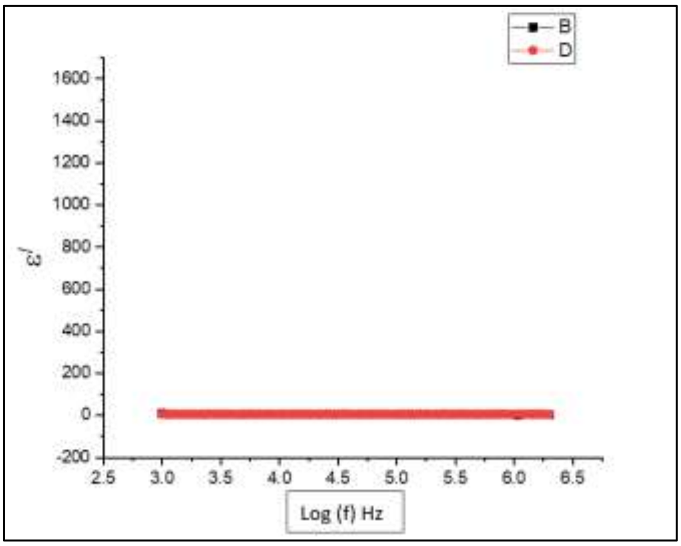


Fig. 5.3: Dielectric Analysis

Dielectric properties represented by the real part of dielectric constant (ε'), the imaginary part dielectric constant (ε'') and the loss factor (tan) in addition to the alternating conductivity (σac) of the sample $NiFe_2O_4$ was studied as a function of frequency at room temperature in a frequency 1kHz-2MHz range. The real and imaginary dielectric constant have been calculated by the following equations.

$$\mathcal{E}, = - / 0_{\circ}\ 9$$
$$\varepsilon'' = C_p.d/A.\mathcal{E}$$

Where C is the capacitance of the pellet, d is the thickness, A is the cross-sectional area and $\varepsilon_{\circ}$ is the permittivity of free space ($\varepsilon_{\circ} = 8.854\times10^{-12}\ C^2/Nm^2$).

Frequency for $NiFe_2O_4$ at 1100°C for 4 h. It is seen that the real part of dielectric constant value of the sample decreases with increasing frequency and then it remains constant at high values, which is the normal behaviour of most ferrites. The high values of the real dielectric constant at low frequencies are due to the polarization of the space charge due to the large mass of its dipoles compared to other types of polarization (electronic, ionic, directional) and when increasing the frequency, these dipoles cannot rotate with the electric field and thus the dielectric constant decreases. This behaviour of ferrite has been observed by many researchers.

CONCLUSION

In conclusion, the research on calcium doping of nickel ferrite nanoparticles enhanced dielectric properties. The structural analysis confirms that the Nickel ferrite nanoparticles crystal is in cubic form without forming other secondary phases. The dielectric results showed that these nanoparticles are suitable for high-frequency applications. The study demonstrates the potential of calcium doping as a strategy to improve the performance of nickel ferrite nanoparticles, paving the way for advancements in microwave absorbers, phase shifters, and other electronic devices.

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