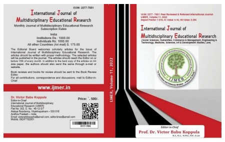
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A REVIEW ON SYNTHESIS AND CHARACTERIZATION OF MNZN FERRITE NANOPARTICLES VIA CITRATE PRECURSOR METHOD

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Abstracts

Researchers are taking great interest in the synthesis and characterization of MnZn ferrites due to their wide range of applications in many areas. MnZn ferrites are a class of soft magnetic materials that have very good electrical, magnetic and optical properties. The properties of MnZn ferrites include high value of resistivity, permeability, permittivity, saturation magnetization, low power losses and coercivity. The above-mentioned advantageous features of MnZn ferrites make them suitable for the use in various applications. In biomedical field these ferrites are used for cancer treatment and MRI. MnZn ferrites are also used in electronic applications for making transformers, transducers and inductors. These ferrites are also used in magnetic fluids, sensors and biosensors. MnZn ferrite is highly useful material for several electrical and electronic applications. It finds applications in almost every household appliance like mobile charger, LED bulb, TV, refrigerator, juicer mixer, washing machine, iron, microwave oven, mobile, laptop, desktop, printer and so on. Therefore, the present review focuses on different techniques for synthesis of MnZn ferrites in literature, their characterization tools.

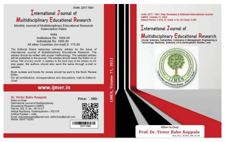
Keywords: Resistivity, Permeability, Permittivity, Coercivity, XRD, Citrate Precursor Method.

1.1 Introduction

Nanoparticles are the particles amidst 1 and 100 nanometer in size. In nanotechnology, a particle is defined as a tiny object that behave as a entire unit with respect to its transport and properties. Nanoparticles have uses in biomedical, optical and electronics fields [1]. Nanoparticles are of great scientific interest as they are effectively a bridge amidst bulk materials and atomic or molecular structure. Nanoparticles can exhibit size related properties significantly apart from those of bulk materials [2]. This is typically because nanoparticles have a great surface area per weight then huge particles which cause them to be more reactionary to other molecules [3]. Nanoscience and Nanotechnology are two terms related to materials at atomic, molecular scale whereas Nanotechnology are designed, functionalization, characterization and make use of material at the nanometer scale. So, in the field of nanometer, the nanometer is classified according to their diameter.

Nanoparticles are of great scientific interest as they are effectively a bridge amidst bulk materials or atomic and molecular structure. Nanoparticles can exhibit size related bulk materials or atomic and molecular structure. Nanoparticles can exhibit size related properties significantly apart from those of either fine particles or bulk materials. The property of nano-conventional material change when formed from nanoparticles. This is typically because nanoparticles have a great surface area per weight then large particles which cause them to be more reactive to some other molecules. Nanotechnology is made up of two words Nano+ Technology, Word Nano indicates something that is so small that it can only be seen with a powerful microscope. Nanotechnologyis the study and use of structure between 1 nm to 100 nm in size. Mr. Richard P. Feynmanis known as the father of Nanotechnology. Important properties of materials such as the electrical, optical, thermal and mechanical properties, are determined by the way molecules and atom assemble on the Nano scale in them on macro scale, because quantum mechanical effect become important. Nanotechnology is so important because it could have the potential to solve many of the problems, such as microchips capable of storing millions of bytes of information in a device as small as the tips of a pin. These implications of nanotechnology are wide ranging and could include medicine, military applications, computing and astronomy. Preparation and applications of Nano sized material are scientifically and industrial interest due to their unique and enhanced properties present, in scientifically and industrial interest due to their unique and enhanced properties present, in scientific knowledge and commercial applications. At the Nano scale, the properties may change in uncertain way.

MnZn Ferrites are preferred over other ferrites due to their low cost and wide range of applications. These Ferrites are very important for stress insensitivity and low noise and are generally used for applications where frequency requirements are below 2MHz. MnZn ferrites are also advantages due to their almost zero magneto crystalline anisotropy. In the class of soft ferrites, MnZn ferrites are preferred due to high permeability, saturation induction, low power losses and high magnetic induction. MnZn ferrites are of great interest due to their wide range of applications such power applications, magnetic field, high frequency power supply, memory storage devices, TV sets, biomedicines etc. there is a continuous progress in the size and shape control of MnZn ferrites and also on the morphological and magnetic properties of MnZn ferrites by using different methods and many more other methods. The study provides an update survey of the main synthesis method of MnZn ferrites nanoparticles in order to obtain Nano-system for various electronics applications for making transformer, transducers and inductors. Below are the ways Nanotechnology impact our lives on a daily basis.



- Faster, smaller and more powerful computers that consume far less power with longer lasting batteries.
- Water filters that are only 15-20 nanometer wide can remove all the virus and bacteria.
- Carbon Nanotubes have a variety of uses, including making sports equipment stronger and lighter weight.
- Most sunscreens today are made from Nanoparticles that effectively absorb light, they also spread more easily over the skin.

1.11 Ferrites

A Ferrites is a ceramic material that is made up of iron oxide (Fe_2O_4) in a large proportion mixed with metallic elements such as barium (Ba), manganese (Mn), nickel (Ni), zinc (Zn), in small proportions. The nature of both the iron oxide and the metal oxide is electrically non-conducting and ferri-magnetic. Ferrimagnetic material is one that possesses unequal opposing magnetic moments which allows such materials to retain spontaneous magnetization. Ferrites are generally classified into two types: Hard Ferrites and Soft Ferrites, based on their crystallography structure.

