**A DISSERTATION ON**

# "Synthesis and Characterization of $Ni_xMn_{1-x}Fe_2O_4$ Nanoparticles for Super Capacitive Applications"

**Submitted To The**
**Department Of Physics**
**Faculty Of Science**
**Integral University, Lucknow**

**IN PARTIAL FULFILMENT**
**FOR THE**
**DEGREE OF MASTER OF SCIENCE**
**IN PHYSICS**

**BY,**
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**M.Sc. Physics (IV Semester)**
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**UNDER THE SUPERVISION OF,**

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## CERTIFICATE BY SUPERVISOR

This is to certify that, **Nikhil Singh**; a student of M. Sc. (Physics-IV Semester) has successfully completed the dissertation on "**Synthesis and Characterization of $Ni_xMn_{1-x}Fe_2O_4$ Nanoparticles for Super Capacitive Applications"** under my supervision during the year 2021-2022. It is certified that the work has not been submitted anywhere else for the award of any other diploma or degree of this or any other University.

I wish him good luck and bright future.

**Date:18 May 2021**
**Place: Lucknow**

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## CERTIFICATE BY HOD

This is to certify that **Nikhil Singh** is a student of M. Sc. (Physics-IV Semester) Session 2020-21 to 2021-22 at Integral University, Lucknow. He completed his project entitled "**Synthesis and Characterization of $Ni_xMn_{1-x}Fe_2O_4$ Nanoparticles for Super Capacitive Applications**" successfully under the supervision of Ms. Tahira Khatoon, Assistant Professor (Level II), Department of Physics, Integral University.

I wish him good luck and bright future.

**Date:18 May 2022**
**Place: Lucknow**

**(Dr. Seema Srivastava)**
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**NIKHIL SINGH**

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# Abstract

Sol–gel auto-combustion was used to make nanoparticles Ni $_x$ Mn $_{1-x}$ Fe$_2$ O$_4$. Due to the presence of a secondary phase, X-ray diffraction investigation demonstrates that the produced nanomaterials are spinel ferrite.

The regulation of the crystalline size many syntheses variable have been adjusted to precisely control the size molarity of reactants and the continuous stirring temperature and the amount of the citric acid. The Empirical formulae is ($ABFe_2O_4$) Ni $_x$ Mn $_{1-x}$ Fe$_2$ O$_4$ (x=0.2, 0.4, 0.6, 0.8 and 1.0).

The prepared ferrite is further tested for the analysis of Application in Capacitors Spherical morphology is revealed by the Sem analysis and images and size were found to approximately 50 nm. The electrochemical properties and CV Voltammetry analysis shows that mixed metal ferrites ($ABFe_2O_4$) have better capacitive performance as compared to metal ferrites ($AFe_2O_4$).

# Chapter 1

# 1 Introduction

## 1.1 Nanomaterials and Nanotechnology

The Unique advantage of property change in material with respect to size has made the nanomaterials one of the most sought after in many field-like electronics as well as biological, nanotechnology has developed with one of the most significant areas for researchers in Bio-chemistry, Biotechnology, chemistry, physics, and electronics, among many others, due to its huge prospects towards multiple fields such as healthcare, environment, agriculture, and energy. There are three factors:

- A greater understanding of matter's characteristics at the atomic level.
- Advancement based molecular approaches that living beings may be using,
- The improvement in the aspects of information processing.

Nanostructure materials have received a lot of attention since their physical, chemical and electrical, properties differ significantly from those of their higher-dimensional counterparts and are influenced by their shape and size.

**“There's Plenty of Room at the Bottom”**

-Richard Feynman

On December 29, 1959 in California Institute of Technology (Cal-Tech), long before the term nanotechnology was dreamed up. Feynman proposed a technique by which scientists may manipulate and control individual atoms and molecules in his talk.

## 1.2 Ferrites

The Ferrites are oxide of iron with magnetic as well as electric application and properties. The general formula that the ferrite follows is AFe2O4. A can be single or combination of multiple atoms.

The eddy current loss in ferrite is low so ferrite can act as better and more superior to other magnetic material. The have show some high DC current resistivity. It's been a long time that ferrites are being a major part of scientific investigation. And discovery of new better ferrites has resulted into drastic change in the scientific community.

Considering that ferrites have impacted on many levels and have been used in several field for majority oof time they are now classified Ferrites can be classified into three different types.

Types of Ferrites: -

1. Hexagonal Ferrites
2. Garnets
3. Spinel ferrites

### 1.2.1 Hexagonal ferrite

$M(Fe_{12}O_{19})$, where M is commonly barium (Ba), strontium (Sr), or lead, is the formula for so-called hexagonal ferrites (Pb). Although the crystal structure is complex, it may be defined as hexagonal with a single c axis (vertical axis). In the fundamental structure, this is the simple magnetization axis. Hexagonal ferrites are referred described as "hard ferrites" because the direction of magnetization is difficult to change to another axis.

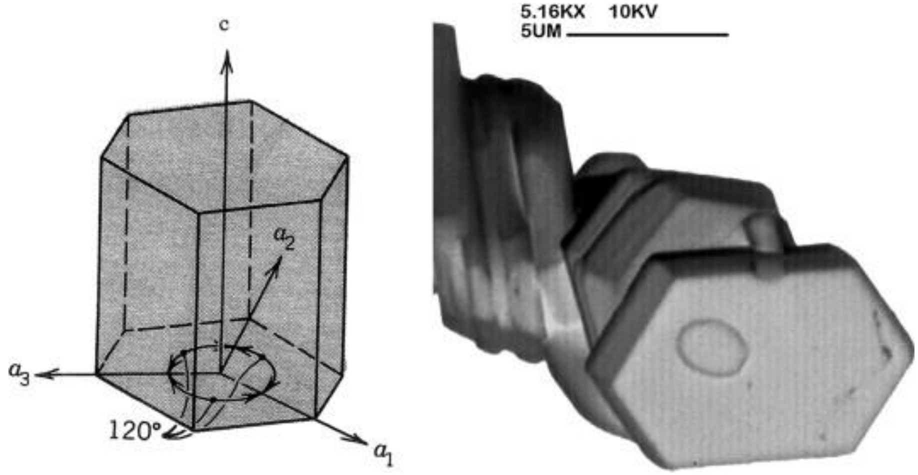



*Figure 1.1 Hexagonal ferrite*

### 1.2.2 Garnets

Garnet ferrites have the chemical formula $M_3(Fe_5O_{12})$, where M is yttrium or a rare-earth ion, and have the structure of the silicate stone garnet. Garnets feature dodecahedral (12-coordinated) sites in addition to tetrahedral and octahedral sites like those seen in spinels. As a result of the antiparallel spin alignment among the three types of sites, net ferrimagnetism is a complex result. Garnets are magnetically hard as well.

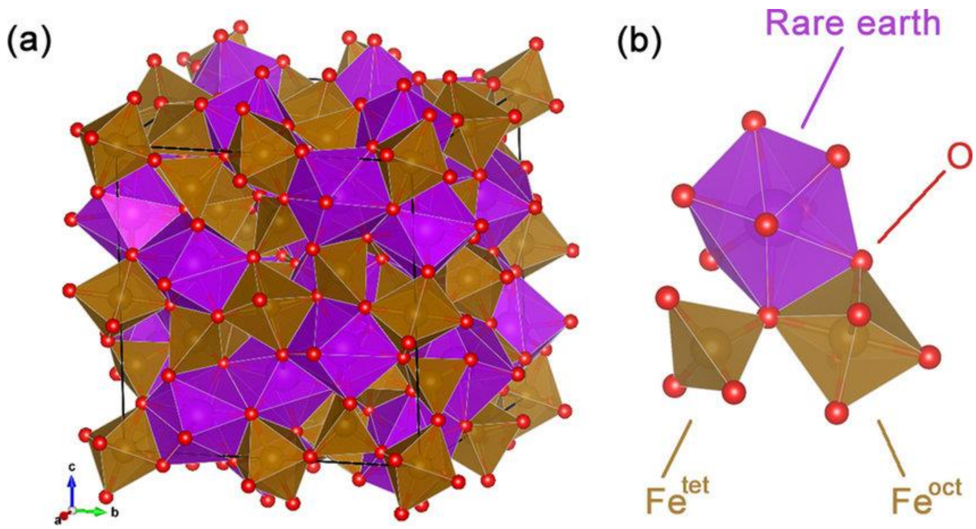
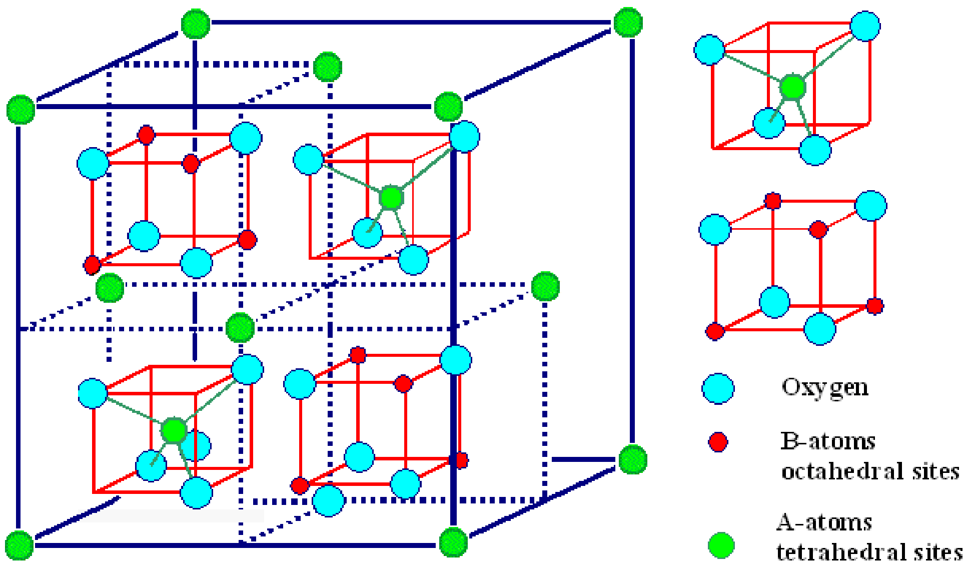



