

**SYNTHESIS AND CHARACTERIZATION OF MANGANESE OXIDE
NANOPARTICLES**

**Submitted in partial fulfillment of the requirements for the award of Master of
Science degree in CHEMISTRY**

By

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(DEEMED TO BE UNIVERSITY)

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This is to certify that this Project Report is the bonafide work of **KM LINGESAN (Reg.No.40910004)** who carried out the project entitled "**SYNTHESIS AND CHARACTERIZATION OF MANGANESE OXIDE NANOPARTICLES**" under my supervision from **NOVEMBER 2021 to MARCH 2022**

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DECLARATION

I KM LINGESAN (Reg.No.40910004) Student of SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY hereby declare that the project report entitle "**SYNTHESIS AND CHARECTERIATION OF MANGANESE OXIDE NANAPARTICLES**" done under the guidance of **Dr.ELANTHAMIZHAN** is submitted in partial fulfillment of the requirements for the award of Master of Science degree in **CHEMISTRY**

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PLACE: Chennai

K.M. Lingesan

SIGNATURE OF THE CANDIDATE

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(KM.LINGESAN)

ABSTRACT

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Manganese oxide nanomaterials have paying attention massive interest as anode in lithium-ion batteries for their low cost and environmental benignity. In this study, the roll of metal precursor in the synthesis of manganese oxide nanoparticles has been studied. The manganese oxide nanoparticles were prepared by using manganese salts of two different anions which are manganous sulphate monohydrate and manganese Oxalate. The obtained samples were characterized by X-ray diffraction (XRD), Transmission Electron Microscope (TEM) and Fourier transform infrared (FT-IR) Spectroscopy. The XRD exhibits that the prepared nanoparticles are crystalline in nature with tetragonal structure. The TEM exhibits the prepared manganese oxide were nano in scale between the ranges of 25 to 60 nm. The FT-IR spectrum conformed the presence of manganese oxide

The process for the green synthesis of manganese oxide nanoparticles (Mn_2O_3 NPs) via hydrothermal synthesis is given in this paper. The production of Mn_2O_3 NPs was indicated by Fourier-transform infrared spectra. The synthesised Mn_2O_3 NPs' ultraviolet-visible absorption spectra showed absorption peaks around 410 nm, which were attributed to the band gap of the Mn_2O_3 NPs. X-ray diffraction analysis was used to identify the crystal phase of the Mn_2O_3 NPs, and the development of crystalline Mn_2O_3 NPs was confirmed. The average size of Mn_2O_3 NPs was also about 80 nm, according to the X-ray diffraction pattern. The synthesised Mn_2O_3 NPs also have a spherical form, according to scanning electron microscope investigation. Photocatalytic activity of Mn_2O_3 NPs in dye degradation the zone of visible light Acid Orange was used as an organic contaminant to test the photocatalytic activity of Mn_2O_3 NPs for dye degradation.

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LIST OF ABBREVIATIONS

- SEM –SCANNING ELECTRON MICROSCOPE
- XRD –X-RAY DIFFRACTION
- EDX – ENERGY DISPERSIVE X-RAY
- TiSi_2 - TITANIUM DISILICIDE
- CVD – CHEMICAL VAPOUR DEPOSITION
- PVD – PHYSICAL VAPOUR DEPOSITION
- AFM –ATOMIC FORCE MICROSCOPE

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CHAPTER 1

INTRODUCTION

Nanomaterials, defined as materials with a length scale of less than 100 nanometers, are gaining popularity due to their fundamental scientific importance as well as the possible applications derived from their interesting electrical, magnetic, and catalytic capabilities [1]. Nanomaterials exhibit better electrochemical activity than bulk active electrode materials, such as higher capacities, larger surface areas, and lower current densities. As a result, nanomaterials have a wide range of applications in electrochemistry. Manganese oxide composites, such as MnO, MnO₂, and Mn₃O₄, have been employed in wastewater treatment, catalysis, sensors, supercapacitors, and alkaline and rechargeable batteries [2–6]. MnO and MnO₂ nanoparticles, in particular, have piqued interest as anode materials in lithium-ion batteries (LIBs) due to their high theoretical capacity, low cost, and environmental friendliness unique characteristics[7–9].

Nanotechnology has gained a lot of attention recently as a result of recent advancements in the scientific and engineering sectors. Nanotechnology can be considered a technology of [10]. At the nanoscale, nanotechnology has created a variety of materials. Rapid advances in nanotechnology have permitted the fabrication of artificial nanoparticles of various types, diameters, and morphologies in recent years [11]. Nanoparticles are a broad category of materials that includes particulate materials with at least one dimension of less than 100 nanometers [12]. They can keep physico-chemical features like large surface area, conductivity, homogeneity, structural stability, or specific optical qualities, which are important in materials science and other domains [13]. Catalysis, pharmaceuticals, and other applications have benefited greatly from these nanoparticles. biomedicine, biosensor cancer treatment, healthcare, and drug delivery [14], optics, food technology, cosmetics, water treatment, electrochemistry, energy, biomedicine, biosensor cancer therapy, healthcare, and drug delivery In comparison to their bulk counterparts, nanoparticle characteristics in the therapeutic arena have improved significantly. Metal and metal oxide nanoparticles in this nanomaterial exhibit

distinct physical and chemical properties. Metal oxide nanomaterials, in particular, are extremely stimulating in the field of semiconducting constituents and promote an ideal prospect for the advancement of biological activities [15-17]. Trimanganese tetraoxide nanoparticles (Mn_3O_4) is one of the important nanoparticles in these nanomaterials, consisting of diverse crystal structures created from octahedral MnO_6 were manganese dioxide (MnO_2) and dimanganese trioxide (Mn_2O_3) [18-20]. Precipitation, hydrothermal, microemulsion, sol-gel, and sonochemical techniques are some of the methods used to create metal/metal oxide NPs, and they all work well [21-23] Products for varied uses. Manganese oxides, a mixed oxide material, are suitable for a wide range of applications such as catalysis, electrochemistry, and medicine due to their low cost, environmentally benign nature, manganese's presence in many forms, and natural abundance [24-25].