1.12 Hard Ferrites

Hard Ferrites have high corecivity and such materials are difficult to magnetized. Therefore, these materials are used in making permanent magnets which are used for applications in refrigerator, loudspeaker, washing machine, T.V, switch mode power supply, communications system, absorbing system, high frequency applications, refrigerator, loudspeaker etc. In contrast, permanent ferrites magnets are made up of hard ferrites which have high resonance after magnetization. These are composed of iron and barium oxides. They are cheap, and widely used in household products such as refrigerator magnetics. The most common hard ferrites are Strontium ferrite, Barium ferrite, Cobalt ferrite.

1.13 Soft Ferrites

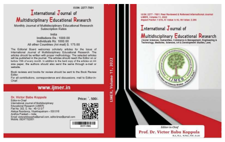
Soft ferrites have low corecivity as a result of which their magnetization can easily be altered. Soft ferrites are good conductor of magnetic field which has led to its wide range of applications in electronic industry such as developing Transformer core, high frequency, inductor and as microwave components. Furthermore, advantages of soft ferrites include high resistivity, low cost, time and temperature stability, low loss and high permeability. Most common ferrites are MnZn ferrites, NiZn ferrites etc. the low corecivity means the materials magnetization can easily reverse direction without dissipating much energy (hysteresis losses), while the materials high resistivity prevents eddy currents in the core, another source of energy loss.

1.14 Manganese Zinc Ferrites

Ceramic Ferromagnetic materials of MnZn ferrites family are the most broadly known category of soft electromagnetic materials. They are polycrystalline materials crystallized in the cubic structure similar to that of the mineral "Spinal". Their magnetic properties arise from interaction between the magnetic dipoles of the metallic irons (Fe, Mn), that have uncompensated spin electron which means that their spins are antiparallel but their magnitude are not same and because of this uncompensated anti-ferromagnetism their net magnetic moment is not zero and this magnetic moment gives spontaneous magnetization in ferrites. MnZn ferrites are most preferred as they have high permeability and saturated magnetization as compared to NiZn ferrites. Because of low value of resistivity of MnZn used for low frequency applications. The properties of MnZn ferrites are essentially dependent on the synthesis method and doping concentrations inside Nano ferrites. MnZn ferrites suitable to be used in many applications of daily life. The change in concentration of cations and sintering conditions change the magnetic, electrical properties and structural properties of Nano ferrites which leads to its wide range of applications. In additions, the shape, morphology, electrical. Magnetic properties are affective by the cation distribution in MnZn ferrites.

1.15 Crystal Structure

The spinel AB_2O_4 structure can be generally described as a cubic close pack arrangement of oxygen ions in which tetrahedral A and octahedral B interstitial lattice sites are occupied by cation. In Normal spinel, the tetrahedral sites are occupied by divalent cations while trivalent cations occupy octahedral sites. In the contrast, 2-Valent cations occupy octahedral sites in inverse spinel, whereas 3-Valent cations are distributed equally among A and B sites. In ferrites, Fe^{3+} serve as the trivalent cation. Generally, the structure of spinel ferrites could be described with the $(Me^{2+}_{1-x}Fe^{3+}_{x})[Me^{2+}_{x}Fe^{3+}_{2-x}]O_4$ formula, where round and square brackets represent tetrahedral and octahedral sites respectively. The degree of inversion X is the proportion of the trivalent cationFe^{3+} occupying tetrahedral sites. MnZn ferrite have spinel structure. The spinel structure has one major unit cell composed of 8 subunit cells having Face Centre Cubic (FCC) structure with two types of sites in each unit cell i.e tetrahedral (A) site and octahedral (B) site in the complete structure of MnZn ferrite. Tetrahedral (A) sites are surrounding by 4 neighbor oxygen atoms and octahedral (B) sites have 6 nearest oxygen atoms around it as shown in figure 1.