*Figure 1.2 Garnets*

### 1.2.3 Spinel Ferrite

WL Bragg developed spinel ferrite in 1915, and its name was derived from the mineral spinel, which has an ionic structure. Ferrites piqued the interest of nanotechnology researchers due to their applicability as semiconductors across a wide variety of frequencies ranging from microwaves to radio waves. Spinel ferrites are a variety of crystalline solid that is renowned because of its "tunable" magnetic characteristics. Spinel is by far the most common ferrite, so much so that the name is nearly synonymous with ferrite.




**$AB_2O_4$ spinel** The red cubes are also contained in the back half of the unit cell

*Figure 1.3 Spinel Ferrite*

## 1.3 Manganese Ferrite

Manganese ferrite is a cubic spinel structure ferrite that has been widely employed in a variety of technical applications. Manganese ferrite characteristics are extremely dependent on composition, shape, and size, all of which are directly related to the preparation circumstances. Manganese ferrite has been synthesized in a variety of shapes and sizes up to this point. A solvothermal technique, for example, has nanoparticles of $MFe_2O_4$ (M = Mn, Co, and Ni) with 5nm to 10 nm in size of diameter.

A large number of high-purity $Mn_{1-x}Zn_xFe_2O_4$ nano crystallites were synthesized and these nano crystallites-oriented aggregation to nanospheres. Solvothermal technique was used to controllably create colloidal nanostructure and hollow spheres of the material that is $MnFe_2O_4$ And that too with identical submicron sizes by merely altering the synthesis microenvironment.

*Figure 1.4 Manganese Ferrite*

## 1.4 Nickel Ferrite

Nickel ferrite $NiO.Fe_2O_3$ or $NiFe_2O_4$ behaves as semiconductors with charge carrier of low mobility. It is 80% inverse and has a magnetic moment of 2.3μiB compared with a theoretical value of 4μB. Ferrite is incorporated into a spinel structural unit cell. The ferric ions preferentially fill the tetrahedral positions, although only half of them can fit. The remaining eight, together with the eight Ni2+ ions, are placed on the octahedral sites.

*Figure 1.5 Nickel Ferrite*

## 1.5 Crystal Structure of Ferrites

The end use is typically linked to the crystal structure. For instance, BaO mixes with $Fe_2O_3$ to generate a hexagonal structure with a distinct crystal axis that lends itself to permanent magnet applications. The cubic crystal structure, on the other hand, has several equal crystal orientations and will be beneficial when it is preferable to avoid a favored direction.

## 1.6 Applications of ferrites

### 1.6.1 Inductors

Many of us have seen ferrites being used in inductor their hysteresis curve is much more better suited for inductive work the perform exceptionally better than their counterpart the also have application in low noise amplifier or voltage controlled oscillator. Their latest uses as inductors follow the overall trend of shrinking and integration as ferrite multilayers for passive functioning electronic devices, among other things. Multilayer technology has emerged as a critical technology for mass manufacture of integrated devices this allows the density of the components to be much more higher. Multilayer capacitors first appeared on the market a few decades ago, while inductors made their debut in the 1980s. A extremely soft ferrite and a metallic coil serve as the essential components for producing inductance.

Furthermore, in order to offer a high permeability at the operating frequency, the ferrite film should be manufactured using a procedure that is compatible with the integrated circuit production process. Sputtering produces dense films, but the composition can be difficult to manage precisely, and the annealing procedures can reach high temperatures. Although pulsed laser deposition produces high-quality films, a process involving the creation of the ferrite film by a mix of sol-gel and spin-coating appears to be easier and less expensive.

*Figure 1.6 Inductors*

### 1.6.2 High Frequency

Ferrites have the attitude of good magnetic property and they can be utilized in high frequency application like in the generation of the microwave or the radar systems that needs higher frequencies and bandwidths of up to 100 GHz. ferrites are insulating in nature that makes them a good dielectric, allowing entire penetration of electromagnetic fields, as opposed to metals, where the skin effect significantly inhibits high-frequency field penetration. The magneto-strictive characteristic of ferrites is used to generate ultrasonic waves from a ferrite rod using an alternating magnetic field.

### 1.6.3 Power

In the electronic industry we can see that the ferrites have been widely used in power application for a wide range of devices such as computers, various peripherals, TV and video systems, and various small and medium instruments. The primary application is in switched-mode power supply systems (SMPSs).

The main Ac signal is rectified via ferrite core transformer initially in this application, then switched as regular pulses (usually rectangular) at a high frequency to feed into a ferrite transformer, and lastly rectified again to supply the appropriate power to the instrument. By raising the working frequency of the transformer, you may improve power delivery and efficiency.

A recent strategy to improving ferrite core efficiency is to reduce eddy current which is done by increasing resistivity of the material. Apart from the use of nonconducting additives that preferentially dwell on grain boundaries (reducing intragrain conductivity)

### 1.6.4 Biosciences

Magnetic nanoparticles, primarily magnetite ($Fe_3O_4$), are found in a variety of living species and may be exploited in a variety of applications. Magnetic nanoparticles can, of course, be prepared in the laboratory using well-known methods; however, magnetic biogenic particles have superior properties to synthetic ones: they have a defined size range and width/length ratio, high chemical purity, are almost perfect crystallographically, and sometimes have unusual crystallographic morphologies. Various species of bacteria have been shown to produce nanoscale magnetite particles extracellularly. In many situations, the biogenic particles maintain a lipid covering, making them extremely durable and biocompatible.

Hypothermia is severely deadly but here ferrite can be used for heating particular part of the organ and this can be done via radiotherapy. Radio therapy is also utilized in the treatment in cancers In addition to magnetic properties the spinel ferrites as well as hexaferrites have shown promising scope in the medical industries.

### 1.6.5 Electronics and IT

Ferrites are exploited in temperature sensing switches used in refrigerators, air conditioners, electronic ovens, and other electronic devices. Ferrite's insulating characteristics are applied in electric motors; they are also used as flat rings for loud speakers, wind screen wiper motors, and TV correction magnets. As certain ferrites have a high rectangular hysteresis loop, they can be used to build computer memory devices for rapid store and retrieval of digital information. The magneto-strictive characteristic of ferrites is used to generate ultrasonic waves from a ferrite rod using an alternating magnetic field.

