

STRUCTURAL, MORPHOLOGICAL, AND OPTICAL PROPERTIES OF MANGANESE OXIDE NANO PARTICLES

Submitted in partial fulfilment of the requirements for the award of
Bachelor of Science in Chemistry

By

Gangashree.V (40030008)

Akanksha kumari (40030001)

Geetha.V(40030009)



**DEPARTMENT OF CHEMISTRY
SCHOOL OF SCIENCE AND HUMANITIES**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **GANGASHREE.V (40030008)**, **AKANKSHA KUMARI (40030001)** and **GEETHA.V (40030009)** who carried out the project entitled "**STRUCTURAL, MORPHOLOGICAL, AND OPTICAL PROPERTIES OF MANGANESE OXIDE NANO PARTICLES**" under my/our supervision from August 2022 to April 2023.

INTERNAL GUIDE

Dr.S.SUNITHA

(Assistant professor)

EXTERNAL GUIDE

Dr.G.MURUGADOSS

(Associate Professor/Research)

08/05/2023
HEAD OF THE DEPARTMENT

Dr.J.KARTHIKEYAN Ph.D.

(Associate Professor)

Department of Chemistry

SATHYABAMA
INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)
Rajiv Gandhi Salai
Chennai - 600 119

Submitted for Viva voce Examination held on 08/05/2023

Internal Examiner

External Examiner

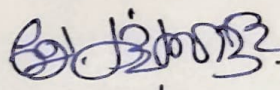
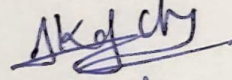
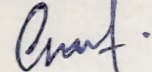
DECLARATION

We GANGASHREE.V (40030008), AKANKSHA KUMARI (40030001) and GEETHA.V(40030009) hereby declare that the project Report entitled “**STRUCTURAL, MORPHOLOGICAL, AND OPTICAL PROPERTIES OF MANGANESE OXIDE NANO PARTICLE**” done by us under the guidance of Dr.S.SUNITHA (Internal) and Dr.G.MURUGADOSS (External) centre of nanotechnology, Sathyabama Institute of science and Technology is submitted in partial fulfilment of the requirements for the award of Bachelor of Science degree in Chemistry.

DATE: 08/05/2023

PLACE: CHENNAI

SIGNATURE OF THE CANDIDATES

 (Gangashree.V)
 (Akanksha Kumari)
 (GEETHA.V)

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ABSTRACT

This project aims to study the structural morphological and optical properties of Manganese oxide (Mn_2O_3). Most recently manganese dioxides nanomaterials have attracted great interest anode materials in lithium-ion batteries for their high theoretical capacity, environmental benignity, low cost and special properties. The thrust to develop eco-friendly procedures for the production of nanoparticles arises from the extremely recent nanotechnology research. Mn_2O_3 nanoparticles were synthesized by co-precipitation method. The size, structure and morphology of Mn_2O_3 nanoparticles were characterized by UV-Visible, X-Ray diffraction (XRD), FTIR techniques. The average particle size of manganese oxide nanoparticles was calculated from the XRD. SEM images showed that the particles are almost spherical with varying sizes. Mn_2O_3 nanoparticles thus synthesized have large number of potential applications in the field of pharmaceutical industries, sensors, piezoelectric crystals, fuel cell electrodes and catalysis etc.

Keywords: Mn_2O_3 nanoparticles, Co-Precipitation Method, UV-VISIBLE, SEM, XRD FT-IR.

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LIST OF ABBREVIATION

- 1) nm – Nanometer
- 2) NPs – Nanoparticles
- 3) XRD - X-Ray Diffraction
- 4) UV – Ultra Violet
- 5) SEM – Scanning Electronic Microscope
- 6) FT-IR – Fourier Transform Infrared
- 7) MnO₂ - Manganese Dioxide
- 8) 1D - One Dimensional
- 9) 2D – Two Dimensional
- 10) 3D – Three Dimensional
- 11) NaOH -Sodium Hydroxide
- 12) DI – De-Ionized Water
- 13) PVP - Polyvinyl pyrrolidone

STRUCTURAL, MORPHOLOGICAL, AND OPTICAL PROPERTIES OF MANGANESE OXIDE NANO PARTICLES

CHAPTER 1

INTRODUCTION

1.1 NANOPARTICLES

Nanoparticles are extremely small particles, typically ranging in size from 1-100 nanometers (nm), which exhibit unique physical, chemical, and biological properties. They are composed of various materials, such as metals, polymers, ceramics, and carbon-based materials. Due to their small size and large surface area-to-volume ratio, nanoparticles have numerous applications in fields such as medicine, electronics, energy production, and environmental remediation. However, their small size also makes them potentially hazardous to human health and the environment. Therefore, understanding the properties and behavior of nanoparticles is critical to ensure their safe and effective use. Nanoparticles have revolutionized many fields with their unique properties. In medicine, nanoparticles can be engineered to specifically target cancer cells or deliver drugs to specific locations in the body. In electronics, nanoparticles can be used to improve the efficiency of solar cells and enhance the performance of electronic devices.

1.2 TYPES OF NANOPARTICLES

Nanoparticles can also be classified based on their dimensional properties. The three main types of nanoparticles based on dimensionality are zero-dimensional (0D), one-dimensional (1D), and two-dimensional (2D) nanoparticles. In addition to these, there are also hierarchical nanoparticles, which have multiple levels of dimensionality, such as core-shell nanoparticles. These types of nanoparticles have unique properties and find applications in various fields, such as drug delivery and biomedical imaging.

1.2.1 ZERO DIMENSIONAL NANOPARTICLES

Zero-dimensional nanoparticles, also known as quantum dots, are particles with all dimensions in the nanoscale range. They have a spherical shape and are used in various applications such as biological imaging and photovoltaic cells.

1.2.2 ONE DIMENSIONAL NANOPARTICLES

One-dimensional nanoparticles have one dimension in the nanoscale range and the other two dimensions in the micro or macro scale. Examples of 1D nanoparticles include nanowires and nanotubes, which have applications in electronics and energy production.

1.2.3 TWO DIMENSIONAL NANOPARTICLES

Two-dimensional nanoparticles have two dimensions in the nanoscale range and the third dimension in the micro or macro scale. Examples of 2D nanoparticles include graphene and other 2D materials, which have applications in electronics, energy production, and catalysis.

1.2.4 THREE DIMENSIONAL NANOPARTICLES

Three-dimensional nanoparticles (3D-NPs) are a class of nanomaterials that have all three dimensions in the nanoscale range. Unlike zero-dimensional nanoparticles (0D-NPs) and one-dimensional nanoparticles (1D-NPs), which have only one or two dimensions in the nanoscale range, respectively, 3D-NPs have unique physical, chemical, and biological properties that make them attractive for a variety of applications.