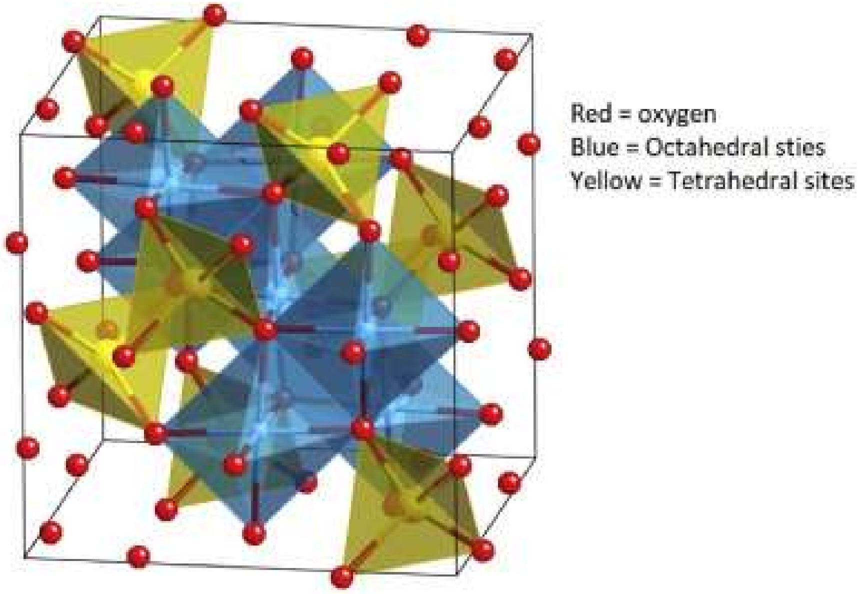


Figure 1 Crystal Structure of Ferrite

In MnZn spinel ferrite lattice, Zn ions are on the tetrahedral sites while Fe and Mn ions occupy both tetrahedral and octahedral sites. Due to this spinel structure, different metallic ions can be introduced that causes change in the electric and magnetic properties of ferrites. The metal ions introduced may enter the spinel structure by replacing Fe^{3+} ions. The magnetic properties of the spinel ferrites materials originate from the antiferromagnetic coupling between the octahedral and tetrahedral sub-lattices. Diamagnetic Zn^{2+} is usually used in mixed ferrites to decrease the magnetic moment of A-sub-lattice, thus increasing the overall magnetization of the crystal. Pure $ZnFe_2O_4$ bulk material is paramagnetic at room temperature, since it has the normal spinel structure with Zn incorporated almost exclusively interaction between the two sub-lattice which would result in ferrimagnetism. In the pure $MnFe_2O_4$ ceramics, approximately 80% of Mn enters the spinel structure at tetrahedral sites and 20% are enter at octahedral sites $(Mn_{0.8}Fe_{0.2})[Mn_{0.2}Fe_{0.8}]O_4$.

The structure of Nanoparticles has been shown to differ significantly from that of the cross-ponding bulk materials. For example, $ZnFe_2O_4$ become ferromagnetic, when prepared in the form of nanoparticles, because it adopts a partially inverted spinel structure. The structure of ferrites nanoparticles with size ~ 10 nm, studied so far, is sufficiently closed to the structure of the bulk spinel, so that ceramic standard has been used to calibrate some parameters of the structure model. In order, to know the best advantages of MnZn ferrites for various applications, one has to be aware of different synthesis and characterization technique.

1.16 Applications of Ferrites

- Ferrites are used in digital computers and data processing circuits. Ferrites are used to produce low frequency ultrasonic waves by magnetostriction principle.
- Ferrites are used to make permanent magnets for applications such as refrigerator magnets, loudspeakers and small electric motors.
- Ferrites are used in electronic industry to make efficient magnetic core called ferrites core for high frequency inductors, transformers and antennas, and in various microwave components.

1.17 Literature review

Nanoparticles research are currently an area of intense scientific research, due to a wide variety of potential applications in biomedical, optical and electrical field. The word “Nano” is a Greek word synonymous to dwarf meaning extremely small. The properties of materials change as their size approaches to Nano-scale. Nanomaterial often shows unique and considerable change in physical, chemicals, biological properties compared to their micro scale counterparts [4]. Nanoparticles often has unexpected visible properties because they are small enough to confirm their electrons and produce quantum effect [5]. For example, Gold appears deep red to black in solution [6]. Nanoparticles are very high surface area to volume ratio and this provide a tremendous driving force for diffusion, especially at elevated temperature [7].

Properties of ferrites are known to be sensitive to the processing technique [8,9]. Soft ferrites such as Mn-Zn, Ni-Zn and Mg-Mn are well known ferrites covering wide ranging applications [10,11]. Electrical conductivity in Mn-Zn ferrites has been attributed to electron hopping [12] between the two valence states of iron, $Fe^{2+} \leftrightarrow Fe^{3+}$an octahedral site. A nanoparticle is a ball or ball like molecules which consist of 10 to 100 atoms interconnected by inter atomic forces. Due to fascinating properties of MnZn ferrites among the class of soft ferrites like high value of saturation, magnetization, low value of coercivity, high initial permeability narrow size distribution of the ferrite's particles, the researchers taking interest in the synthesis of these ferrites. Synthesis of nanosizeMnZn ferrites particles from metal nitrate solution through citrate precursor method are required. Soft ferrites have been prepared by a wet chwmical process, known as the citrate precursor method, which involves the formation of citrate complexes of the constituent cation. The electrical and magnetic measurement on MnZn ferrites have shown electrical resistivity higher by more than two orders of magnitude compared to that observed for sample prepared by conventional ceramic method and low magnetic losses. Making these ferrites specially suitable for higher frequency applications. The saturation magnetization, initial permeability and curie temperature are found to be more in precursor method as compared with the ceramic method because of uniformity and homogeneity in grains and smaller grain size in precursor method. Curie temperature and saturation magnetization has shown initial increase and then decrease with sintering temperature. These change in properties are explained on the basic of various theories and models. Nanomaterials particularly with magnetic properties are great interest for their applications in variety of areas such as magnetic refrigeration at high level temperature [13]. Huge density information storage [14], color imaging [15]. Ferrofluids [16], bioprocessing [17], medical diagnosis [18], electromagnetic wave absorption [19] etc. Generally metal nanoparticles can be prepared and stabilized by physical and chemical approach such as chemical reduction, electrochemical technique and photochemical reduction is most widely used [20]. Number of applications are available for the synthesis of nanoparticles, such as reduction of metal salt in aqueous phase micro-emulsion approach, sol-gel technique, hydrothermal technique several other parameters such a temperature, the incident light, the nature of metal salts and reaction conditions are also reviewed.