Manganese dioxide, dimanganese trioxide, and trimanganese tetraoxide are formed as a result of the various manganese oxidation states. Mn_3O_4 nanoparticles have gotten a lot of interest because of their unique structural and electronic properties, which include ion exchange, catalysis, molecule adsorption, magnetic, and electrochemical capabilities. Manganese, a critical component of metabolism, has been efficiently regulated by biological systems, and Mn_3O_4 NPs have better biological characteristics and are less hazardous [26-28]. As a result of their synergistic performance, Mn_3O_4 NPs have been adequately explored as potential antibacterial systems. The ions " Mn^{2+} " produce innocuous free radicals, which play an important role in clinical illnesses such as Alzheimer's, heart, diabetes, and a variety of diseases, including cancer. Furthermore, nanomedicine is seen to be a promising research path in the treatment of cancers. Khan and colleagues (2016). The current situation in cancer treatment is complicated by the partial availability of certain anticancer drugs or nanomedicines, as well as a scarcity of competent delivery vehicles and the fact that the bulk of currently available anticancer agents have low solubility in aqueous solutions. As a result, significant efforts have been made to develop innovative anticancer medicines with improved water solubility and efficacy in cancer treatment. Metal oxide nanoparticles, which have synergistic and excellent physicochemical features, have attracted a lot of attention in cancer therapy [29-30]. In this paper, we use a precipitation approach to demonstrate a precipitation method for the synthesis of

Mn₃O₄ nanoparticles utilising manganese nitrate as a precursor material (Scheme 1). Several microscopic, spectroscopic, and thermal properties of as-synthesised Mn₃O₄ nanoparticles SEM, EDX, UV-Vis, XRD, and FT-IR are examples of methods. *S. aureus*, *B. subtilis*, *E. coli*, *P. aeruginosa*, *C. albicans*, and *A. flavus* were used to test the biological characteristics of the as-prepared Mn₃O₄ NPs against diverse fungal and bacterial strains. Mn₃O₄ NPs suppressed the development of *S. aureus* with a MIC of 40 lg/ml and *C. albicans* with a MIC of 15 lg/ml.

CHAPTER 2

LITERATURE SURVEY

In this paper, Marugán et al [31] do a full thermodynamic investigation of the first phase of the $\text{Mn}_2\text{O}_3/\text{MnO}$ thermochemical cycle for solar hydrogen production. The heat reduction of Mn_2O_3 is accomplished using a two-step reaction sequence. Finally, this research shows that the first step of the manganese oxide thermochemical cycle for hydrogen synthesis may be carried out at temperatures compatible with solar energy concentration devices with 100% conversion. The necessary temperature range for the manganese oxide cycle is lower than that frequently reported in the literature, based on theoretical and thermodynamic investigations..

The goal of Aparna Iyer and colleagues [32] was to split water into hydrogen and oxygen. The $\text{Mn}_4\text{O}_4\text{Ca}$ manganese cluster, on the other hand, catalyses water oxidation in plants. Furthermore, manganese minerals are found all over the world, with layered and tunnel structures. As water oxidation catalysts, mixed valent porous amorphous manganese oxides (AMO), cryptomelane type tunnel manganese oxides (OMS-2) and layered birnessite (OL-1) were utilised in this investigation. In water oxidation tests using Ce^{4+} , AMO (290 mmol $\text{O}_2/\text{mol Mn}$) had much larger turnovers than tunnel structure OMS-2 (110 mmol $\text{O}_2/\text{mol Mn}$) and layered structure OL-1 (27 mmol $\text{O}_2/\text{mol Mn}$). Under photochemical circumstances, oxygen evolution was also confirmed using $\text{Ru}(\text{bpy})_3^{2+}$ as a photosensitizer and persulfate as a sacrificial agent.

S. Ardizzone et al. [33] used a two-step technique to make Mn_2O_3 powders by calcining $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ at 600°C . The charge and capacitance vs. pH graphs were used to characterise the oxide's electrification behaviour at the solution boundary. The occurrence of a solution-supported disproportionation reaction of Mn is invoked to explain the unique pattern observed at high KNO_3 concentrations (III).

Chung-Hao Kuo and colleagues [34] Manganese oxides (MnO_x) have been identified for their enormous promise as highly efficient and resilient materials for water oxidation reaction, inspired by the natural oxygen evolution reaction of Photosystem II (WORs). In comparison to RuO_2 and IrO_2 , the majority of

heterogeneous, manufactured MnOx catalysts still have lower WOR activity. They describe a soft-templated method for developing a single-step and scalable synthesis method for mesoporous MnOx materials.

3 The pseudocapacitive performance of nitrogen-doped and undoped reduced graphene oxide/tetragonal hausmannite nanohybrids (N-rGO/Mn₃O₄ and rGO/Mn₃O₄) produced using a one-pot hydrothermal technique was investigated by Katlego Makgopa et al [35]. For symmetric pseudocapacitors, the nanohybrid electrode materials outperformed their separate precursors (reduced graphene oxide (rGO), nitrogen-doped reduced graphene oxide (NrGO), and tetragonal hausmannite (Mn₃O₄)) in terms of electrochemical performance.