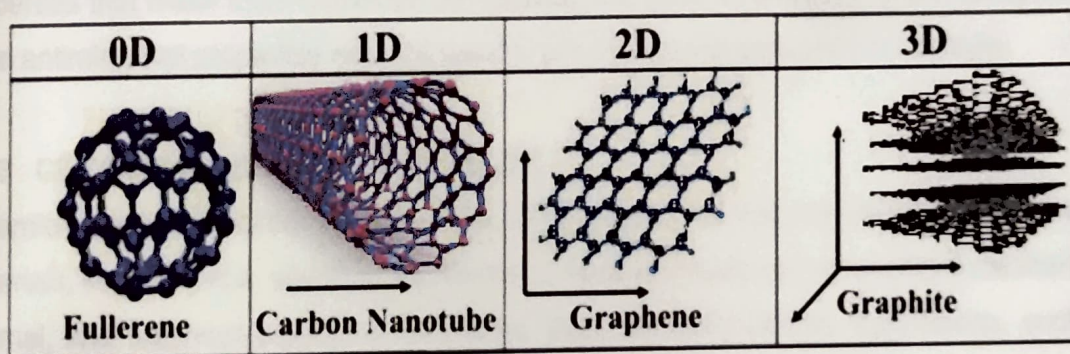


FIGURE 1.1 TYPES OF NANOPARTICLES IN DIMENSIONAL

1.3 CLASSIFICATION OF NANOPRTICLES

Nanoparticles can be classified based on various factors, including their composition, size, and shape. One common classification is based on the material they are composed of. Metal nanoparticles, such as gold, silver, and copper, find wide applications in fields such as electronics, catalysis, and biomedicine. Polymer nanoparticles, made up of polymers like polyethylene glycol and polystyrene, are often used in drug delivery and gene therapy. Quantum dots are semiconductor nanoparticles made of materials like cadmium selenide and zinc sulfide, which have applications in biological imaging and photovoltaic cells

1.3.1 CARBON BASED NANOPARTICLES

Carbon-based nanoparticles are a class of nanomaterials that are composed of various forms of carbon, including carbon nanotubes, fullerenes, and graphene. These nanomaterials have unique properties that make them attractive for various applications in fields such as electronics, energy production, and drug delivery. Graphene is a two-dimensional sheet of carbon atoms that are arranged in a hexagonal lattice. It has excellent electrical and thermal conductivity, high surface area, and is mechanically strong. These properties make it useful in applications such as electronics, energy storage, and water purification.

1.3.2 METAL BASED NANOPARTICLES

Metal-based nanoparticles are a class of nanomaterials that are composed of metals or metal alloys, such as gold, silver, and iron oxide. These nanomaterials have unique physical and chemical properties that make them attractive for various applications, such as catalysis, sensing, and biomedical imaging. For example, gold nanoparticles have unique optical properties that make them useful for bio-sensing and medical imaging. Silver nanoparticles have antimicrobial properties and are used in wound healing and water purification.

1.3.3 CERAMIC BASED NANOPARTICLES

Ceramic-based nanoparticles are a class of nanomaterials that are composed of ceramic materials, such as silica, alumina, and titania. These nanomaterials have unique mechanical, thermal, and chemical properties that make them useful in various applications, such as catalysis, energy production, and biomedical imaging.

1.3.4 SEMICONDUCTOR NANOPARTICLES

Semiconductor-based nanoparticles are a class of nanomaterials that are composed of semiconductor materials, such as silicon, cadmium selenide, and zinc oxide. These nanomaterials have unique electronic and optical properties that make them useful in various applications, such as electronics, energy production, and sensing. For example, cadmium selenide nanoparticles are used in solar cells and LEDs due to their unique optical properties, while silicon nanoparticles are used in electronic devices. Zinc oxide nanoparticles are used in UV sensing and photo catalysis.

1.4 MANGANESE OXIDE NANOPARTICLES

Manganese oxide is a chemical compound that consists of manganese and oxygen atoms. It exists in various forms, including MnO , MnO_2 , Mn_2O_3 and Mn_3O_4 . Manganese oxide has many industrial applications, such as in the production of batteries, ceramics, and glass. It is also used as a catalyst in various chemical reactions and as a pigment in paints and coatings.

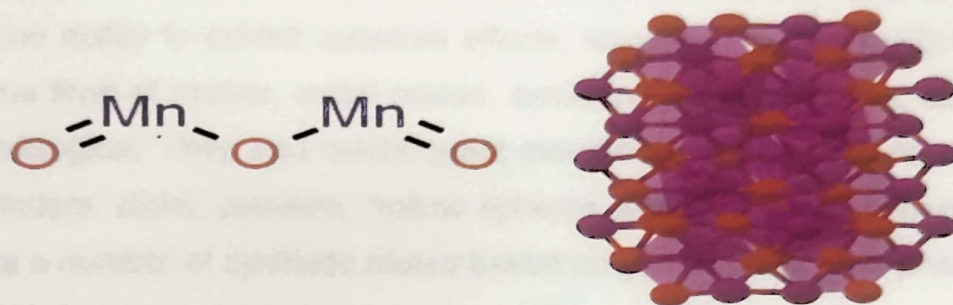


FIGURE 1.2 Mn_2O_3 STRUCTURE

1.4.1 APPLICATIONS OF MnO_2

New research shows that manganese dioxide nanomaterials also have great potential in gene therapy and nuclear magnetic imaging. In recent days, manganese dioxide nanoparticles (Mn_2O_3 nanoparticles) have intrigued material science researches extensively due to its wide range of applications. They are widely used in energy storage devices (lithium-ion batteries, capacitors), catalysts, adsorbent, sensors and imaging, therapeutic activity, etc. Since they hold a lot of distinguished potentials, a robust protocol for cheap, stable, biocompatible and eco-friendly Mn_2O_3 nanoparticles is necessary

1.4.2 Mn_2O_3 NANOCOMPOSITE

Manganese oxide nanoparticles are a class of nanomaterials that are composed of manganese and oxygen atoms. They have unique physical and chemical properties that make them attractive for various applications in fields such as catalysis, energy storage, and biomedical imaging. Manganese oxide nanoparticles also find applications in energy storage, particularly in the development of super capacitors and lithium-ion batteries. Mn_3O_4 nanoparticles have been shown to have high electrochemical performance and high stability, making them attractive for use in battery electrodes. In biomedical applications, manganese oxide nanoparticles can be used as contrast agents in magnetic resonance imaging (MRI). Mn_3O_4 nanoparticles have been shown to have superior MRI contrast properties compared to other contrast agents, making them useful for diagnostic imaging.

CHAPTER 2

2.1 LITERATURE SURVEY

Nanoparticles are defined as particles with size in the range of 1 to 100 nm at least in one of the three dimensions. Because of this very small size scale, they possess an immense surface area per unit volume, a high proportion of atoms in the surface and near surface layers, and the ability to exhibit quantum effects. Nanoparticles exist with great chemical diversity in the form of metals, metal oxides, semiconductors, polymers, carbon materials, organics or biological. They also exhibit great morphological diversity with shapes such as spheres, cylinders, disks, platelets, hollow spheres and tubes, etc. Nanoparticles can be generated via a number of synthetic routes based on gas, liquid or solid phase approaches. The synthesized nanoparticles have to be surface modified in most cases, in order to passivate and stabilize them since their nanoscale renders them chemically very reactive and/or physically aggregative. The nanoparticles are also surface functionalized in order to meet the needs of specific applications (Nagarajan et al., 2008)

(Oskam et al., 2006) The synthesis of metal oxide nanoparticles is described in terms of precursor formation, nucleation, growth, and aging processes. The main parameters governing these processes are the solution properties, including the solvent viscosity, dielectric constant and the presence of adsorbing anions, the solubility of the metal oxide, and the metal oxide surface energy.