Objective

- To synthesis Manganse-Zinc ferrite nanoparticles via citrate precursor method.
- To Investigate the structural properties of manganese-zinc nanoferrite.
- To investigate the (a) the lattice parameter (b) x-ray density, the hopping length of (A) site, the hopping length of octahedral (B).

2. Material and Methods

2.1 Materials

The following materials are used in the preparation of the MnZn ferrites;

- Manganese nitrate
- Zinc nitrate
- Iron(III) nitrate
- Hydrated Citric acid
- Distilled water

2.12 Citrate Precursor Method

Polymeric citrate precursor method, a chelated – based method involves the reaction between mixed cations with citric acid, and then these cations are cross-linked with the help of ethylene glycol for the esterification process. Gel composites were heated whichBurns the organic moieties leaving behind the nanoparticles, and burning gels become essential for the reduction of nanoparticles. The prepared nanoparticles by using such low cost elegant methods are uniform with a small size distribution, reproducible with good yield as per the demand applications.The low temperature synthesis methods such as polymeric citrate precursor are efficient enough to control the preparation of novel nanoparticles with morphological differences that leads to the novel devices with desired technological performance.These method are very simple, less expensive and are easy to handle to operate for the synthesis of nanoparticles as per the expected morphology and dimensions.This method have better control for the desired shape morphology and size and provide many opportunities for the use of these prepared nanoparticles in various aspect of science and technology.

2.13 Sample Preparation of MnZn Ferrite

In a stoichiometry ratio, Manganese nitrate, zinc nitrate, was prepared in 100ml of distilled water on beaker. Iron (III) nitrate was dissolved in distilled water by warming at 40^0C. Citric acid was added to manganese nitrate and zinc nitrate separately. The solution is placed on magnetic stirrer for 20 minutes and then added few drops of ammonia using burette to maintain PH level. The precursor mixture was heated at 40^0C with constant stirring to obtain a dried product of uniformly brown color. After cooling at room temperature, the precipitate was calcinated in box type furnace for 2h at 500^0 C to obtain a ferrite powder. These samples were sintered at 700^0C at a heating rate of 250C/h. The final product was collected for characterization

2.14 Methodology

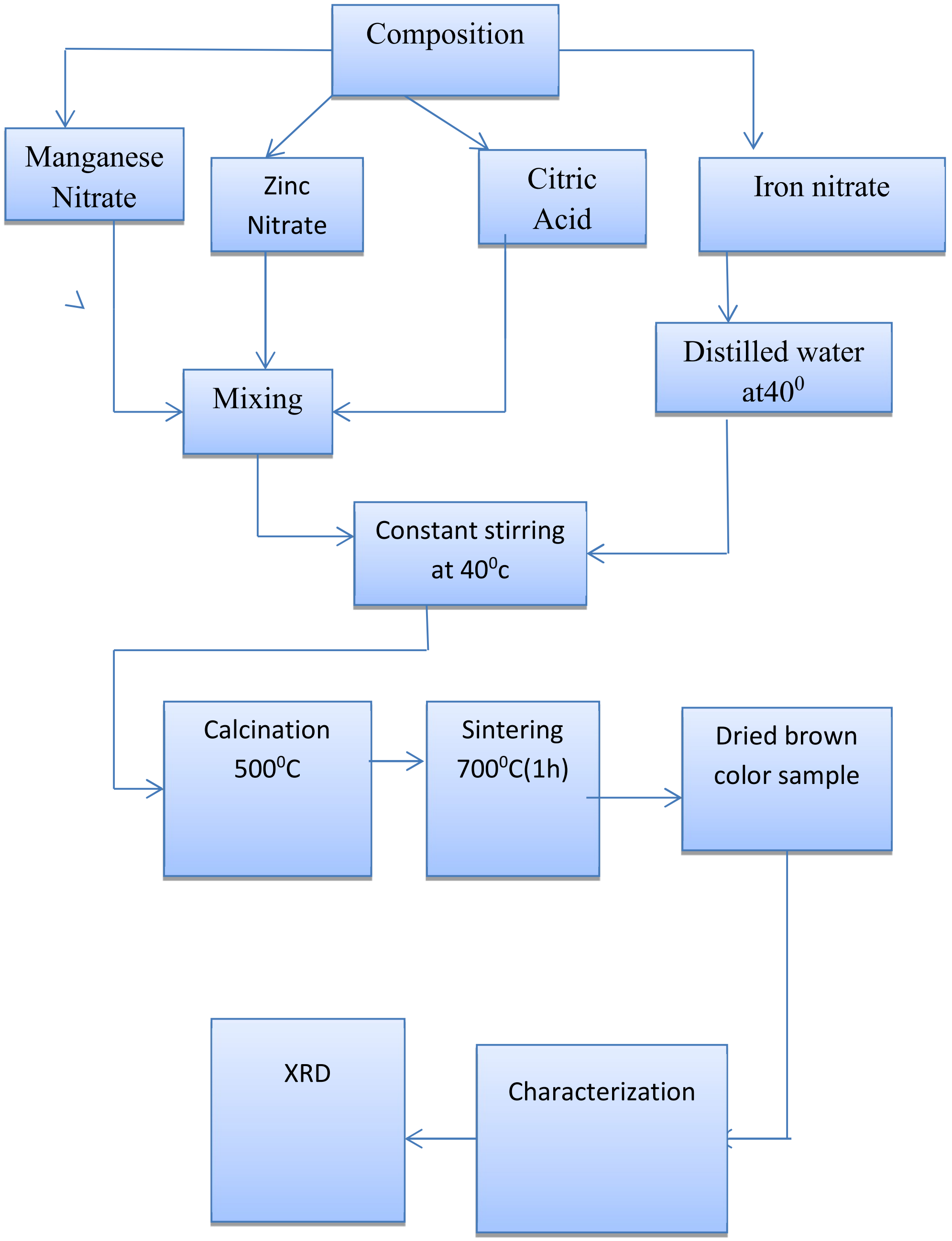
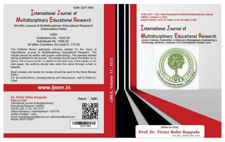