Ren Z and colleagues [36], Manganese oxide nanoparticles were made using a simple hydrothermal process and used to remove thallium in this work (1). After heat-drying, the adsorbent was composed of many needle-like nanorods with an average volume diameter of 230 nm. The adsorbent's crystal shape was discovered to be -MnO₂. The adsorbent adsorption rate was substantially faster than most previously reported adsorbents, with over 66.4 percent of equilibrium adsorption capacity achieved in the first 10 minutes. The adsorption process was shown to be substantially impacted by solution pH, with adsorption capacities greater than 100 mg/g achieved in a pH range of 6.0–10.0..

One of the greatest obstacles in meeting future sustainable fuel demand, according to Hendrik Antoni et al [37], is developing nonprecious catalysts for water splitting into hydrogen and oxygen. Thin layers of manganese oxide nanosheets supported on nitrogen-functionalized carbon nanotubes (NCNTs) were formed by treating NCNTs dispersed in aqueous solutions of KMnO₄ or CsMnO₄ under reflux or hydrothermal (HT) conditions, and then used as electrocatalysts for the oxygen evolution reaction (OER) in alkaline media and a significantly more efficient purification sequence involving partial oxidation of the as-receive.

The Properties of porous manganese oxide adsorbents for adsorptive removal of tert-butylmercaptan (TBM) from CH₄ fuel gas were examined at ambient temperature and atmospheric pressure in this study by Ho, P.H et al [38]. The adsorbents were made by oxidising Mn²⁺ with KMnO₄ and then reducing KMnO₄ using fumaric acid as the reducing agent in the sol-gel process. The structure and desulfurization properties of the resultant adsorbents were investigated in relation to the synthesis process, precursor, temperature, and time..

At ambient temperature and atmospheric pressure, Phuoc Hoang Ho et al [39] investigated the properties and characteristics of cryptomelane manganese oxide octahedral molecular sieve (OMS-2)-based adsorbents for the adsorptive desulfurization of dimethyl sulphide (DMS), tertbutylmercaptan (TBM), and tetrahydrothiophene (THT). Adsorption selectivity for TBM in a ternary mixture of DMS, TBM, and THT in a methane fuel stream was above 90% for OMS-2 adsorbents, which is unprecedented for zeolite-, metal oxide-, and activated carbon-based adsorbents.

By employing dilute aqueous solutions of manganese sulphate and ammonia in the presence of sodium lauryl sulphate — a surface active agent — Navin Kumar Chandra et al [39] generated nano-size crystals of Mn₂O₃. The phases were identified and the size of the nano-size crystals was calculated using the Scherrer method employing X-ray diffraction spectrometry. The crystals' shape was investigated using TEM/HRTEM, and Edax was utilised to assess their elemental contents. The conversion of MnOOH to nano-sized Mn₂O₃ occurs when the obtained nano-size material is sonicated in acetone.

Manganese oxide electrodes with promising pseudo-capacitive behaviour were made by sol-gel technique using manganese acetate as the precursor in this study by Lin, C. K et al [40]. The effects of postheating on material properties and electrochemical properties were studied.

CHAPTER-3

3.1 Aim of the investigation

Manganese oxides are a mixed oxide substance that are suitable for a wide range of applications including catalysis, electrochemistry, and medicine due to their low cost, environmentally benign nature, manganese in multiple forms, and natural abundance. Keeping these considerations in mind, the goal of this inquiry is to

- Manganese-oxide nanoparticles Synthesis
- Characterization of the synthesised material:
 - ✚ X-ray diffraction (XRD)
 - ✚ Fourier transform infrared spectroscopy (FTIR)
 - ✚ Scanning electron microscopy (SEM)
 - ✚ Energy dispersive X-ray spectroscopy (EDAX)
 - ✚ Antibacterial research

CHAPTER - 4

MATERIALS AND METHODS

4.1 SYNTHESIS OF Mn_3O_4 NANOSTRUCTURE

1.0 g Polyvinyl alcohol (PVA) and 2 mmol manganese acetate were dissolved in 100 mL distilled water with magnetic stirring at 90 °C for 30 minutes to

produce Mn_3O_4 NPs. The solution combination was then transferred to a Teflon-coated autoclave. The autoclave was sealed and heated to 120°C for 12 hours before being cooled to ambient temperature. After cooling, the precipitate was filtered and washed numerous times with distilled water and ethanol (to remove the soluble nitrates). Finally, dry for 12 hours in an oven at 80°C . Finally, the dried precipitates were annealed at 1000°C for 3 hours under air to generate Mn_2O_3 NPs, which were stored until needed. The blueprint ;Figure 1 depicts the creation of Mn_2O_3 NPs by the hydrothermal synthesis technique.