Over the last three decades nanoparticles have received an increasing amount of research interest. This is due to unique size dependent properties of nanoparticles, which are often thought as a separate and intermediate state of matter lying between individual atoms and bulk material (Schnid, 2004).

Nanoparticles consisting of a large range of transition metals and metal oxides have found to exhibit advantageous size dependent catalytic properties and are being investigated. Physical vapor deposition, chemical vapor deposition, aerosol processing, Sol-Gel process, reverse micelle method, mechanical alloying/ milling, are some of the commonly used methods by which nanoparticles can be synthesized.

Among the various wet chemical methods to synthesize nanoparticles, hydrothermal (Yanagisawa et al, 2000; Xu et al, 2000), microemulsion (Sujata et al, 2002; Kim et al, 2000) and sol-gel (Shukla et al, 2004), conventional co-precipitation method is commercially widely used because of its cost-effective (Nam et al, 2001; Kim et al, 2006; Kim and Park et al, 2006; Praminik et al, 2002; Seo et al, 2006; Kim et al, 2002)

Co-precipitation method offers advantages like, simple and rapid preparative method, easy control of particle size and composition can be made in this method and also, there are various possibilities to modify the particle surface state and overall homogeneity. Co-precipitation of various salts (nitrates, sulphates, chlorides, perchlorates etc.) under a fine control of pH by using NaOH or NH_4OH solutions yields corresponding spinel oxide nanoparticles.

In recent days, manganese oxide nanoparticles have intrigued material science researches extensively due to its wide range of applications. They are widely used in energy storage devices catalysts, adsorbent, sensors and imaging, therapeutic activity, etc. Since they hold a lot of distinguished potentials, a robust protocol for cheap, stable, biocompatible and eco-friendly Mn_2O_3 NPs is necessary. They can be categorized into different phases like α , β , δ and others. Thus, owing to their peculiar character, they could be utilized for various purposes depending on the mode of action and applications. Hence, this review has summarized conventional methods, such as hydrothermal, sol-gel, oxidation-reduction used for the generation of Mn_2O_3 NPs. (Dawadi et al., 2020)

Mn_2O_3 nanoparticles were synthesized by co-precipitation method. As an important functional metal oxide, manganese oxide nanoparticles are one of the most attractive inorganic materials because of its physical and chemical properties and wide applications in catalysis, ion exchange, molecular adsorption, biosensor, and particularly, energy storage (Qi et al.1999; Shen et al., 1993; Cao & Suib, 1994)

Magnesium oxide (Mn_2O_3) nanostructures were prepared by chemical route from hydro manganese chloride salt using Potassium hydroxide as reducing agent. X-Ray diffraction (XRD), SEM and Fourier transformation infrared (FTIR) used to characterize both particle size and structure of Mn_2O_3 nanoparticles. XRD results confirmed impurity of synthesized powder with α - Mn_2O_3 as predominant phase. The average particle size of manganese dioxide was in the range 25- 30 nm. Photographs of scanning electron microscope (SEM) showed cluster agglomeration and chain appearance. (Catalano et al., 2021)

Manganese dioxide nanoparticles were synthesized using co-precipitation method for 0.1 mol and 0.2mol concentration, which is the simple way to prepare the aqueous solution. The dried samples were characterized using various techniques such as XRD, SEM, FTIR and UV-Visible spectroscopy. The average particle size of manganese oxide nanoparticles was calculated from the XRD study. The Morphology and size of the synthesized Mn_2O_3 particles are studied by scanning electron microscopy (SEM). The band gap energy for Mn_2O_3 is found using UV-Visible spectroscopy. The FT-IR spectra of Mn_2O_3 shows the peaks corresponding to stretching and bending vibration. There are large number of potential applications of Mn_2O_3 metal nanoparticles such as in the field of electrode materials in different rechargeable batteries, biosensors, coatings, and bioscience applications. (Subramanian et al., 2004)

(Ganeshan et al., 2017) Manganese oxide nanostructures are of considerable importance in technological applications and have been intensively investigated as promising electrode material in primary/secondary batteries and electrochemical capacitors due to their excellent electrochemical properties, low cost, environmentally benign and ease of preparation. Mn_2O_3 nanoparticles are synthesized using Co-Precipitation method. The structural and optical characterization was carried out using Powder X-ray Diffraction method, Scanning Electron Microscope and Energy Dispersive X-ray Spectroscopy, The powder X-ray Diffraction studies revealed the polycrystalline nature of Mn_2O_3 nanoparticles. SEM images showed that the particles are almost spherical with varying sizes. Energy Dispersive X-ray Spectroscopy analysis confirms the presence of Manganese and Oxygen.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

To study the structural, morphological, and optical properties of manganese oxide nanoparticles.

3.2 SCOPE

Characterization of the synthesized Mn_2O_3 nanoparticles is crucial for understanding their properties and potential applications.

Determination of structural properties of Manganese (III) oxide nanoparticles.

Determination of optical properties of Manganese (III) oxide nanoparticles.

Morphology of Manganese (III) oxide

Techniques such as FTIR, UV-visible, X-ray diffraction, scanning electron microscopy, and spectroscopy can be used to study the size, shape, crystallinity, and surface properties of the nanoparticles.

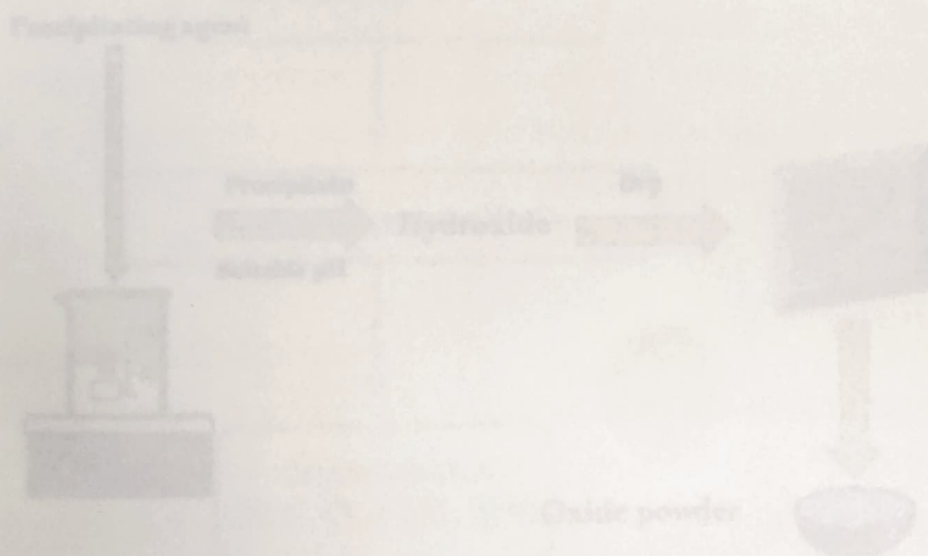


FIGURE 4.1 CO PRECIPITATION METHOD

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIAL

Manganese chloride, Distilled water, Ethanol, Acetone, Sodium hydroxide PVP, Ethylene glycol.