Figure: 3.12 Flow Chart of Synthesis of $Mn_{1-x}Zn_xFe_2O_4$



3.Characterization Techniques

3.1 X – Ray Diffraction

x- ray diffraction (XRD) is a intense analytical technique primarily used for phase identity of a crystalline materials and cell dimensions. X –ray diffraction is multilateral, non - destructive analytical technique for identification and quantitative of various crystalline form, Knowas 'phase' of compound appear in powder and solid samples. The analytical material is finely grounded homogenized and average bulk conformation is determind. A crystal is made up of lattice planes consisting of periodic of lattice point. The set of planes are designated by Miller Indices. When an X- ray strikes a family of planes, it well as shown in fig 3.2.

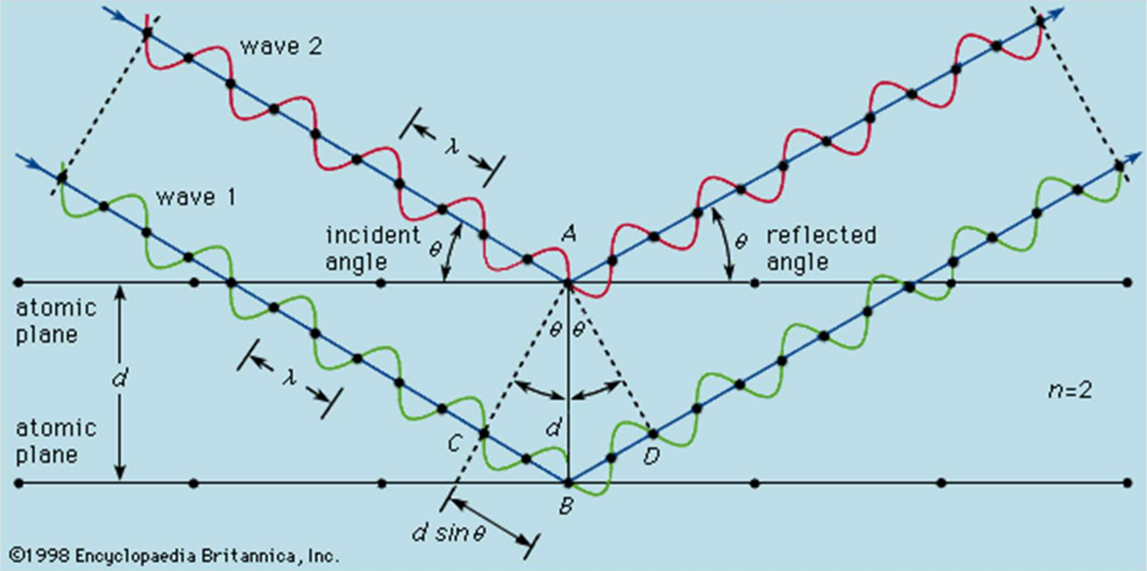


Figure 3.1 Ray diagram of XRD

The reflected rays from apart planes interfere with each other and the resultant intensity distribution is modify. For crystalline Material the diffracted wave consist of rapid interference maxima. The position of peaks in an X – ray diffraction pattern are directly belong to atomic distance. The wavelength λ of the x – ray, the spacing d amidst lattice planes and angle of reflection Ө are related as

$(2d\sin\Theta = n\lambda)$
λ = wavelength of X-ray
d = interplanar spacing
Θ =diffraction angle

(n) this is known as Ray diagram of XRD. Using the relation the d value of the material can be calculated [21].

3.2 Ultraviolet (UV) Spectroscopy

Ultra Violet perceptiable spectroscopy tends to absorption spectroscopy or reflectance spectroscopy in part of the ultraviolet and the full proximate visible spectral regions. This means it uses light in the perceptiable and adjacent ranges. The absorption or reflectance in in the perceptiable range direct effects the perceived color of the chemical involve. In this reason of the electromagnetic spectrum, atoms and molecules undergo electronic transitions. Absorption spectroscopy is supplementary to fluorescence spectroscopy, in that fluorescence deal with transition from the provocative state to the ground state, while absorption measures transitions from the ground state to provocative. Molecules containing bonding and non- bonding electrons can absorb energy in the form of ultra violet or perceptiable light to excite these electrons to higher anti bonding molecular orbitals. UV Spectroscopy is generally used in analytical chemistry for the quantitative determination of different analytes, such as transition metal ion, highly conjugated organic compounds, and biological macromolecules.