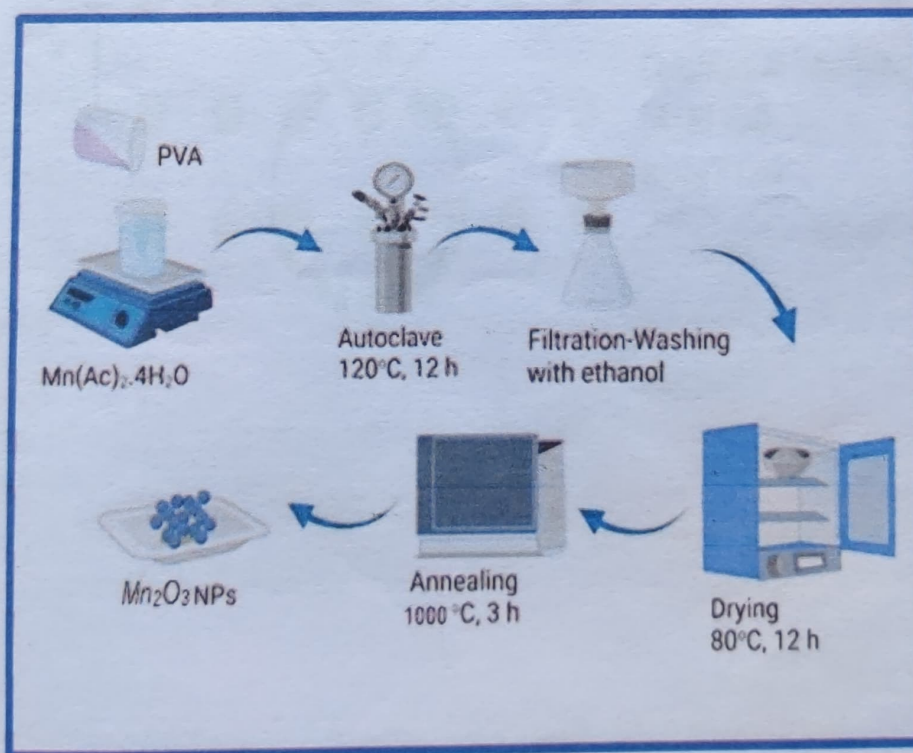
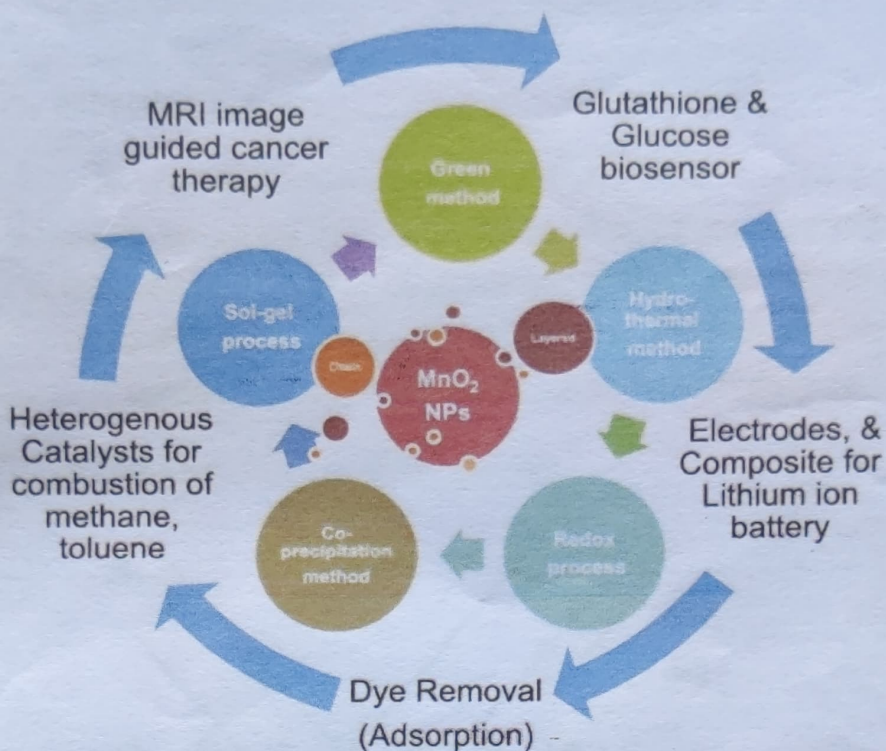
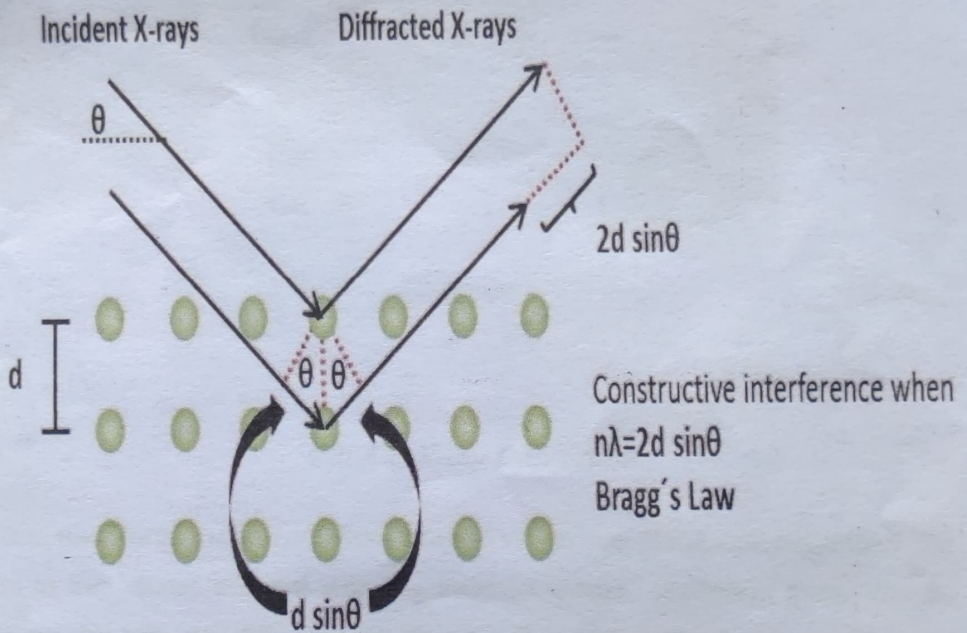


Fig. 1 Schematic representation for synthesis of Mn_2O_3 NPs

4.2 X-ray diffraction

This is used to determine the material's crystallinity and provides information on unit cell dimensions. Wilhelm Rontgen created the company in 1895, and Max von Laue developed it in 1912. It is generally utilised in research domains, and it may be obtained by orienting it at various temperatures, but it does not reveal its chemical makeup.



It's a highly effective non-destructive device.