4.2 EXPERIMENTAL METHOD

Co-precipitation is a standard synthesis method for preparation of oxide nanoparticles. In this method a salt precursor, commonly nitrate, chloride, or oxychloride, is dissolved in water and then the corresponding hydroxide is precipitated by addition of a base such as sodium hydroxide or ammonium hydroxide.

Co-precipitation is a cost-effective and fast process and is easily implemented on a larger scale for industrial application. It is an efficient method for synthesizing nanomaterials without requiring hazardous organic solvents.

The Co-precipitation method was performed by using manganese salt like Manganese (II) chloride of 0.5M dissolved in double distilled water with continuous stirring at constant temperature of 120°C. While stirring, 1.0M NaOH solution was added till the pH of the solution become 12. The stirring was continued for 2 hour at a constant temperature of 120°C. Black precipitate formed was then filtered and washed with ethanol or acetone

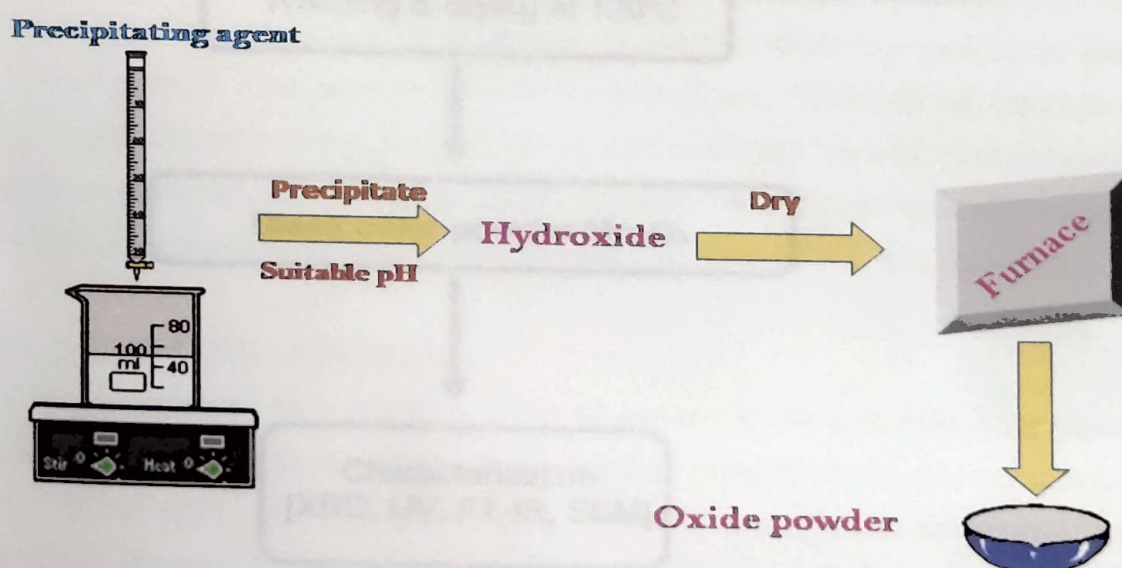
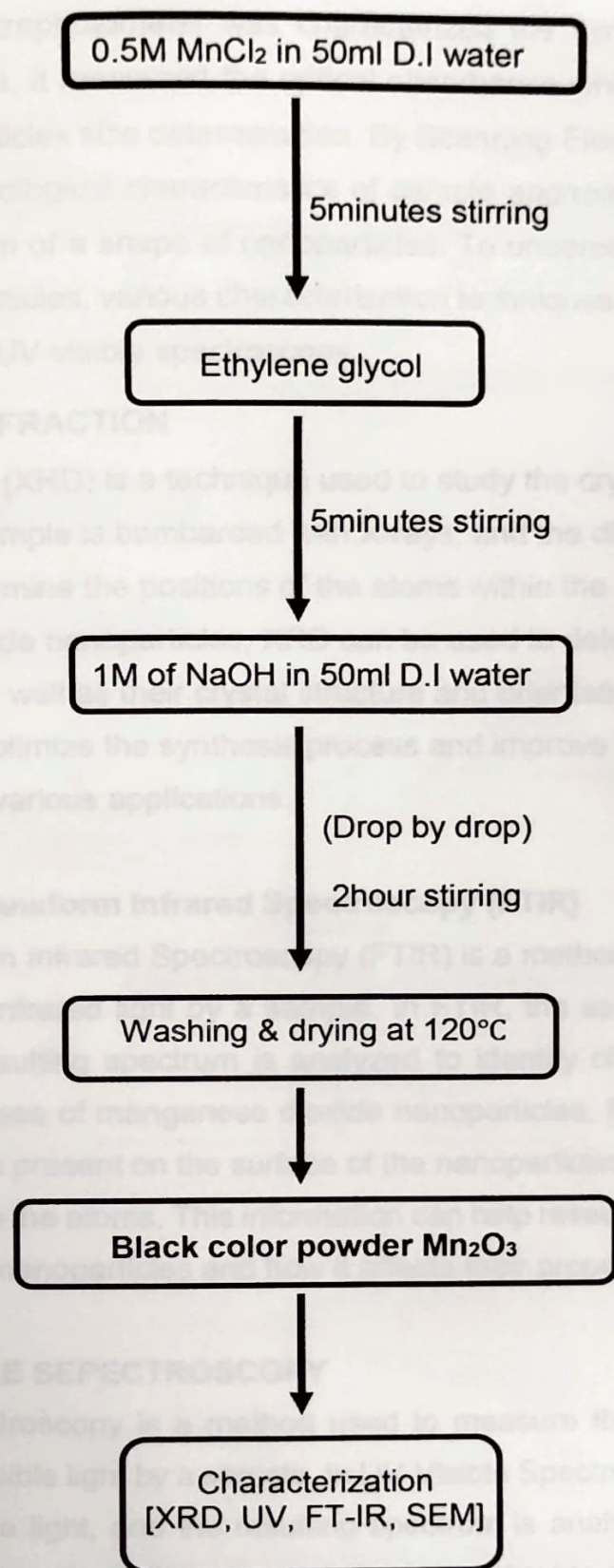


FIGURE 4.1 CO PRECIPITATION METHOD

4.2.1 SYNTHESIS OF NANOPARTICLES

SYNTHESIS



4.3 CHARACTERIZATION TECHNIQUES

UV-visible spectrophotometer was characterized the synthesized nanoparticles of peak absorbance curve, it measured the optical absorbance which is demonstrate the values of bandgap and particles size determination. By Scanning Electron Microscopy (SEM) analysis measured morphological characteristics of sample approximation to determine the particle diameter and form of a shape of nanoparticles. To understand the properties and behavior of Mn_2O_3 nanoparticles, various characterization techniques can be employed, such as XRD, FTIR, SEM, and UV-visible spectroscopy.