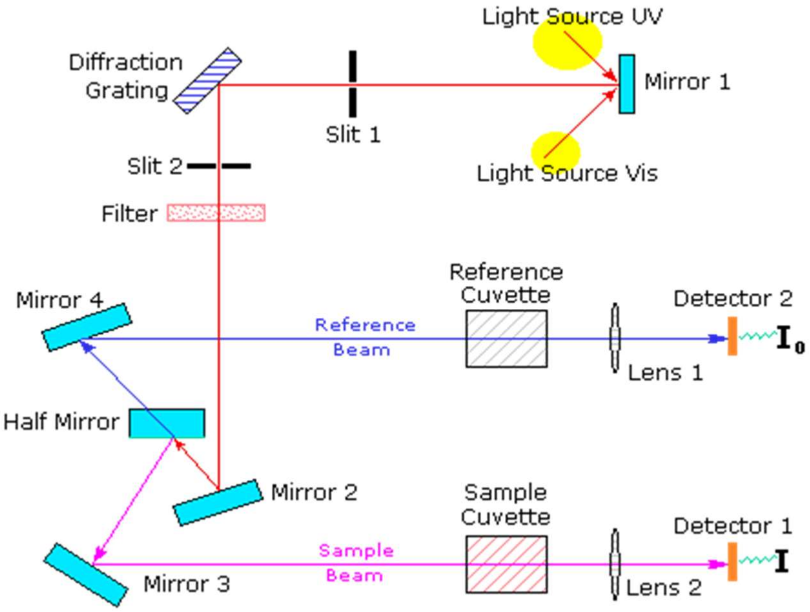


Figure3.2 Ray diagram of UV Spectroscopy

3.3 Scanning Electron Microscope (SEM)

A scanning electron microscope is a type of electron microscope that generates images of sample by scanning the surface with a focused beam of electrons. The Scanning Electron Microscope (SEM) is widely used method in a number of industries and laboratories to check out the microstructural morphology and chemical of conformation of specimens up to a nanometer scale. The electron interacts with the atom in the specimen, producing various signals that contain intimation about the surface topography and conformation of the sample. The electron beam is scanned in a raster scan pattern, and the position of beam is mixed with the intensity of detected signal to generate an image. In the most common SEM mode, secondary electrons emitted by atoms provocative by the electron beam are detected using an Everhart – Thornley detector. The number of secondary electrons that can be detected, and thus the signal intensity, depends, among other things, on sample topography. SEM can achieve resolution better then 1nm.

In a typical SEM, an electron beam is thermionic emitted from an electron gun fitted with tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapor pressure of all metals, there by allow it to be electrically heated for electron emission, and because of its low cost.

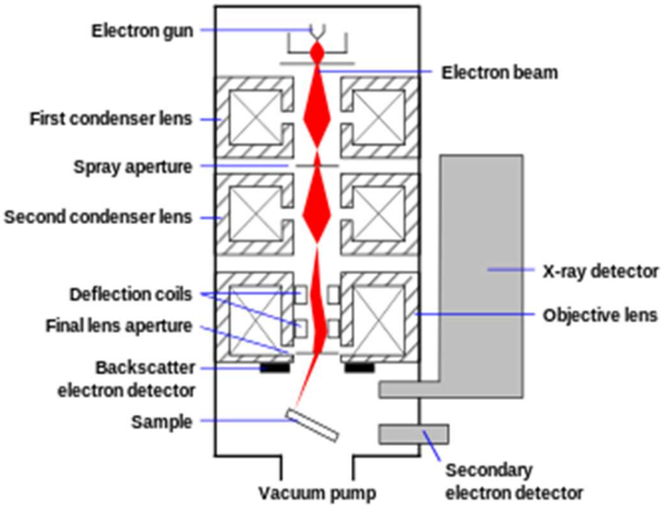


Figure3.3 Scanning Electron Microscope

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3.4 Transmission Electron Microscopy (TEM)

TEM is a microscopy method in which a beam of electrons is transmit through a specimen to form an image. Transmission Electron Microscopy (TEM) is one of the most important superfine tools in nanotechnology for characterizing material at a length scale from atoms to hundreds of nanometers. The specimen is most often an ultrathin section less than 100 nm plump or a suspension on grid. An image is formed from the interaction of electrons with the sample as the beam is transmit through specimen. The image is then magnified and focused on to an imaging device, such as fluorescent screen, a layer of photography film, or a sensor such a scintillator connected to a charge coupled device. Modern TEMs have the efficiency to directly image atom in crystalline specimen at resolve close to 0.1 nm.

Transmission electron microscopy is a major analytical method in the physical, chemical and biological science. TEMs find applications in cancer research, virology, and materials science as well as pollution, nanotechnology and semiconductor research, but also in other fields such as paleontology and palynology. TEM instruments boastanspacious array of operating modes including conventional imaging, scanning TEM imaging, diffraction, spectroscopy, and amalgamation of these.