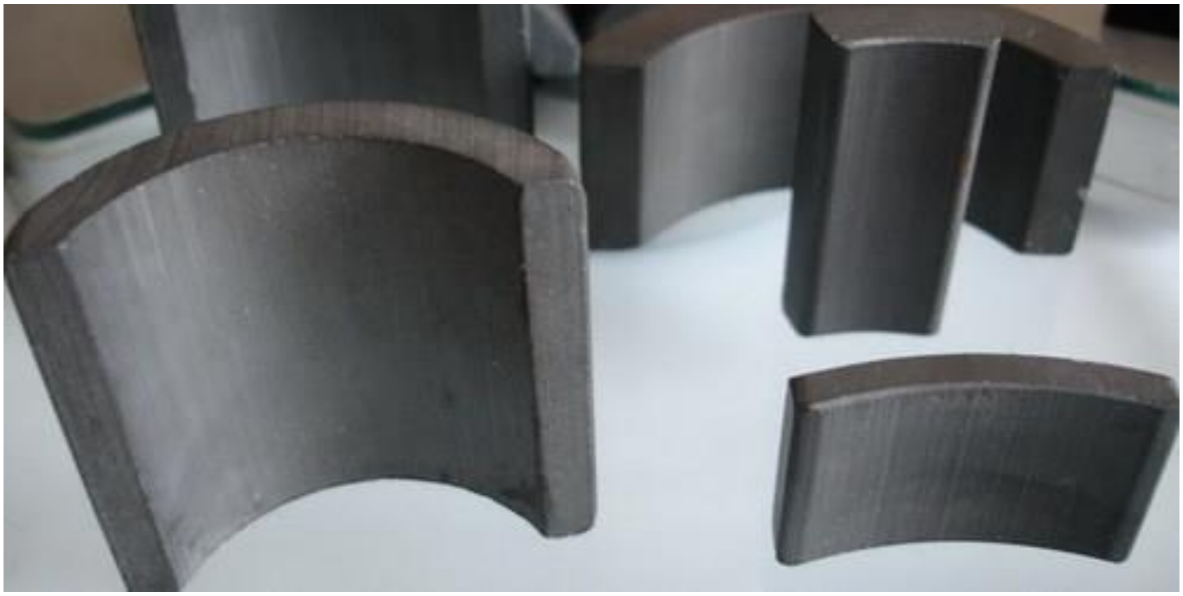


*Figure 1.7 Electronics and IT*

## 1.7 Capacitance

The capacity of any material to hold Electric charge in it in the form of electric potential is called Capacitance. Those material which has this property are called Capacitors. The capacitors are of various types based on their application. Usually, their Construction include two very thin film of material separated by a very small distance of d in which a dielectric material is filled. The dielectric is insulator which on the application of External Electric Field produces internal Electric field due to polarization. This field is generated due to internal small dipole crated by External field and its direction is opposite to the direction of the external electric field.

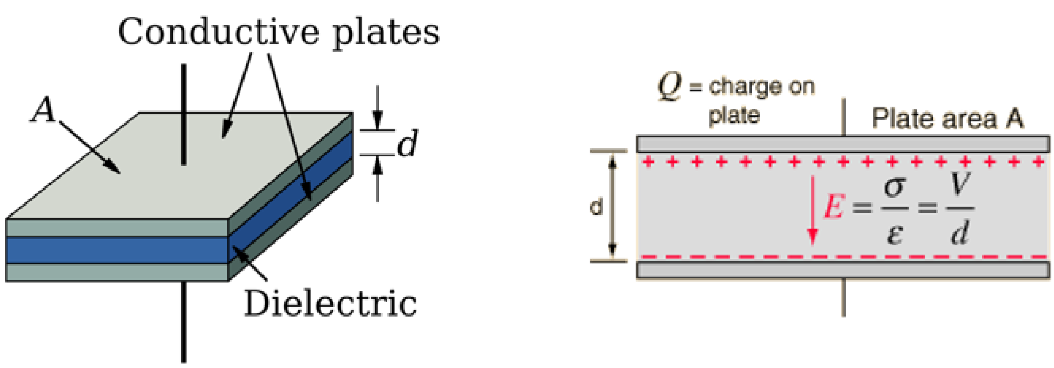



*Figure 1.8 Capacitance*

**Capacitance**

$$C = \frac{Q}{\Delta V} = \frac{Coulombs}{Volts} = Farads$$

**Electric Field between flat plates**

**Potential Difference between flat plates**

$$E = \frac{\sigma}{\varepsilon_o} = \frac{Q}{\varepsilon_o A}, \quad \Delta V = Ed = \frac{Qd}{\varepsilon_o A}$$

$$C = \frac{Q}{\Delta V} = \frac{Q}{Qd/\varepsilon_o A} = \frac{\varepsilon_o A}{d}$$

**Capacitance depends on the Area of the plates and the distance between the plates**

*Figure 1.9 Capacitance Equations*

The capacitance of the material is dependent on the Area of its plate as the more are of the plate the more charge will be accumulated on the surface of the plate. The SI unit of capacitor is farad. which is a very large Quantity, generally the Commercial market have the capacitor of value of 10 to 0.5 Pico farad. The supper capacitor has been crated nowadays up to 1000 micro farads.

Although the capacitor has similar functionality like and ordinary battery and store less amount of charge in it but the capacitor have some Additional benefits.

1. Capacitor can be charge in few milliseconds.
2. Capacitor can have higher potential tolerance.
3. The Efficiency of the Capacitor are much higher.
4. The Capacitor can deliver its peak power instantaneously.

# Chapter 2

# 2 Methodology

## 2.1 Material Used

1. Iron (III) nitrate nonahydrate
2. Nickel (II) nitrate hexahydrate
3. Manganese (II) nitrate tetrahydrate
4. Citric Acid
5. Ethylene Glycol
6. Ammonia (liquor)

### 2.1.1 Iron (III) nitrate nonahydrate

Iron (III) nitrate nonahydrate can be used as an inorganic precursor in the modified sol-gel process to create iron (III)-doped titania nanoparticles. Nonahydrate ferric nitrate supported on silica can be used to make benzyls. It has been found that iron (III) nitrate nonahydrate thermally decomposes. At 523K, the primary breakdown product was $Fe_2O_3$.

*Figure 2.1 Iron (III) nitrate nonahydrate*

### 2.1.2 Nickel (II) nitrate hexahydrate

Ammonium nickel molybdate is the precursor of "green powder," which is employed as a selective oxidation catalyst for low molecular weight alkanes.

*Figure 2.2 Nickel (II) nitrate hexahydrate*

### 2.1.3 Manganese (II) nitrate tetrahydrate

Manganese (II) nitrate is an inorganic chemical having the formula Mn $(NO_3)_2(H_2O)$ n. These substances are nitrate salts with various quantities of water. The tetrahydrate, Mn $(NO_3)_{24}H_2O$, is the most frequent derivative, but mono- and hexahydrates, as well as the anhydrous substance, are also known. Some of these chemicals are valuable precursors to manganese oxides. It is paramagnetic and generally light pink as a manganese (II) combination.

*Figure 2.3 Manganese (II) nitrate tetrahydrate*

### 2.1.4 Citric Acid ($C_6H_8O_7$ or $CH_2COOH\text{-}C(OH)COOH\text{-}CH_2COOH$)

Citric acid is a mild acid present in all citrus fruits. These foods contain the most naturally occurring citric acid. Lemons, limes, oranges, grapefruits, and berries.

Citric acid's major roles in the sol-gel process detailed in multiple articles are as a

- (i) complexing agent for metal ions.
- (ii) medium viscosity regulating agent
- (iii) organic fuel during the calcination process.

*Figure 2.4 Citric Acid*

### 2.1.5 Ethylene Glycol

Ethylene glycol also known as is a vicinal diol with chemical formula $(CH_2OH)_2$, has major uses in the production of polyester fibers and in antifreeze compositions. it poses viscous, sweetening but strongly poisonous liquid.

### 2.1.6 Ammonia (Liquor)

Ammonia in Water, often known as Aqua Ammonia, makes it safe to handle and helps to avoid the problems associated with Ammonia Gas usage by considerably decreasing its vapor pressure. It is an inorganic chemical that is also known as Ammoniacal Liquor and is utilized as a liquid chemical in numerous sectors such as rubber and pharmaceuticals. The chemical formula for Ammonium Hydroxide is $NH_4OH$.

## 2.2 Equipment Used

1. Beaker (500ml)
2. Weigh Scale
3. Magnetic stirrer
4. Pipet (1ml)
5. Ph scale
6. Heating mantle
7. Crucible cups
8. Muffle Furnace


*Figure 2.5 Weight Scale*


*Figure 2.6 Beaker*

*Figure 2.7 Magnetic Stirrer*

*Figure 2.8 Pippet*

*Figure 2.9 pH Scale*


*Figure 2.10 Heating Mantle*

The temperature and the mass of the precursors were properly calibrated for better yield.