4.3.1 X-RAY DIFFRACTION

X-ray Diffraction (XRD) is a technique used to study the crystal structure of a material. In this method, a sample is bombarded with X-rays, and the diffraction pattern produced is analyzed to determine the positions of the atoms within the crystal lattice. In the case of manganese dioxide nanoparticles, XRD can be used to determine the size and shape of the nanoparticles, as well as their crystal structure and orientation. This information can help researchers to optimize the synthesis process and improve the performance of the nanoparticles in various applications.

4.3.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a method of measuring the absorption or transmission of infrared light by a sample. In FTIR, the sample is illuminated with infrared light, and the resulting spectrum is analyzed to identify chemical functional groups in the sample. In the case of manganese dioxide nanoparticles, FTIR can be used to identify the functional groups present on the surface of the nanoparticles, as well as the type of chemical bonding between the atoms. This information can help researchers to understand the surface chemistry of the nanoparticles and how it affects their properties and behavior.

4.3.3 UV-VISIBLE SEPECTROSCOPY

UV-Visible Spectroscopy is a method used to measure the absorption or transmission of ultraviolet and visible light by a sample. In UV-Visible Spectroscopy, the sample is illuminated with UV or visible light, and the resulting spectrum is analyzed to determine the electronic structure of the sample. In the case of manganese dioxide nanoparticles,