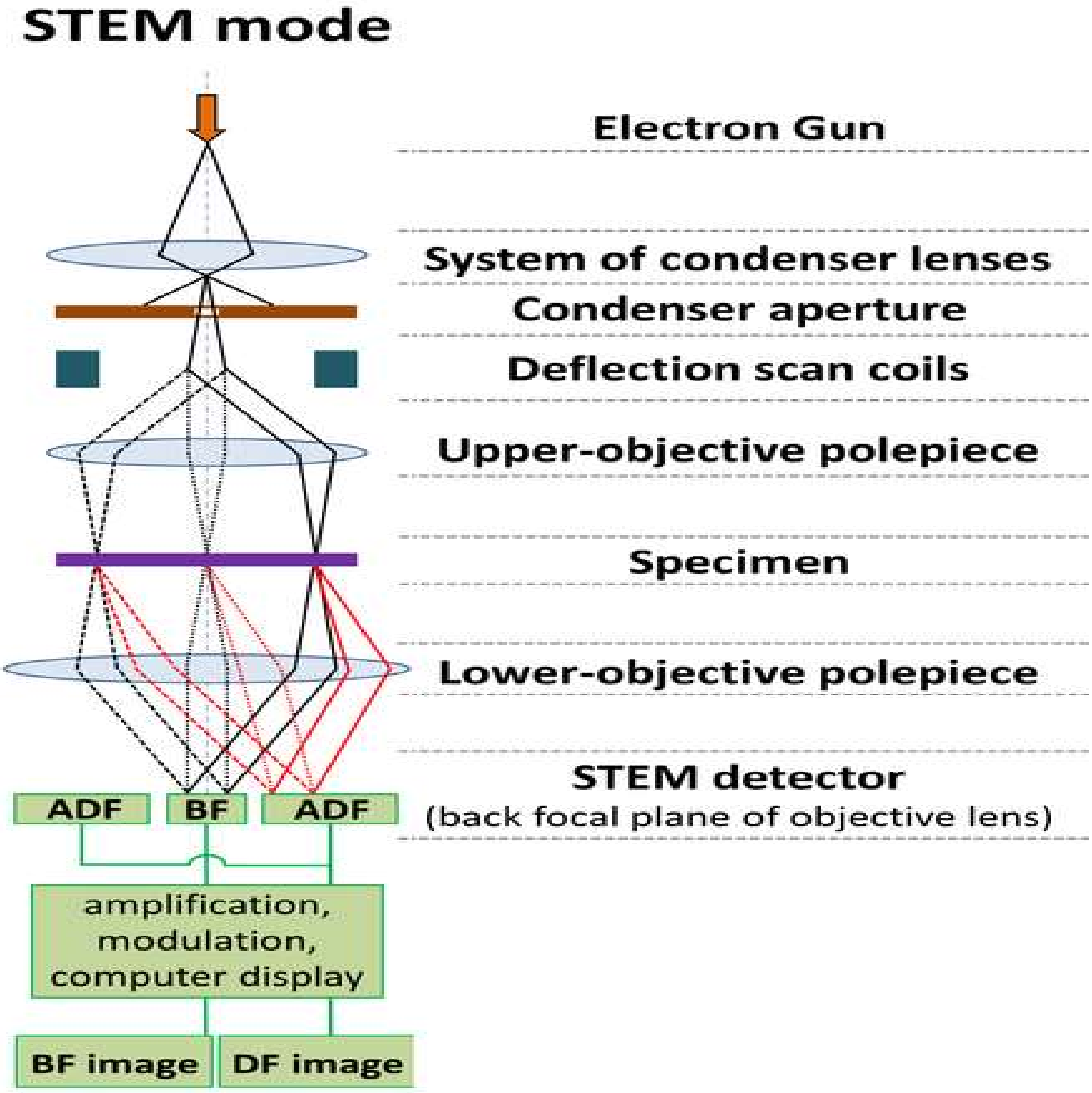
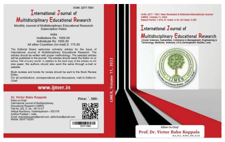


Figure3.4 Transmission Electron Microscopy



4.Result and Conclusions

4.1 XRD Analysis

X-Ray Diffraction is intense analytical technique primarily used for phase identity of a crystalline material and cell dimension. The analytical materials is finely grounded homogenized and average bulk conformation is determined. A crystal is made up of family of lattice planes consisting of periodic of lattice point. The set of planes are designed by Miller Indices. The position of peaks is an X-ray diffraction patterns are directly belong to atomic distance. The XRD pattern of MnZn ferrites has characteristic peaks showing the cubic spinel phase having Fd3m phase group. The shape of prepared ferrites is nearly spherical but some distortion may be observed after doping.

Grain size is another important contributing factor to the resistivity of the sample. Sample sintered at low temperature possess smaller grain size. Sample with grain consists of more number of grain boundaries. The boundaries are the region of mismatch between energy states of adjacent grains and hence act as barrier to the flow of electrons. Therefore, the sample sintered at low temperature is found to exhibits higher resistivity as compared to ceramic ferrites. Applying the Scherer's formula, the crystalline size of MnZn sample found to be approximately analyzer. Here it is seen that the Diffracto-grains are almost same but there are some undesired picks had been seen, due to the impurity phase present in the samples. The variations of Curie temperature with sintering temperature observed that the values are in close agreement with the reported values, which implies that Curie temperature of the ferrites increase with increase in sintering temperature and then decreases. This increase can explain that the grain size become more uniform. Hence, it is concluded that MnZn ferrite prepared by Citrate precursor technique results in better magnetic and electric properties then other technique. The MnZn ferrites are done with various instruments such that X-ray diffractometer, scanning electron microscopy and atomic force microscopy and atomic force microscope. The magnetic properties of the ferrites are studied by vibrating sample magnetometer magnetization hysteresis loops and electron spin resonance hysteresis loop measurements. The X-ray investigation is done using X-ray diffractometer. Various formulas for the determination of lattice constant, X-ray density and crystallite size.