## 2.3 Method Of Synthesis

Sol–gel auto combustion is a rapid and cost-effective particulate synthesis technology that has been widely employed for the synthesis of a variety of metal and alloy nanoparticles, generating nano-sized, homogenous, and highly reactive powders by mixing different elements at the atomic level. This is also a **bottom-Up Method** (in which we start from small and cluster them together to make desired shape and sized nano particle).

In this procedure, the molecular precursor (typically a metal alkoxide) is dissolved in water or alcohol and then heated and stirred to form a gel by hydrolysis/alcoholysis. Because the gel produced by the hydrolysis/alcoholysis process is wet or damp.

The process began with measuring 50ml of double distilled water at normal temperature and pressure and pouring it into the beaker. The cleaned beaker of 500ml was then placed on the magnetic stirrer with rotating bead at 400rpm rotation speed and no heat, and the salt was measured by weight and added to the solution in the order specified by the chemical formulae. After adding all of the nitrates to the solution, the beaker is left to mix properly. After the solution has been mixed, it is treated to citric acid, which is measured by weight as a chelating agent for the metallic ions. The addition of citric acid significantly decreased the solution's pH to very acidic. The liquid ammonia correctly balances the solution's high acidic ph. in a controlled atmosphere with a high precision digital ph. scale. The ph. is adjusted until it reaches a value of 7, which is regarded totally neutral. After the solution has reached 80 degrees Celsius equally throughout, ethylene glycol is added to the solution for its gelling agent property, and the solution is held at 80 degrees Celsius for appropriate evaporation of water molecules and conversion of solution to gel. The solution is then combusted using the heating mantle once it has been turned to gel.

The combusted material, which has been ground to power, is put in crucible cups for heating in the muffle furnace. The muffle furnace's extremely high temperature evaporates and eliminates any impurities left behind by carbon-based compounds such as citric acid and ethylene glycol. The activated nanoparticle is then placed in a clean container for further examination and research. The entire procedure is performed for each distinct combination of Ni and Mn doping. For comparison purposes, Ni is combined in increasing amounts while Mn is lowered. The molar ideas for the quantity of Ni are (x=0.2, 0.4, 0.6, 0.8 and 1.0). Mn is reduced in the same proportion as x (x=0.5,0.4,0.3,0.2.0.1, and 0.0). This change is made only for the purpose of Comparative study of the different combination of the ions with their nano particle property.

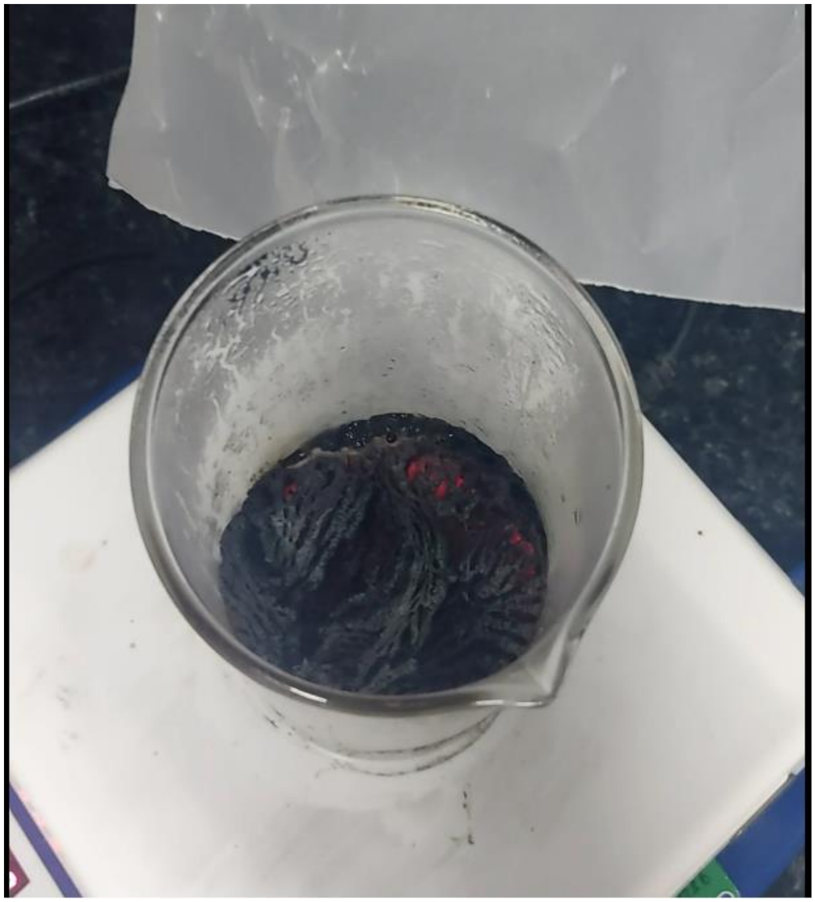


*Figure 2.11 Synthesis Sample A*

*Figure 2.12 Synthesis Sample B*

## 2.4 Advantages of the Sol-Gel method

i. It’s a simple process for synthesis
ii. It produces material with high purity.
iii. Very highly efficient process
iv. High chemical reactivity as the precursors is in solution phase
v. Precise control of the formation of the nanomaterial
vi. Good quality return on nanomaterial with low investment.
vii. In Solution phase surface coverage is maximum.
viii. Different phase of material can be mixed to obtain homogenous material.

## 2.5 Application of the Nano particle synthesized by Sol-gel Method

i. Low energy is required Synthesis
ii. Optical components can be produced with various shapes
iii. Oxides in form of composites can be produced
iv. Porous material can be produced for the enrichment of the organic compound.
v. Amorphous phase can be synthesized
vi. The efficiency is very high in comparison to other methods

## 2.6 Characterization Technique Used

### 2.6.1 XRD

the X-Ray diffraction technique also known as XRD is one of the nondestructive and most effective with material that are crystalline in nature. This technique is utilized for the purpose of analyzing the crystal structure and molecular structure of the lattice or crystal.

Because the substance being analyzed is often finely powdered down to a uniform condition, the process is commonly referred to as x-ray powder diffraction. When light bends slightly as it goes around the edge of an object or contacts an impediment or opening, this is referred to as diffraction

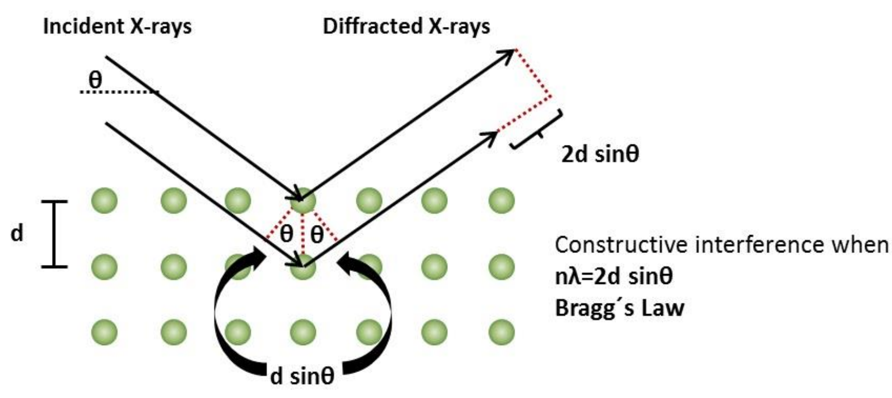


*Figure 2.13 Brags Law*

What will XRD reveal?

X-ray diffraction can perform the following functions: Calculate the average spacings between atom layers and rows in a material. Determine the direction of each grain or crystal. Small crystalline regions' size, shape, and internal stress should be measured. Determine the crystal structure of a previously unknown material.


*Figure 2.14 PANalytical EMPYEREAN XRD*

### 2.6.2 FE-SEM Analysis

FE-SEM is abbreviation for Field Scanning electron microscopy and it its used for the analysis of the material at the elemental information of 10x to 300000x with virtually unlimited depth of the field. In Comparison to Conventional SEM the Fe-SEM produces clearer, less electrostatically distorted images with 3 to 6 times better resolution.