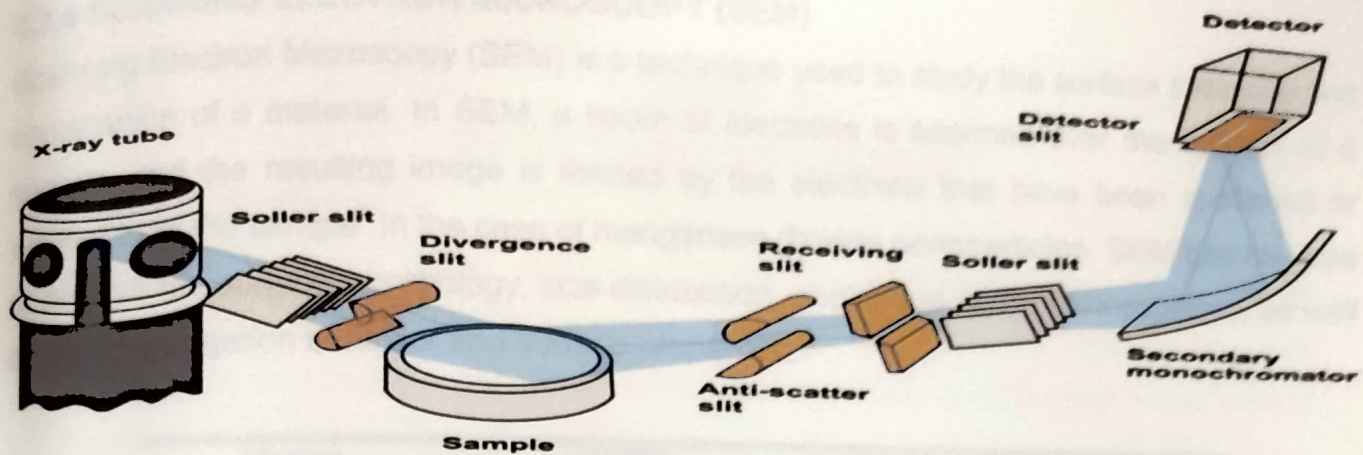


FIGURE 4.2 XRD WORKING PRINCIPLE

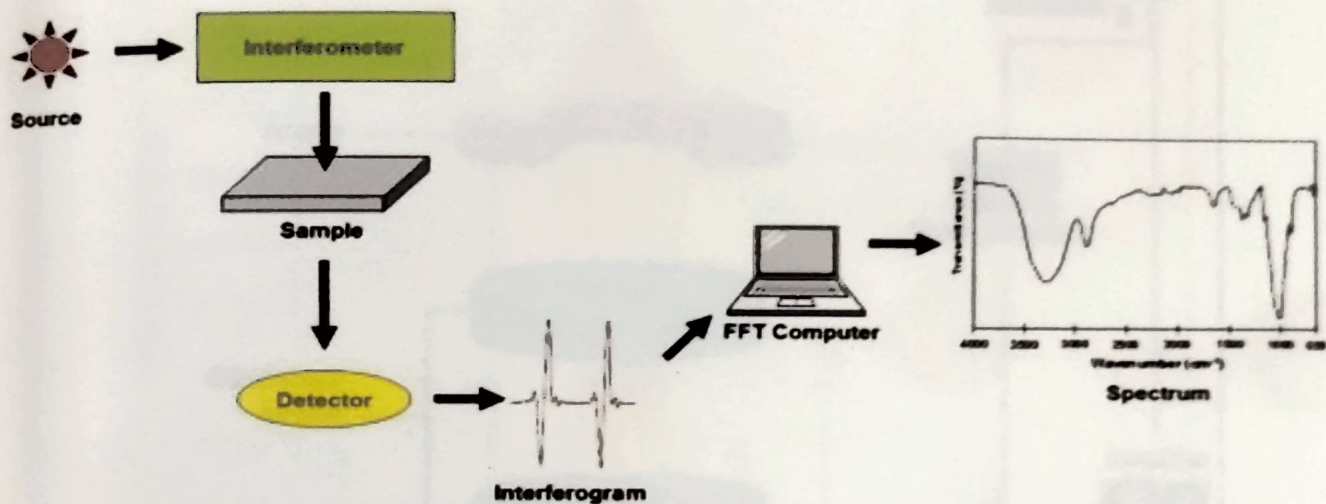


FIGURE 4.3 FTIR WORKING PRINCIPLE

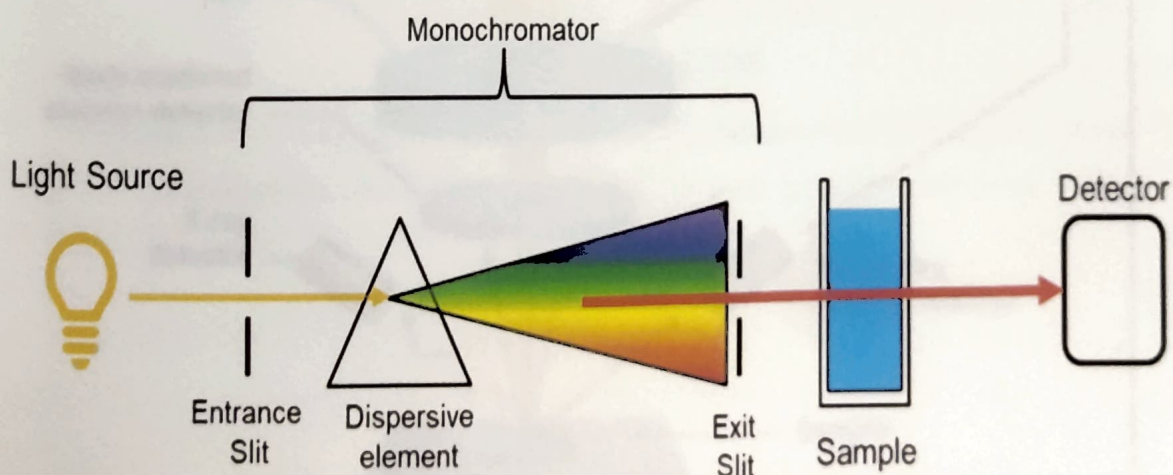


FIGURE 4.4 UV-VIS WORKING PRINCIPLE

4.3.4 SCANNING ELECTRON MICROSCOPY (SEM)

Scanning Electron Microscopy (SEM) is a technique used to study the surface structure and composition of a material. In SEM, a beam of electrons is scanned over the surface of a sample, and the resulting image is formed by the electrons that have been scattered or emitted from the sample. In the case of manganese dioxide nanoparticles, SEM can be used to analyze the surface morphology, size distribution, and shape of the nanoparticles, as well as their aggregation behavior and surface properties.

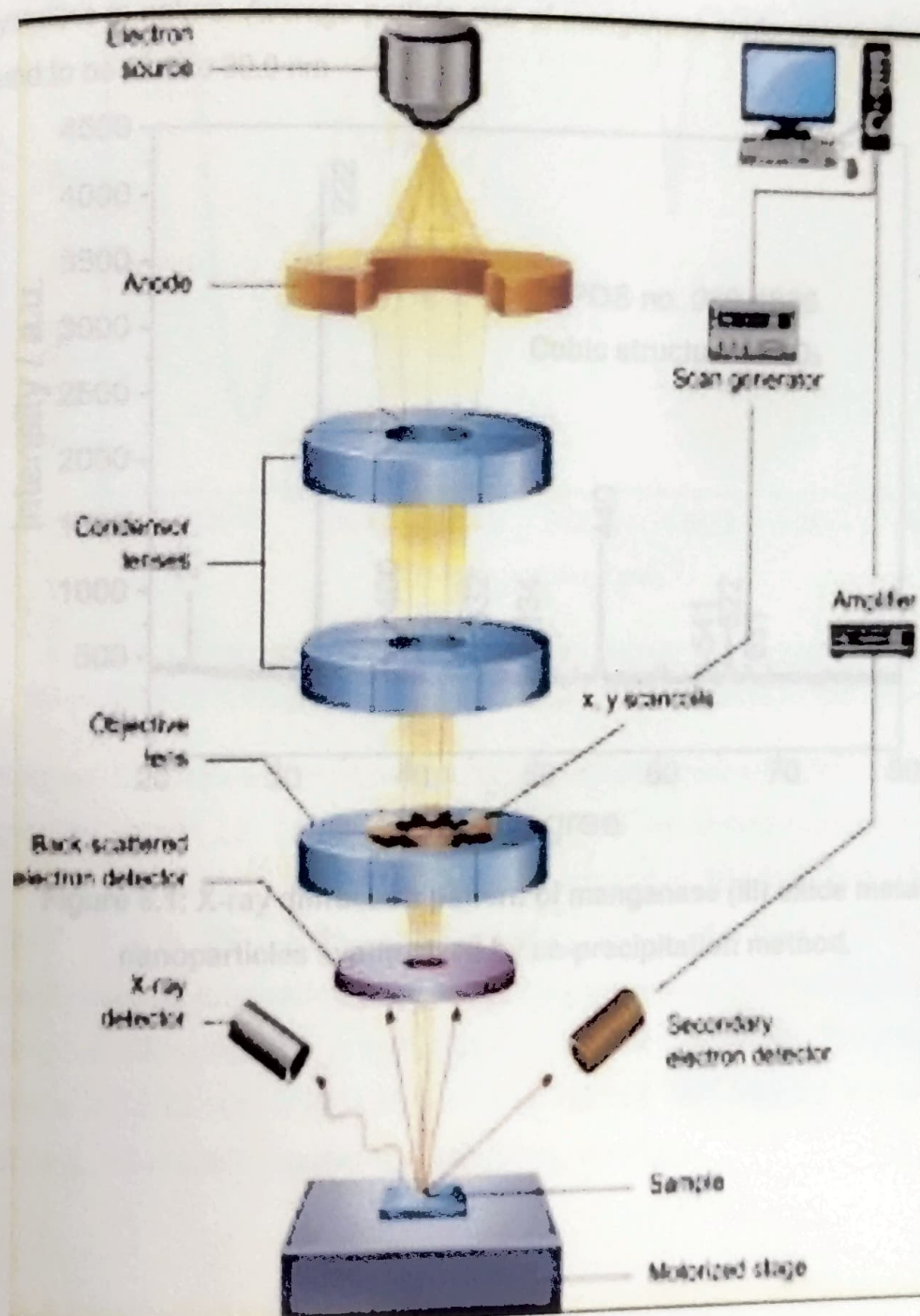


FIGURE 4.5 SEM WORKING PRINCIP

Chapter 5

RESULT AND DISCUSSION

X-RAY DIFFRACTION

Figure 5.1 shows X-ray diffraction study of manganese (III) oxide metal nanoparticles synthesized by co-precipitation method. From the XRD pattern it is clear that manganese (III) oxide metal nanoparticles synthesized were purely crystalline in nature. Average particle size of manganese oxide nanoparticles was found to be 23.0 to 30.0 nm