Measurement of lattice constant (a): (0.6714)

From the analysis of XRD data, the lattice constant can be calculated using the formula:

$$a = d_{hkl}\,(h^2 + k^2 + l^2)^{1/2} \text{--------(1)}$$

Where a is the lattice constant, d is the interplanar spacing, and h, k, and l are the miller indices.

Measurement of X-ray density (d_{x-ray}): ($5.27\,{}^{gm}/_{cm^3}$)

Theoretical density can be calculated using the relation:

$$d_{xrd} = {}^{8M}/_{Na^3} \text{----------(2)}$$

Where M is the molecular mass of each component, N is the Avogadro's number ($6.023 \times 10^{23}\ {}^{\text{particles}}/_{\text{mol}}$).

Measurement of Experimental density (d_{exp}): ($5.21\,{}^{gm}/_{cm^3}$)

Experimental density can be measured using the formula:

$$d_{exp} = {}^{\text{mass}}/_{\text{volume}} \text{--------(3)}$$

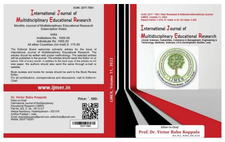
Measurement of crystalline size (D): (7.36585 nm)

Crystalline size is calculated by using the Scherrer's formula:

$$D = \frac{0.9\lambda}{\beta\cos\theta} \text{--------(4)}$$

Where,

D = crystalline size (nm)
K = 0.9(Scherrer constant)
λ = 0.15406 nm (wavelength of the X –ray sources)
β = FWHM (radian)
Θ = peak position



4.2 Structure parameter for nanoparticle ferrite

Sample	Structure size (nm)	Parameter (Å)	Lattice Parameter(gm/cm^3)	X-ray density	L_AÅ	L_BÅ
MnZn ferrite	7.36585	13.1615×10^{23}	0.6714	5.27	0.29072	0.2373399

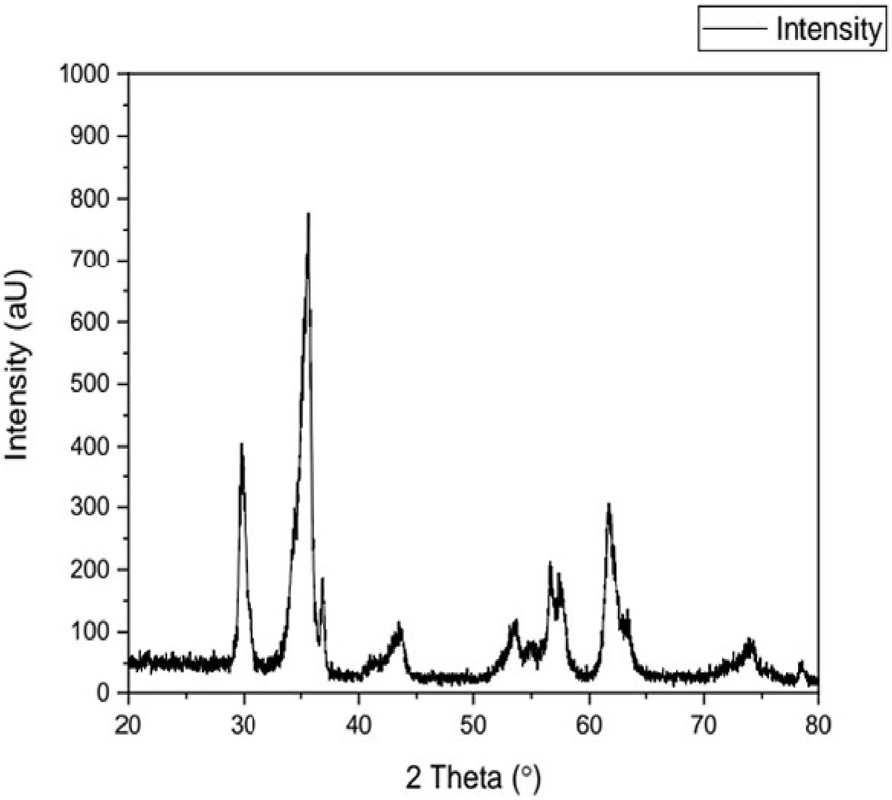


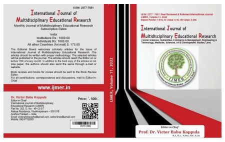
Figure 4.2: Intensity vs. 2Theta

The diffraction peaks are at (220), (311), (400), (422), (511), (440) which are observed and found to be matched well with JCPDS Card No. 17-0464

5. Conclusions

In the present work we have plane to study structural properties of MnZnnanoferrite. The main finding drawn from present work are summerised as below.

(1) The nanoferrites followers were MnZn Nanoparticles Synthesied via Citrate Precursor method.
(2) The XRD PatterenMnZnFe_2O_4 Particles are found to be matching well with JCPDS card No. 170464.
(3) The particle size was observed 7.3658 nm.
(4) The Hopping length of tetrahedral (A) site (L_A) is = 0.29072, The Hopping length of octahedral (B) site (L_B) is = 0.23733, X-ray density = 5.27gm/cm^3



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