Fig 4.2 X-ray Diffraction

X-rays are electromagnetic radiation waves. When we place our materials, the electrons in the atoms interact with the incident x-rays, scattering them. Elastic scattering is the term for this. The electrons collide with cathode rays, which are heavy target materials. The scatterer array produces a regular array of spherical waves. As a result, it has a condition that successive grating lines match the wavelength of light. The destructive interference cancels each other in most directions, but Bragg's law causes them to add constructively in one direction. The wavelength of light should be in the Armstrong range, and the most critical requirement is that the spacing between atoms be equal to the x-ray wavelength. It consists of a glass tube that is to be inserted into Check for extreme temperatures. The cathode, which carries filament, and the anode, which is used for x-ray targeting, are two separate pieces. $2d\sin\theta = n\lambda$, where d is the spacing between diffracting planes, θ is the incident angle, n is an integer, and λ is the wavelength, according to Bragg's law. Electromagnetic waves spotting on a regular array of scatterers can be seen in continuous x-ray diffraction patterns (55).

4.2.1 APPLICATIONS

It is a non-destructive approach for locating crystalline or amorphous materials, whether they be minerals or inorganic compounds. For analysis, only a small sample is required. To determine structural features such as lattice parameters, phase composition, strain, and epitaxy, as well as to detect the orientation. To determine the atomic arrangement and thickness of thin films and multilayers..

4.3 Fourier transform infrared spectroscopy:

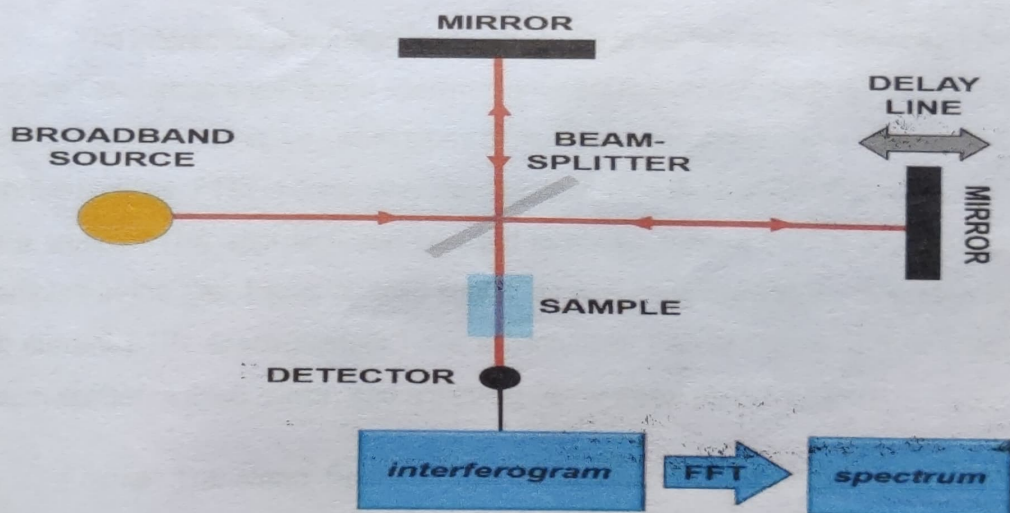




Fig 4.3 *Fourier transform infrared spectroscopy*

The interaction of infrared light with matter is studied using Fourier transform infrared spectroscopy. Infrared spectrum analysis can provide both quantitative and qualitative information, i.e., what types of molecules are present in a sample and at what quantities. FTIR is commonly used to determine the functional groups present in a sample. This approach can be used to obtain spectra from a wide range of samples in the gas, liquid, or solid states, as well as to investigate their structure. All current FTIR spectrometers have a Michelson interferometer at their core. A beam splitter, a fixed mirror, and a moving mirror make up the system.

Fourier Transform Spectroscopy is a basic mathematical approach for separating the frequency components of a complicated wave. Because the sources are faint and the detectors are insensitive, typical IR spectrometers are ineffective in the far IR region. This energy-limited region has become more accessible thanks to FTIR. It has also improved the utility of mid-infrared ($4000\text{--}400\text{ cm}^{-1}$). A time domain plot is turned into a frequency domain spectrum in the Fourier Transform Spectrometer. The interference pattern is created using a two-beam interferometer, and the spectrum is created by fourier transforming the output as the path difference between the two beams is changed. Depending on the region of interest, solid samples are distributed in KBr or polyethylene pellets.

FTIR analysis determines the chemical composition and structure of a sample by measuring the absorbance of infrared light at various wavelengths. However, using transmittance rather than absorbance is more convenient, therefore the FTIR graph is a transmittance (%) vs. wavenumber plot.

4.4 SCANNING ELECTRON MICROSCOPE

An electron microscope is improved with a scanning electron microscope (SEM). It creates an image by scanning a concentrated beam of electrons across a surface. It was founded by Stintzing in 1927 and has since undergone numerous upgrades. The interaction of electrons with the atoms of an object is the principle behind it. The image is created by combining beams and detecting signals. A single electron gun was used to construct it, and no auxiliary lenses were used (56)

Scanning Electron Microscope (SEM)