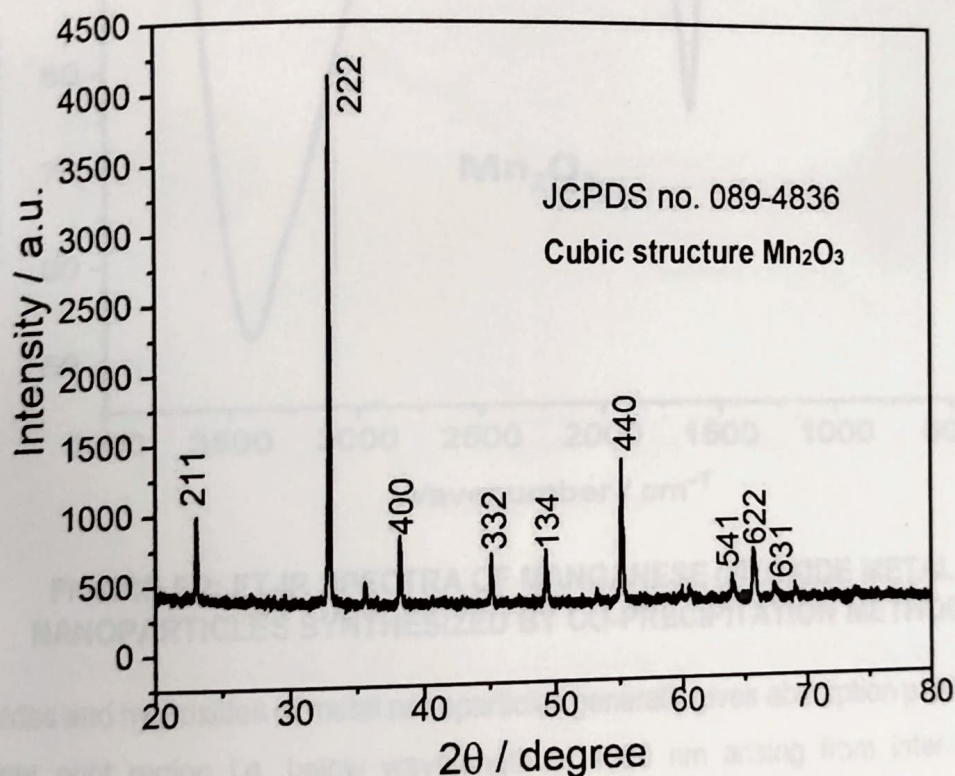


Figure 5.1: X-ray diffraction pattern of manganese (III) oxide metal nanoparticles synthesized by co-precipitation method.

FTIR Spectroscopy

Figure 5.2 shows FT-IR spectra of manganese (III) oxide metal nanoparticles synthesized by co-precipitation method. FTIR spectroscopy was carried out in order to ascertain the purity and nature of manganese (III) oxide metal nanoparticles as synthesized by co-precipitation method

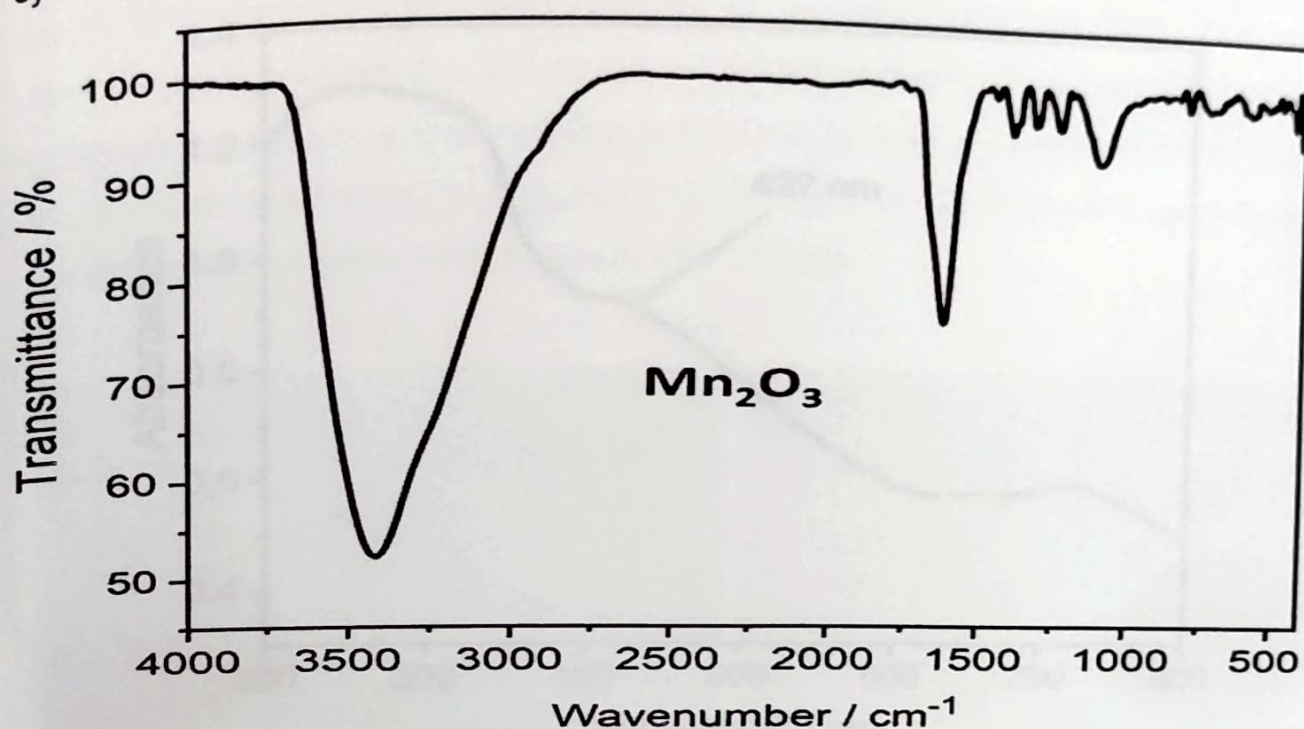


FIGURE 5.2: FT-IR SPECTRA OF MANGANESE (III) OXIDE METAL NANOPARTICLES SYNTHESIZED BY CO-PRECIPITATION METHOD.

Oxides and hydroxides of metal nanoparticles generally gives absorption peak in the finger print region i.e. below wavelength of 1000 nm arising from inter-atomic vibrations. The bands at 515 and 480 cm⁻¹ correspond to the Mn–O bond (Kang et al, 2007; Li et al, 2007; Potter and Ross man, 1979). From the above result we conclude that the synthesized nanomaterial is manganese oxide. Absorption peak observed at 2924.09 cm⁻¹ may be due to –CH₃ stretching vibrations. The absorption peaks at 2852.72 cm⁻¹ , 2021.40 cm⁻¹ and 1382.96 cm⁻¹ may be due to –CH₂ stretching, =C–H stretching and –C–H stretching vibrations

UV-VISIBLE SPECTROSCOPY

Figure 5.3 shows UV-Visible spectra of manganese (III) oxide metal nanoparticles synthesized by co-precipitation method as a function of wavelength. The UV-visible absorption shows sharp absorption at 427 nm due to manganese (III) oxide metal nanoparticles.

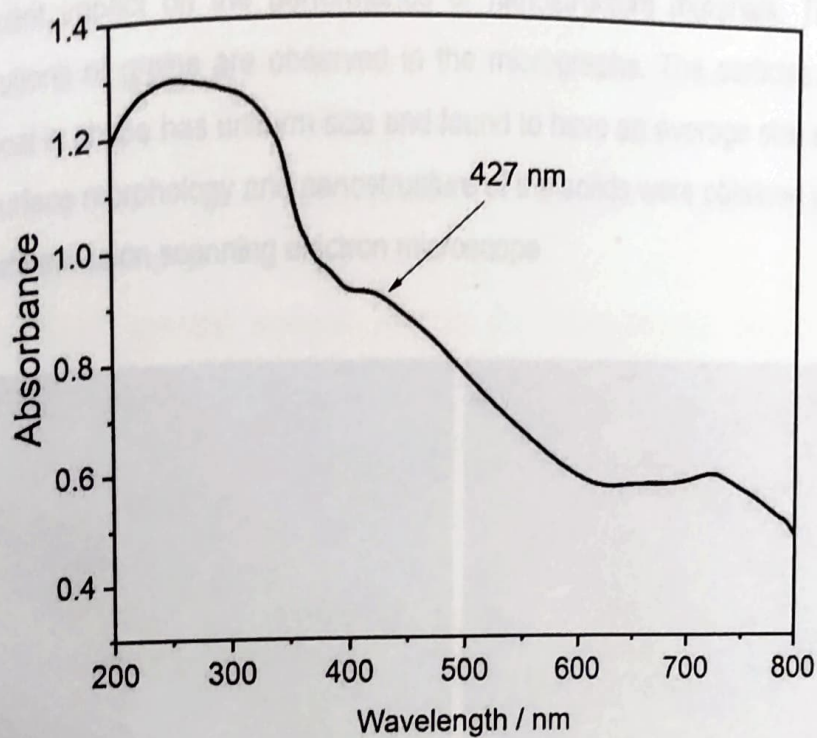


FIGURE 5.3: UV-VISIBLE SPECTRA OF MANGANESE (III) OXIDE METAL NANOPARTICLES SYNTHESIZED BY CO-PRECIPITATION METHOD.