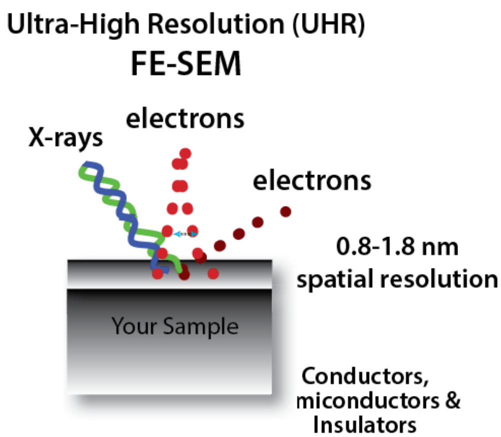



*Figure 2.15 FE-SEM Principle*

*Figure 2.16 FE-SEM Technique*

## 2.7 Electrochemical properties and CV Voltammetry analysis

CV Voltammetry is a technique first briefly described by Randle's. It is widely Used for the Electrochemical Analysis of reactants. It is found to be one of the most Reliable approaches for the investigation of parameters based on electrochemical properties.

# Chapter 3

# 3 Results and Discussion

## 3.1 X-ray Diffraction Analysis

The crystalline phase, as well as the crystalline structure, has been investigated using X-ray diffraction analysis. For a consecutive pattern of diffraction, there should be a regular structure of materials. The synthesized $Ni_xMn_{1-x}Fe_2O_4$ (where x = 0.2, 0.4, 0.6, 0.8 & 1.0) were characterized by X-ray diffraction and peak positions are recorded at room temperature. Crystal structure of synthesized material is found to be spinel cubic as reported in literature. Diffraction planes and full-width half maxima for the same materials are shown below table. It has been found that after increasing the dopant percentage of Ni, the peak position shifted towards a higher angle.

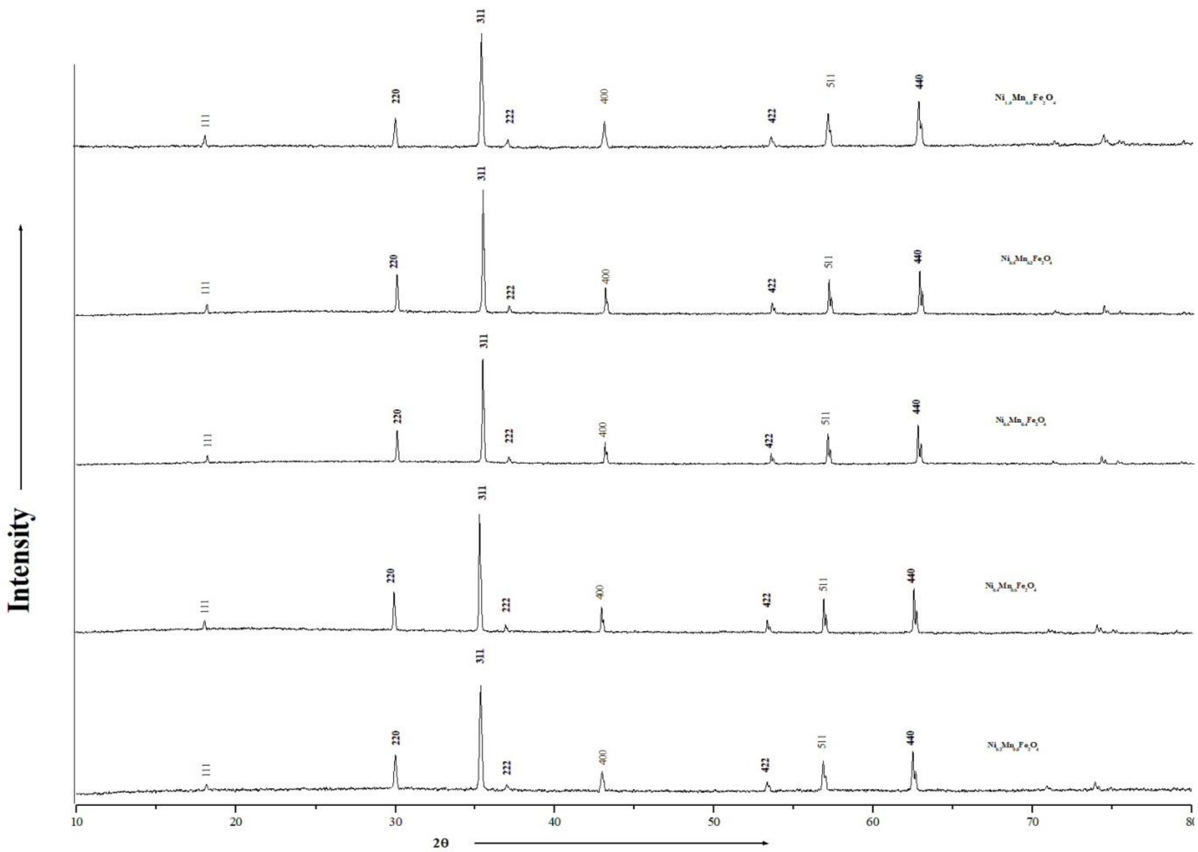



*Figure 3.1 XRD analysis of $Ni_xMn_{1-x}Fe_2O_4$*

The crystalline size of $Ni_xMn_{1-x}Fe_2O_4$ has been calculated using Debye-Scherrer formula.

$$\tau = \frac{K\lambda}{\beta cos\theta} \tag{3.1}$$

Where τ denotes mean size of the crystalline, and $K$ having constant value 0.91, $\lambda$ denotes X-ray wavelength having value of 1.5406Å (Cu K-alpha), full width half maxima has denoted by $\beta$ and it obtained from X-ray diffraction, and $\theta$ known as Bragg angle that can be also obtained from X-ray diffraction.

*Table 3.1 Full Width Half Maxima values of $Ni_xMn_{1-x}Fe_2O_4$*

| Sr. No. | hkl | Samples | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $Ni_{0.2}Mn_{0.8}Fe_2O_4$ | | $Ni_{0.4}Mn_{0.6}Fe_2O_4$ | | $Ni_{0.6}Mn_{0.4}Fe_2O_4$ | | $Ni_{0.8}Mn_{0.2}Fe_2O_4$ | | $Ni_{1.0}Mn_0Fe_2O_4$ | |
| | | 2θ | FWHM | 2θ | FWHM | 2θ | FWHM | 2θ | FWHM | 2θ | FWHM |
| 1. | 111 | 18.16 | 0.128 | 18.16 | 0.102 | 18.20 | 0.063 | 18.20 | 0.127 | 18.20 | 0.204 |
| 2. | 220 | 30.00 | 0.128 | 30.00 | 0.051 | 30.05 | 0.051 | 30.07 | 0.153 | 30.10 | 0.089 |
| 3. | 311 | 35.34 | 0.153 | 35.35 | 0.051 | 35.42 | 0.051 | 35.45 | 0.051 | 35.49 | 0.063 |
| 4. | 222 | 36.99 | 0.204 | 36.99 | 0.051 | 37.06 | 0.153 | 37.10 | 0.153 | 37.14 | 0.127 |
| 5. | 400 | 42.98 | 0.102 | 43.00 | 0.051 | 43.09 | 0.062 | 43.13 | 0.062 | 43.18 | 0.102 |
| 6. | 422 | 53.34 | 0.102 | 53.39 | 0.092 | 53.50 | 0.076 | 53.61 | 0.078 | 53.62 | 0.153 |
| 7. | 511 | 56.87 | 0.076 | 56.92 | 0.092 | 57.05 | 0.062 | 57.12 | 0.092 | 57.19 | 0.153 |
| 8. | 440 | 62.46 | 0.093 | 62.53 | 0.078 | 62.55 | 0.078 | 62.74 | 0.062 | 62.83 | 0.153 |

*Table 3.2 Mean crystalline size of $Ni_xMn_{1-x}Fe_2O_4$*

| Sr. No. | Sample Name | Mean Crystalline Size |
|---|---|---|
| 1 | $Ni_{0.2}Mn_{0.8}Fe_2O_4$ | 2.89nm |
| 2 | $Ni_{0.4}Mn_{0.6}Fe_2O_4$ | 2.88nm |
| 3 | $Ni_{0.6}Mn_{0.4}Fe_2O_4$ | 2.88nm |
| 4 | $Ni_{0.8}Mn_{0.2}Fe_2O_4$ | 2.88nm |
| 5 | $Ni_{1.0}Mn_{0.0}Fe_2O_4$ | 2.34nm |

## 3.2 FE-SEM Analysis

Morphological studies have been done through FE-SEM, using a FEI, QUANTA FEG 450 scanning electron microscope. at a voltage of 10kV in a secondary electron mode. Only one sample was characterized for morphological details. The obtained images reveal a particle size of 50nm on average and spherical shape.