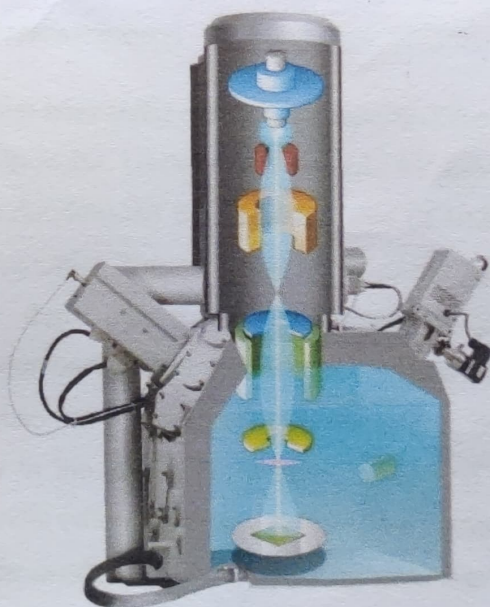
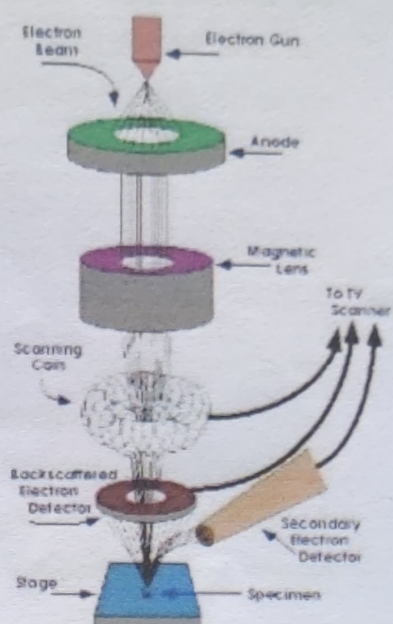
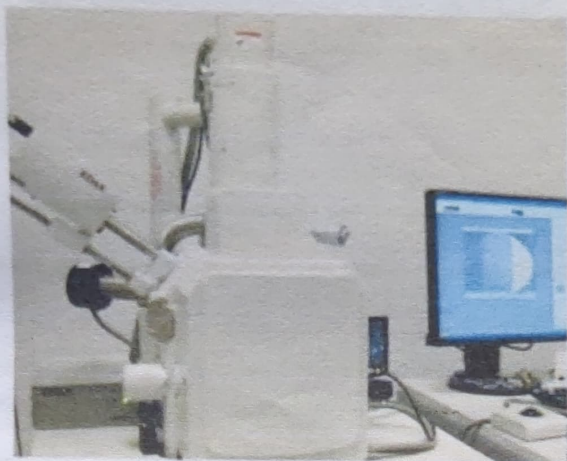


Fig 4.4 Scanning Electron Microscope

When primary electrons strike samples, they produce secondary electrons, which hold information about the object and produce three-dimensional pictures of the samples. The SEM is made up of an electron gun that produces a high-energy electron beam, a voltage that controls the emission current, and a tungsten tip that is condensed using a magnetic condensing lens. The scintillator is there to collect secondary electrons, which are converted into an electrical signal, and the scanning

coil is situated between the condense lens and the sample. A video amplifier can be used to feed this electrical signal into CRO. It may be viewed directly on screen to evaluate enormous thicknesses and be used in a wide depth of focus. When the rays strike the sample, three sorts of beams are produced outside of the sample: x-ray, back reflection, and secondary electrons.

4.4.1 APPLICATIONS

Examines the structure of extremely big 3D images. The samples' results are resolvable down to the nanoscale scale. The surface is mostly used for topography, or how it appears in terms of texture and distinguishable features. The intricacies of how the atoms are organised can then be obtained through elemental analysis. It allows you to examine a variety of items at high and low magnification without concentrating on depth..

4.5 Photo-catalytic measurement

Using Methylene blue (MB) dye, we investigated the photo-catalytic activity of the produced nanopowder. Under direct sunlight for 120 minutes, 40 mg of the sample was used to decompose 80 ml of MB dye (2.10^{-5} mol/L), with samples collected every 15 minutes. The samples were mixed for 30 minutes at room temperature in the dark before the experiment began. Methylene blue's characteristic absorption band in the visible spectrum is a single peak at 665 nm. In compared to the reaction in the dark with photocatalyst, dye absorption intensity dropped dramatically in the presence of light, confirming the sample's effective degrading capacity. With different photocatalysts, the fluctuation of the ratio of the concentration (C) after irradiation to the initial concentration (C_0) as a function of irradiation time is also determined and given.

The efficiency of MB dye degradation is proportional to its absorbance (concentration), which can be determined using the following formula:

$$Efficiency = \frac{(C_0 - C)}{C_0} \times 100\% = \frac{(A_0 - A)}{A_0} \times 100\% \quad (1)$$

Where C_0 , C , A_0 , A are the concentration and absorbance of MB dye before and after irradiation with visible light..

CHAPTER-5

RESULT AND DISCUSSION

5.1 Structural analysis

Mn₃O₄ (JCPDS Card No.00-001-1127) with tetragonal crystal structure (space group 141/amd) was identified as the typical XRD patterns shown in Fig. 5.1. The Debye-Scherrer approximation was used to calculate the crystallite size of Mn₃O₄ NPs from diffraction peaks [23]. The average of the crystallite sizes measured from the peaks (112, 103, 211, 004, 220, and 224) yielded a crystallite size of 21.2 nm. In addition, as seen in the picture, the diffraction patterns produced are crisp, narrow, and of high intensity, all of which support the crystalline character of the generated Mn₃O₄.

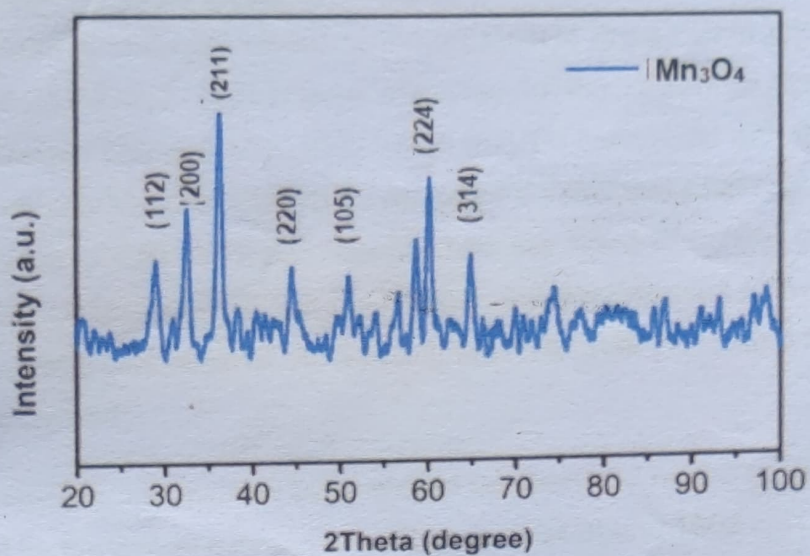


Figure 5.1 Powder XRD patterns of Mn_3O_4 NPs.