FIGURE 5.4 SEM IMAGES OF AS PREPARED Mn_2O_3 NANOPARTICLES

SCANNING ELECTRON MICROSCOPY (SEM)

The surface morphological studies of nanoparticles have been performed by Scanning Electron Microscope (SEM). The SEM images of Mn_2O_3 nanoparticles are portrayed in Fig 5.4. It was well documented that the surface morphology has significant impact on the performance of nanostructure materials. The uniform distributions of grains are observed in the micrographs. The particles are nearly spherical in shape has uniform size and found to have an average size of about 50 nm. Surface morphology and nanostructure of the solids were obtained with the aid of a field emission scanning electron microscope

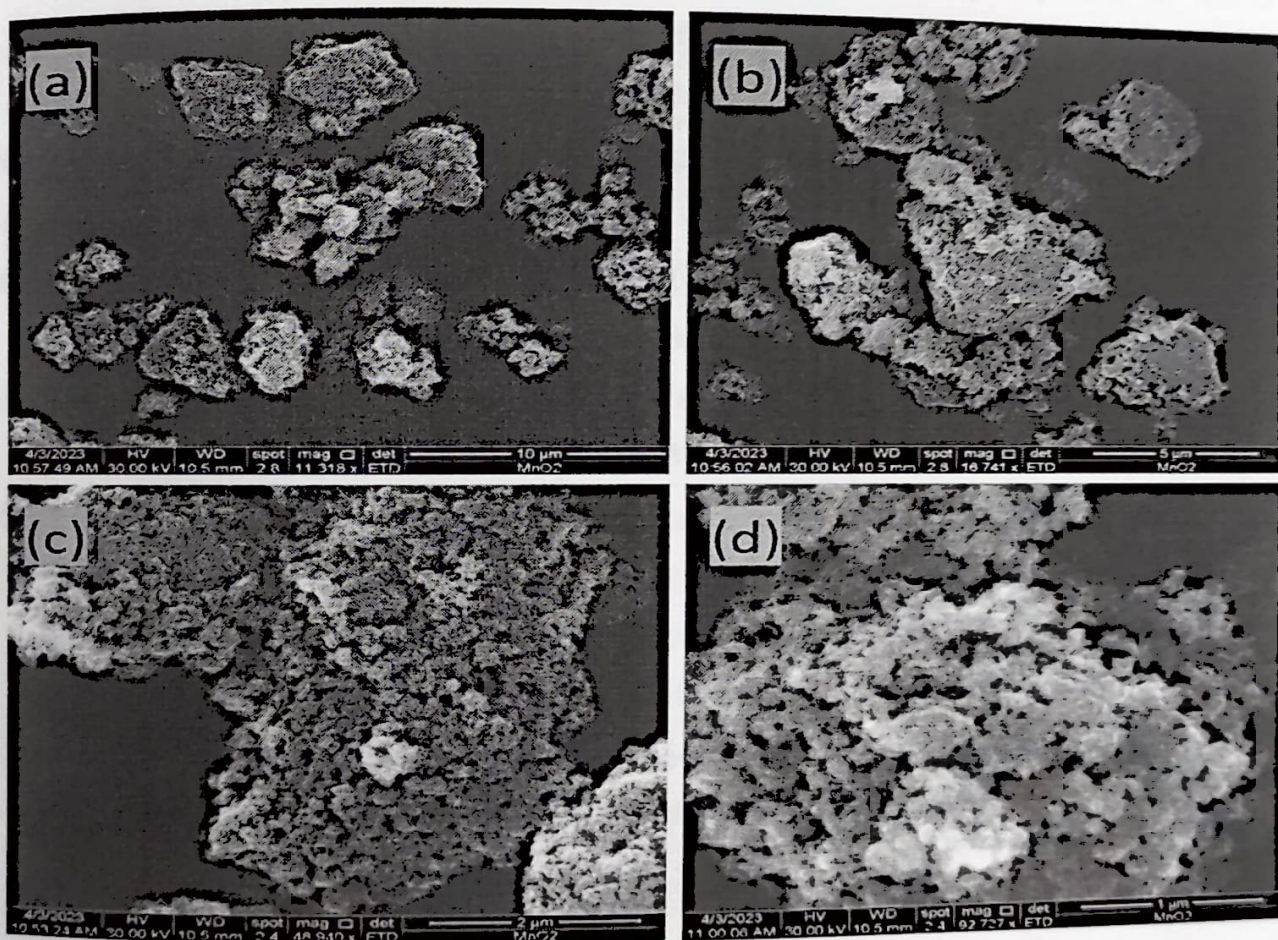


FIGURE 5.4 SEM IMAGES OF AS PREPARED Mn_2O_3 NANOPARTICLES

CHAPTER 6

SUMMARY AND CONCLUSION

- ❖ Mn_2O_3 nanoparticles have been successfully synthesized by Co-Precipitation method.
- ❖ The nanoparticles are characterized using advanced characterization techniques such as XRD, SEM, UV and FTIR techniques.
- ❖ The synthesized Mn_2O_3 nanoparticles have found in in cubic crystal structure with high crystal quality.
- ❖ The FT-IR spectral analysis reveals the characteristics peaks of Mn-O stretching along with some addition surface functional groups due to surfactant was used during the sample preparation.
- ❖ The UV-visible absorption shows sharp absorption peak at 427 nm in the blue region due to metal nanoparticles.
- ❖ Highly homogeneous with spherical shape of Mn_2O_3 nanoparticles was confirmed by the scanning electron microscopy. The homogeneous growth of the nanoparticles production is more suitable for scale-up the materials production in industrial application using the facile chemical method.
- ❖ There are large numbers of potential applications can be used the chemically synthesized Mn_2O_3 metal nanoparticles due to its high crystal quality, visible light absorption and homogeneous growth. So for the Mn_2O_3 nanoparticles are successfully used the following applications such as field of electrode materials in different rechargeable batteries, biosensors, coatings, nanofibers, nanowires and also in specific biogenic and bioscience applications.

CHAPTER 7

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