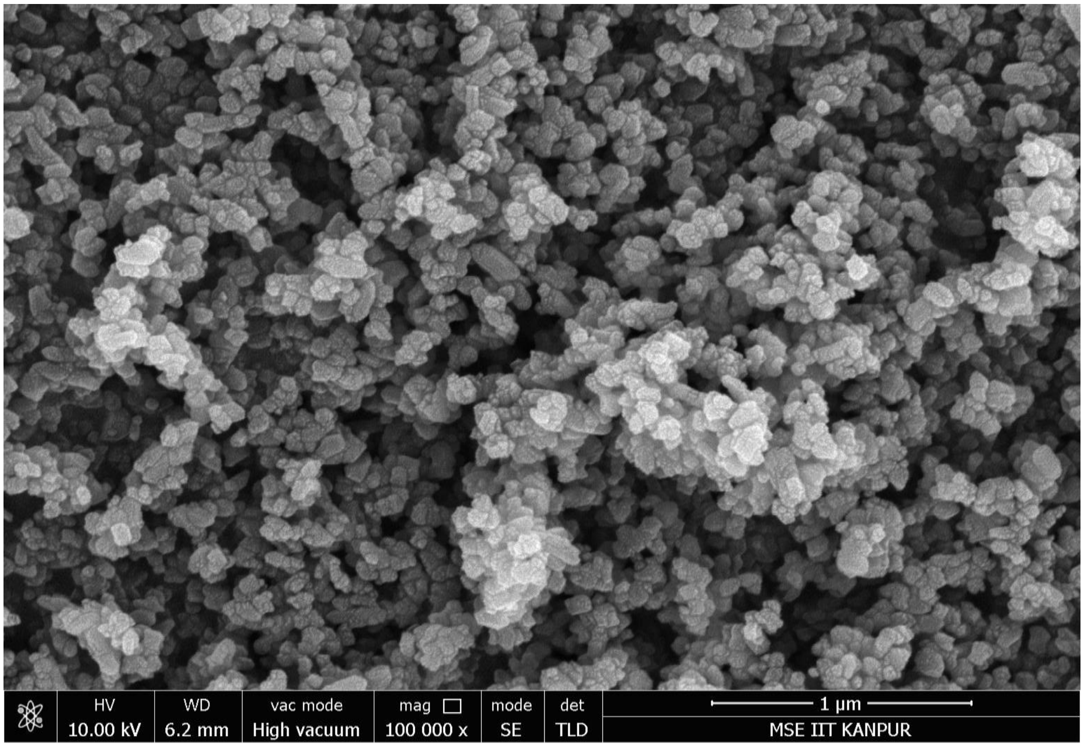



*Figure 3.2 SEM analysis of Ni0.4Mn0.6Fe2O4 A*

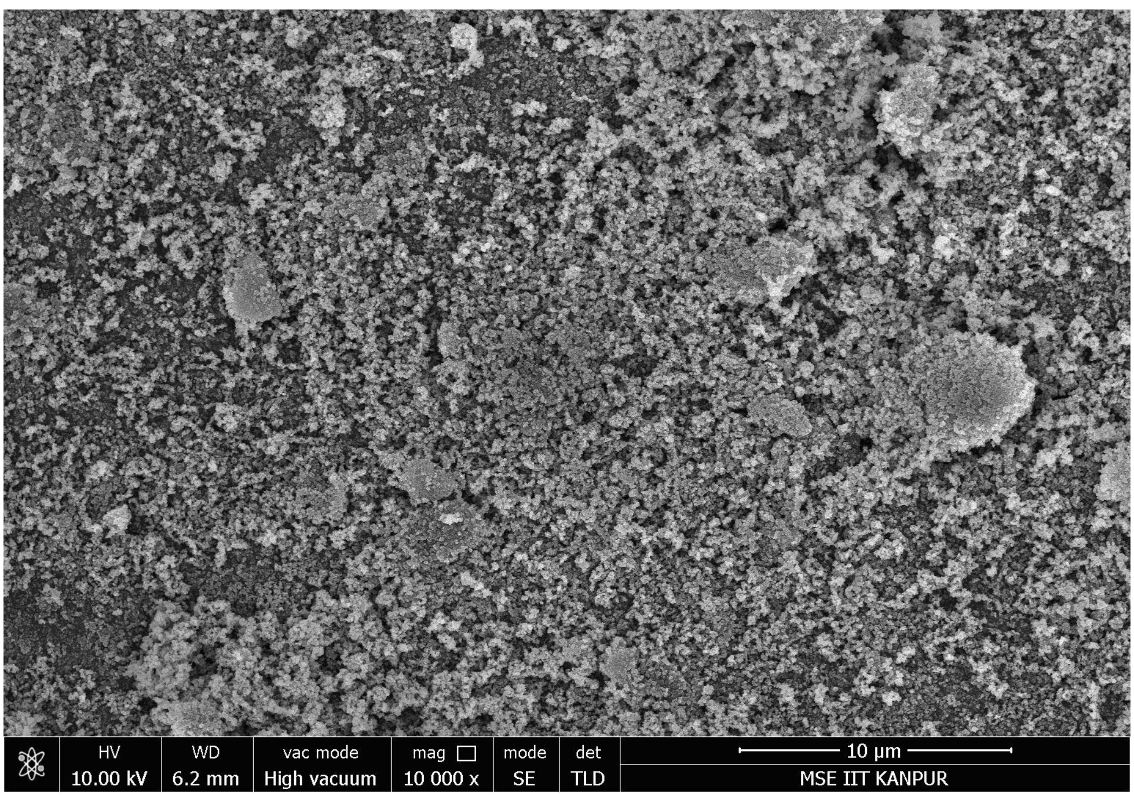



*Figure 3.3 SEM analysis of Ni0.4Mn0.6Fe2O4 B*

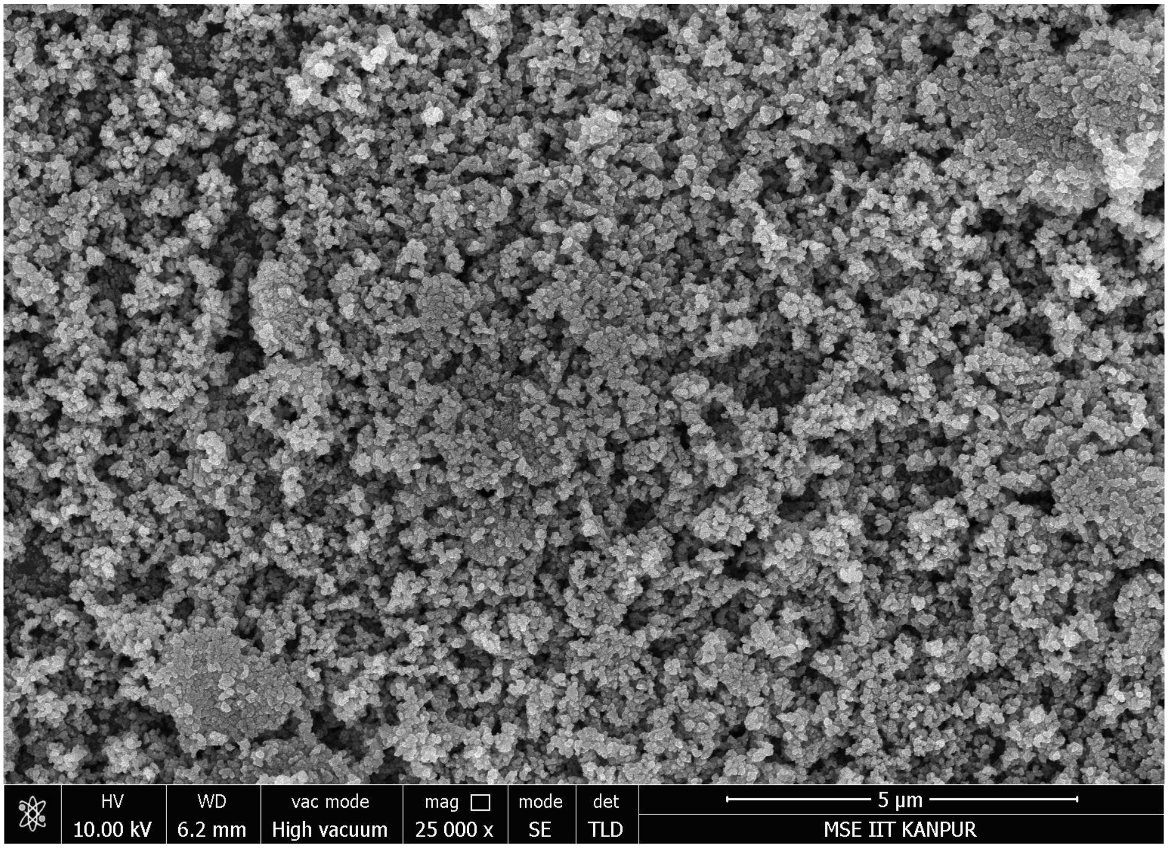



*Figure 3.4 SEM analysis of Ni0.4Mn0.6Fe2O4 C*

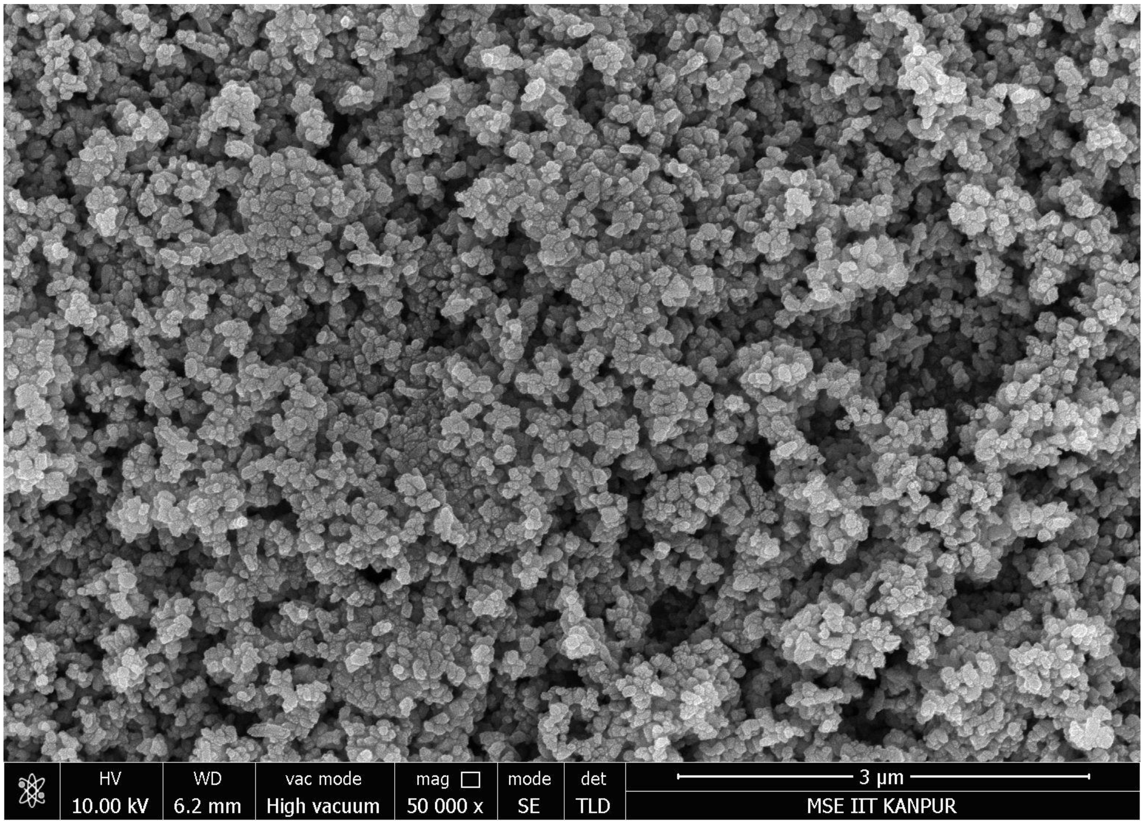



*Figure 3.5 SEM analysis of Ni0.4Mn0.6Fe2O4 D*

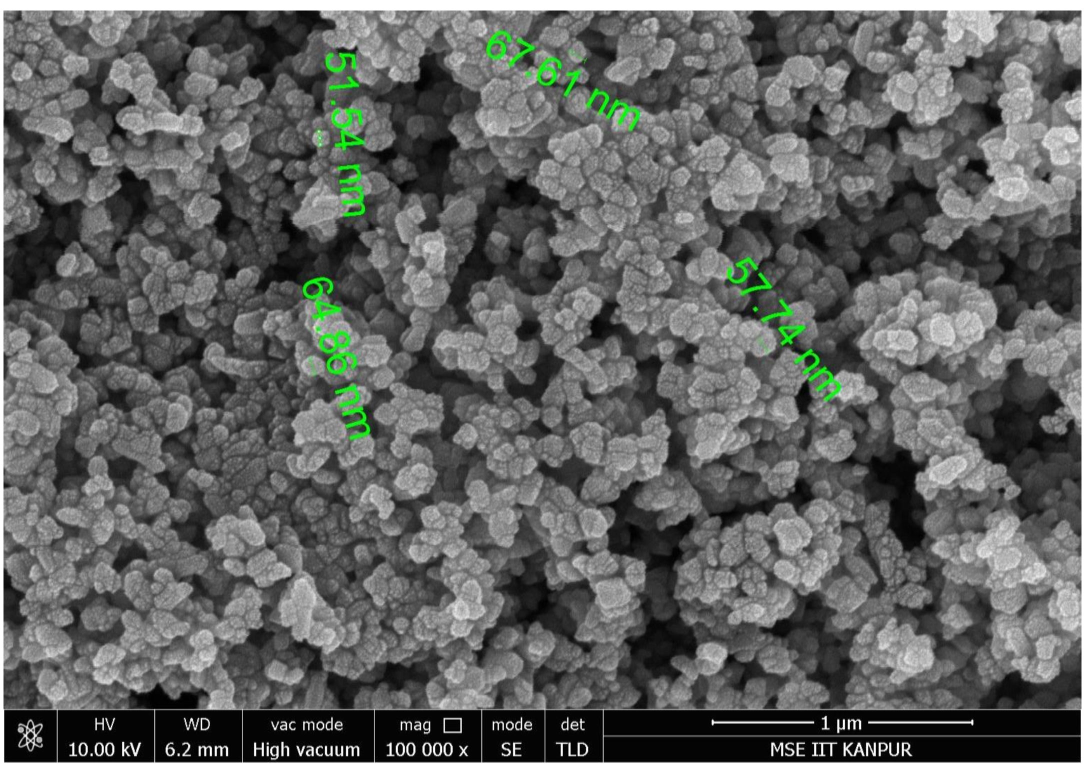



*Figure 3.6 SEM analysis of Ni0.4Mn0.6Fe2O4 E*

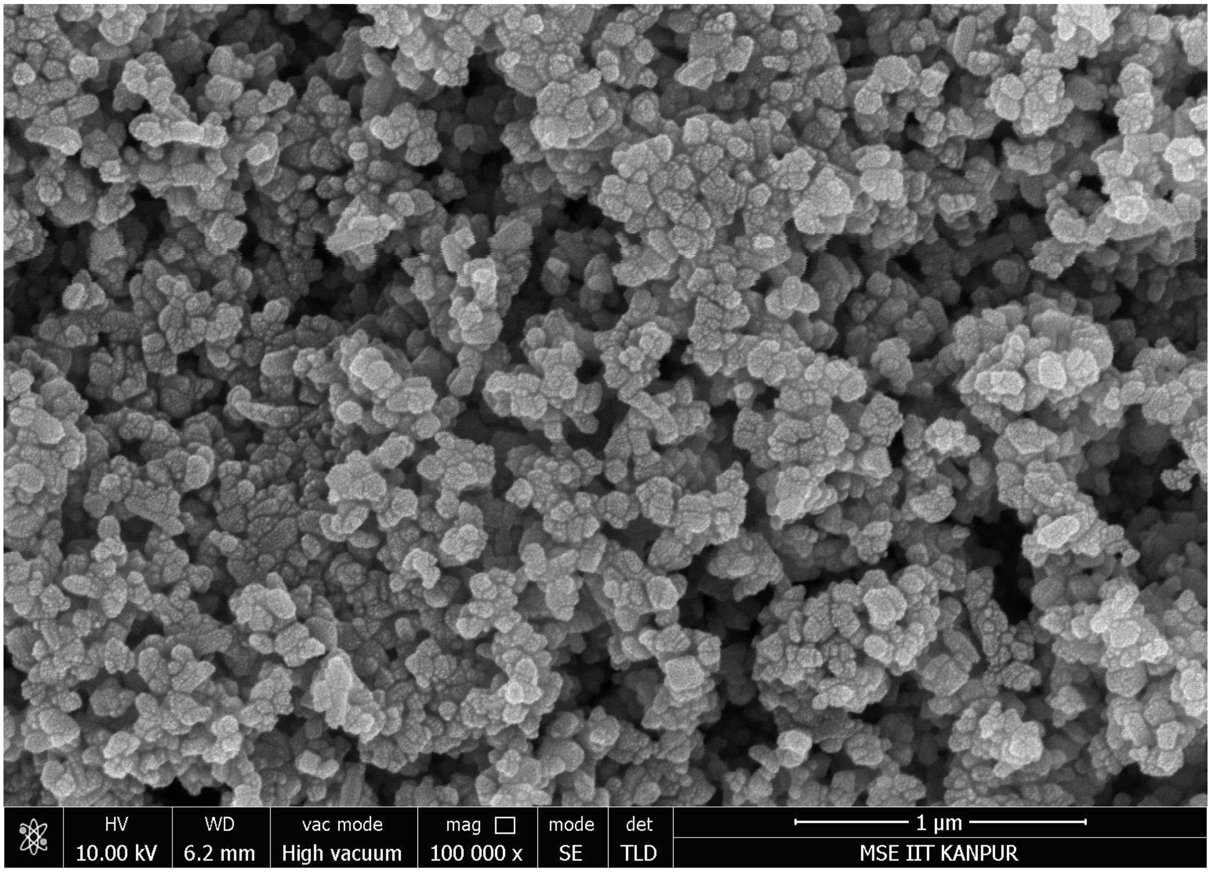



*Figure 3.7 SEM analysis of Ni0.4Mn0.6Fe2O4 Sample F*

SEM analysis of $Ni_{0.4}Mn_{0.6}Fe_2O_4$ at different magnifications

## 3.3 Electrochemical Properties

### 3.3.1 Cyclic Voltammetry Analysis

Prepared NPs (Nickel Manganese Ferrites) were characterized for the electrochemical properties in order to check the energy storage applications of synthesized particles using a three-electrode device system with 6M KOH solution as an electrolyte and the results shown below (Table. 3.3). Also, the CV curves are represented in Figures 3.8 to 3.12.

Specific Capacitance of all five samples were determined by using CV data. It was measured by using below Eq.

$$C = \frac{\int I \, dV}{mv\Delta V} \qquad (3.2)$$

Where, $\int I \, dV$= Area under CV curve and v is the scan rate (V/s), m is mass (g) of electroactive material coated on carbon paper.

*Table 3.3 Specific Capacitance of $Ni_xMn_{1-x}Fe_2O_4$*

| Sr. No. | Name of Sample | Area of CV Plot (Calculated by Origin) (mA x V) | Specific Capacitance F/g |