5.2 FTIR Spectroscopy:

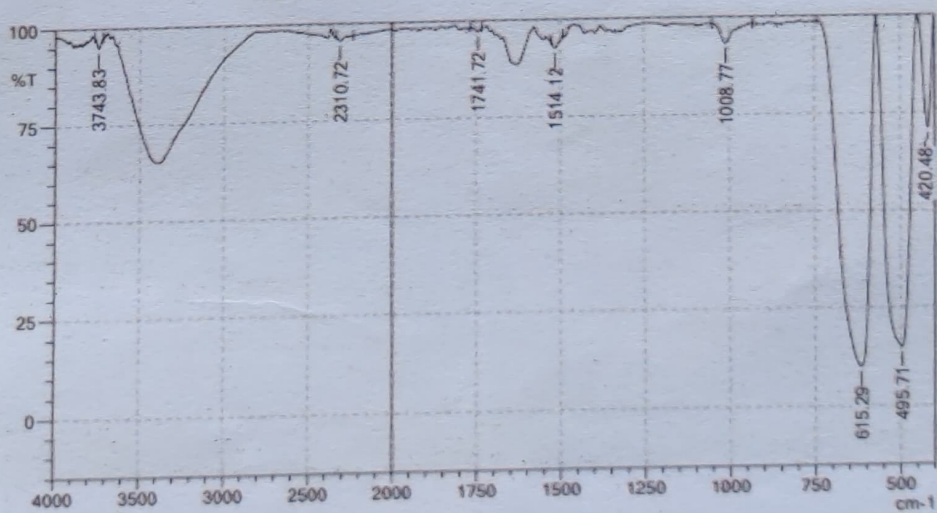


Fig 5.2 FT-IR Spectrum of Mn_2O_3 nanoparticles

Figure 5.2 shows The as-prepared Mn_3O_4 products' Fourier Transform Infrared (FTIR) spectrum is strikingly similar to that of Mn_3O_4 produced in prior research [32,12]. Two absorption peaks were identified in the region of the observed spectrum between 650 and 500 cm^{-1} , which may be connected with the coupling modes between the Mn-O stretching modes of tetrahedral and octahedral sites. The absorption peak at 430 cm^{-1} was identified as the band-stretching mode of the octahedral sites in the range of 500 to 400 cm^{-1} ; the displacement of the Mn^{2+} ions in tetrahedral sites was minimal. As a result, the FTIR spectra confirm that Mn_3O_4 products are formed..

Furthermore, the hydroxy (OH) should be absorbed by the samples or KBr, as indicated by the typical thin and broad bands at 3420 and 1600 cm^{-1} . These FT-IR analysis results are consistent with data obtained from other characterisation results..

5.3 Scanning Electron Microscope (SEM)

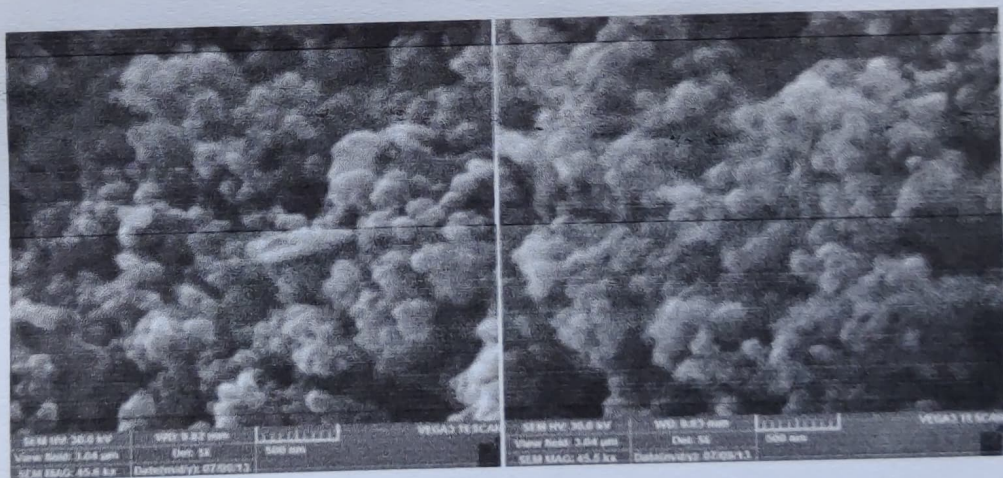


Fig. 5.3. SEM images of as prepared Mn_3O_4 nanoparticles

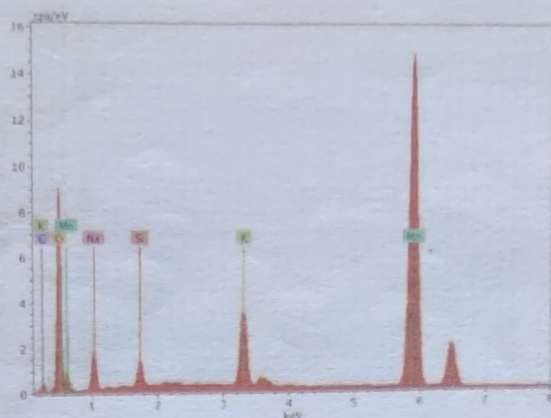


Fig 5.4. EDAX Spectrum of Mn_3O_4 nanoparticles

Scanning Electron Microscope was used to study the surface morphology of nanoparticles (SEM). Figure 5.3 shows the SEM pictures of Mn₃O₄ nanoparticles. The impact of surface morphology on the performance of nanostructure materials has been thoroughly established. The micrographs show grain distributions that are uniform. The particles are virtually spherical in shape and consistent in size, with an average size of about 50 nanometers. The energy dispersive X-ray analysis (EDAX) spectra of Mn₃O₄ nanoparticles is shown in Figure 5.4. The existence of manganese (Mn) and oxygen (O) is confirmed by the EDAX spectrum, and certain contaminants in the produced Mn₃O₄ nanoparticles were removed during the 1000°C annealing procedure. EDAX investigated the chemical composition and product purity of Mn₃O₄ nanoparticles. The produced sample's oxygen storage capability was confirmed by an oxygen-related signal in the EDAX spectrum.

5.4 Optical studies - UV-Visible Spectroscopy

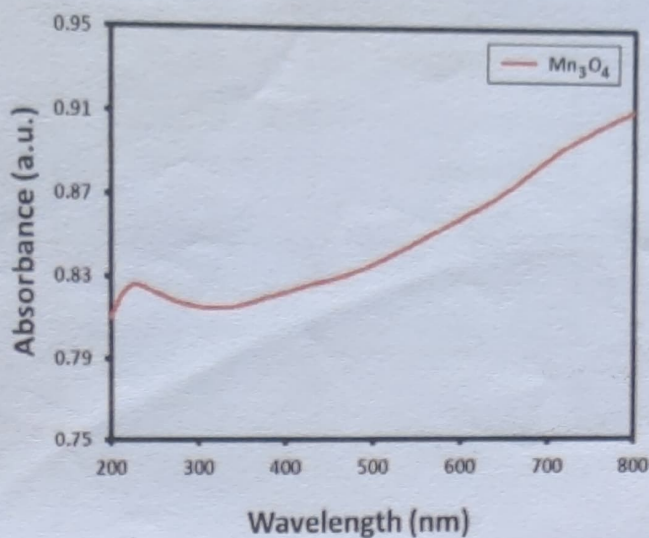


Fig 5.5 UV-Visible Spectra of Manganese oxide nanoparticles

The UV-Vis spectra of as-prepared Mn_3O_4 nanoparticles using manganese nitrate were first studied. Figure 15.5. shows the UV-Vis spectra of as-prepared Mn_3O_4 nanoparticles using manganese nitrate. The characteristic peak at 220 nm in the UV-Vis spectra of Mn_3O_4 nanoparticles clearly indicates Mn_3O_4 nanoparticle production. [14-15].

Antimicrobial Studies

Zone of Inhibition results of the susceptibility of different microbial stains tested against standard antibiotics

Zone of Inhibition (nm)	Mn_3O_4 NPs	Anti-microbial Gentamycin 50µg/ml	Anti-microbial Fluconazole 50µg/ml
E. coli	10mm	21mm	-

S. aureus	19mm	22mm	-
P. aeruginosa	12mm	21mm	-
B. subtilis	18mm	21mm	-
C. albicans	18mm	-	20mm

Our antimicrobial results matched those of Azhir et al., 2015, who found *E. coli* to be sensitive and *S. aureus* to be susceptible to Mn_3O_4 NPs, whereas Chowdhury et al., 2009 found *E. coli* to be susceptible. Packirisamy et al. found that *B. subtilis* is susceptible, as well as *E. coli* and *P. aeruginosa*, as antimicrobial activity increased with increasing the concentration (Packirisamy et al., 2019). *S. aureus* growth was inhibited by Mn_3O_4 NPs at a minimum inhibitory concentration (MIC) of 40 $\mu\text{g/ml}$, and *C. albicans* growth was inhibited at a MIC of 15 $\mu\text{g/ml}$. The findings suggest that as-made Mn_3O_4 NPs generated from manganese nitrate via a precipitation method are a promising candidate for pharmaceutical and food packaging applications.

CHAPTER-6

SUMMARY AND CONCLUSION

The precipitation method was used to successfully produce Mn₃O₄ NPs. The Mn₃O₄ nanoparticles are tetragonal in shape and uniformly distributed and well diffused as they are manufactured. UV-Vis, FT-IR, XRD, SEM, and EDX analyses were used to characterise Mn₃O₄ nanoparticles. The findings revealed the emergence of a tetragonal structure with an average size of 36 nanometers. The biological activity of the produced Mn₃O₄ nanoparticles was tested. Furthermore, Mn₃O₄ nanoparticles' antibacterial activities, zone of inhibition, and minimum inhibitory concentration all showed an inhibitory impact against all microorganisms tested. The Mn₃O₄ nanoparticles, however, were found to be selective against gramme positive bacterial strains *S. aureus* and *B. subtilis*. *S. aureus* and *B. subtilis* are both Gram positive bacteria. *B. subtilis* was susceptible to Mn₃O₄ nanoparticles, with inhibitory zones of 19 and 18 mm, respectively. The figure 6 (a) SEM picture of Mn₃O₄ nanoparticles as synthesised and (b) energy dispersive X-ray spectroscopy of Mn₃O₄ nanoparticles. Table 2 shows the Zone of Inhibition data for various bacteria strains tested against conventional antibiotics. The Inhibition Zone (mm) NPs of Mn₂O₃ Gentamycin (30 mg/ml) is an antibiotic. Fluconazole (50 mg/ml) antibiotic coliform bacteria *S. aureus*, 10 mm 22 mm *P. aeruginosa*, 19 mm, 21 mm *B. subtilis* 12 mm 22 mm *C. albicans*, 18 mm 22 mm *A. flavus* 14 mm – 20 mm 18 mm – 20 mm.

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