|---|---|---|---|
| 1 | $Ni_{0.2}Mn_{0.8}Fe_2O_4$ | 0.21751596 | 302.2 |
| 2 | $Ni_{0.4}Mn_{0.6}Fe_2O_4$ | 0.15992688 | 215.91 |
| 3 | $Ni_{0.6}Mn_{0.4}Fe_2O_4$ | 0.31333628 | 423 |
| 4 | $Ni_{0.8}Mn_{0.2}Fe_2O_4$ | 0.20117142 | 271.58 |
| 5 | $Ni_{1.0}Mn_{0.0}Fe_2O_4$ | 0.27547667 | 371.89 |

Maximum specific capacitance of 423 F $g^{-1}$ was observed for $Ni_{0.6}Mn_{0.4}Fe_2O_4$ i.e., mixed metal ferrite. The CV graphs are almost quasi-rectangular shaped, showing appreciable capacitive results.

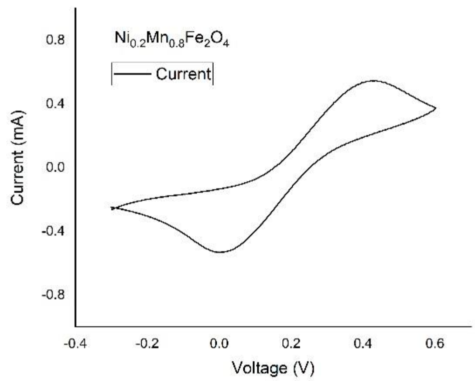



*Figure 3.8 CV Curve A of NixMn1-xFe2O4*

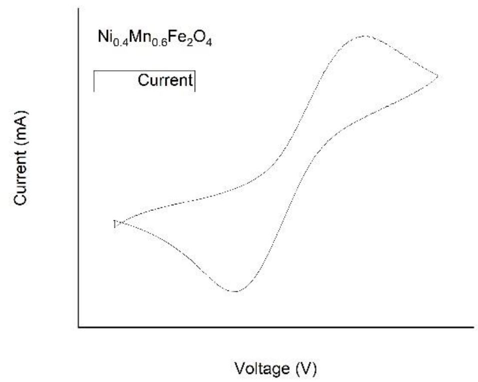



*Figure 3.9 CV Curve B of NixMn1-xFe2O4*

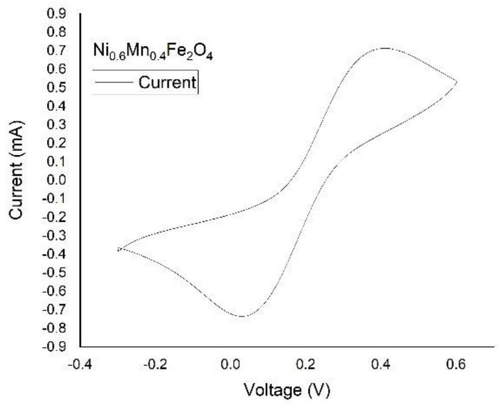



*Figure 3.10 CV Curve C of NixMn1-xFe2O4*

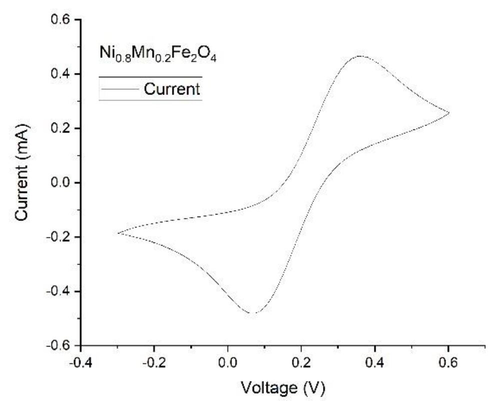



*Figure 3.11 CV Curve D of NixMn1-xFe2O4*

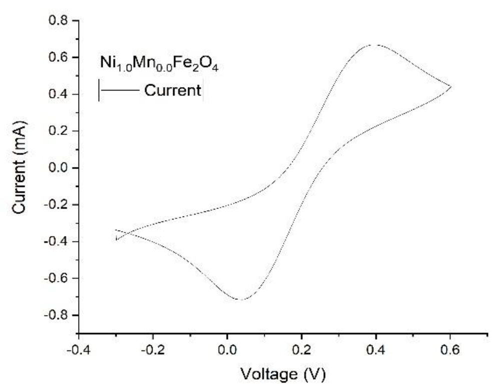



*Figure 3.12 CV Curve E of NixMn1-xFe2O4*

CV Curves of $Ni_xMn_{1-x}Fe_2O_4$

# Chapter 4

# 4 Conclusion

The synthesized samples were characterized for structural information and the results are in agreement with previously reported literature of crystal structures of ferrites and the mean crystalline size was calculated by Scherrer's Formula and was found to be approximately 2nm. Spherical morphology is revealed by the Sem analysis and images and size were found to approximately 50 nm. The electrochemical properties and CV Voltammetry analysis shows that mixed metal ferrites ($ABFe_2O_4$) have better capacitive performance as compared to metal ferrites ($AFe_2O_4$).

# 5 Future Scopes

The Capacitors are the future battery the only one limitation binding it to be successfully implementing it to the real world and its Capacity to hold charge as the amount of energy stored in capacitor is significantly lower than the conventional battery. Yet still many of the world leading technological company are interested and funding for a better material for capacitor. A new material with Super Capacitive application can significantly change the whole perspective of the electronics and real life.

1. Super capacitor has higher life expectancy than their battery counterpart.
2. Super Capacitor can be instantly Charged.
3. Super Capacitor can be recycled easily.

Although this endeavor seems too much more theoretical. But in past decade we already have achieved more better results in developing a better Super capacitor. Due to incorporation of various of the costly technique the current Super capacitor are very Expensive, But the right material which can be prepared easily and better production and management System will significantly bring the price down. This one of the objectives of this thesis to create and test a new Complex ferrite to test its application and comparison to the other ferrites by using Sol-gel method.

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