Solid State Synthesis and its Characterisation of Nickel Doped Mn203 for the Photocatalytic Application

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**ABSTRACT :**The area of photocatalysis has attracted a lot of attention lately because of its potential to solve environmental problems and advance sustainable energy alternatives. Transition metal oxides, in particular, have demonstrated potential as photocatalysts for harnessing solar energy for a range of uses, including hydrogen production and the degradation of pollutants. Because of its distinct electrical structure and appropriate band gap for visible light absorption, manganese trioxide (Mn2O3) has become a notable choice among these oxides.A viable technique for both environmental cleanup and energy generation is photocatalysis. Photocatalysts have demonstrated interest in transition metal oxides, especially Mn2O3. They can have more photocatalytic activity if they are doped with metals like nickel.To evaluate solid state synthesis and its characterisation of nickel doped Mn2O3 for the photocatalytic application.In this work, nickel-doped Mn2O3 nanoparticles were prepared using a solid-state synthesis technique. The produced materials were characterized using a variety of analytical techniques, such as X-ray diffraction (XRD), scanning electron microscopy (SEM).The presence of nickel was found in the doped samples, and the XRD investigation verified the creation of crystalline Mn2O3 . The nanoparticles' shape and particle size distribution were visible in SEM and TEM pictures. The electrical structure and chemical states of the doped Mn2O3 were revealed by XPS analysis.Nickel-doped Mn2O3 nanoparticles were effectively synthesized using the solid-state synthesis technique. The characterization results show that the integration of nickel leads to the creation of an appropriate crystalline structure. These results provide the foundation for additional research into the photocatalytic capabilities of these substances, which may result in uses for solar energy conversion and environmental cleaning.

**keywords:** Ni doped Mn2O3 nano material synthesis, Heating and reaction, Impurity removal, Precursor dissolution, Autoclave preparation, Precursor dissolution

# INTRODUCTION

In recent years, photocatalytic materials have drawn a lot of attention due to their prospective uses in renewable energy production and environmental remediation). Because of their ability to absorb solar energy and speed up a variety of chemical reactions when exposed to light, metal oxides, such manganese oxide (Mn2O3), have shown promise among these materials.Photocatalytic materials have attracted a lot of attention lately due to their potential uses in renewable energy production and environmental remediation. A well-known metal oxide photocatalyst, manganese oxide (Mn2O3) has applications in energy conversion, air purification, and water filtration. Its properties must be improved, nevertheless, in order to fully utilize its photocatalytic capabilities[(Kasabwala et al., 2021; Rajeshkumar & Lakshmi, 2021; Varghese et al., 2023)](https://paperpile.com/c/kLJ271/YAVFD+9xbsf+nfh77). The idea of dopants enters the picture here. The electronic band structure of Mn2O3 can be altered by nickel doping, which may have an impact on its electronic characteristics including density of states and energy band position [(Ramakrishnan et al., 2023; Shenoy & Maiti, 2023; J. S. Sindhu et al., 2023)](https://paperpile.com/c/kLJ271/nkZqA+f7jR2+qkyrM). This alteration might enhance charge carrier separation and mobility, which would increase photocatalytic activity[(Dharman et al., 2023; S. Sindhu et al., 2023; Sreenivasagan et al., 2023)](https://paperpile.com/c/kLJ271/8qYUp+Mcki9+UF345). By changing Mn2O3's absorption and emission characteristics, nickel doping can change its optical characteristics[(Ajay et al., 2023; Chokkattu et al., 2023; Padarthi et al., 2023)](https://paperpile.com/c/kLJ271/XCg1E+OGH6T+8cdku). This could increase the material's sensitivity to visible light wavelengths, increasing its efficiency in visible light-driven photocatalytic activities.In some processes, such as the breakdown of organic pollutants or the splitting of water molecules to produce hydrogen, nickel dopants can increase the catalytic activity of Mn2O3. Controllable Ni2+ Incorporation for High-Performance Zinc Ion Battery: Preventing Manganese Dissolution in Mn2O3 Cathode.Comparable volumetric and gravimetric energy densities of long-lasting ZIBs are also present in the flexible soft-packaged batteries, along with good electrochemical performance. Ni-doped Mn2O3 microspheres are an extremely effective electrocatalyst for Zn-air batteries and oxygen reduction reactions. The resulting Ni-doped Mn2O3 has a high limiting current density and a half-wave potential of 0.801 V, demonstrating outstanding ORR performance in alkaline conditions. “ Ni2P/Mn2O3 nanostructures in one dimension with increased activity in the oxygen evolution reaction. Because the Ni2P/Mn2O3 nanofibers only need minor overpotentials of 280 mV to create the 10 mA cm2 current density in 1 M KOH solution, they performed better in terms of OER than several previously reported nickel-based transition metal phosphides catalysts.Using pulsed potential, a novel Ni-doped ZnMn2O4/Mn2O3 nanocomposite was created as an excellent cathode material for zinc ion batteries. Ni2+ presence enhances the Ni-doped ZnMn2O4/Mn2O3 nanocomposite's specific surface area and conductivity, which in turn enhances the kinetics of electrochemical reactions”. “An effective catalyst for the low-temperature SCR of NO with NH3 is nickel-doped Mn/TiO2: Catalytic assessment and characterization.. The XRD patterns showed that Mn and Ni are widely dispersed throughout the titania, supporting research on the microwave-synthesised Mn2O3-SiO2 nanocomposites' optical, magnetic, and photocatalytic properties. The dye that degraded the fastest after 75 minutes of UV exposure was methyl-violet (94.45%). Ni-doped barium hexaferrite is synthesized via a microemulsion method to improve the photocatalytic degradation of crystal violet dye triggered by visible light . Doping also increased the AC conductivity, which may have been caused by a drop in band gap energy. "Mulberry-like cobalt manganese oxide boosting for visible light driving photocatalytic degradation of pharmaceutical pollutants using in situ N-doped carbon coating." [8]. The results revealed that reduction time, calcination temperature, and environment all had a substantial impact on the morphology, structure, and content of the samples.“Enhanced photocatalytic reduction of CO2 using CdS/Mn2O3 nanocomposite photocatalysts on porous anodic alumina support with solar concentrators”[(“Enhanced Photocatalytic Reduction of CO2 Using CdS/Mn2O3 Nanocomposite Photocatalysts on Porous Anodic Alumina Support with Solar Concentrators,” 2019)](https://paperpile.com/c/kLJ271/rRLR). CdS nanosheets coated with Mn2O3 nanoparticles outperformed CdS alone as a photocatalytic catalyst due to help in anti electron hole recombination.“Europium-doped MnO2 nanostructures for controlling optical properties and visible light photocatalytic activity [(“Europium-Doped MnO2 Nanostructures for Controlling Optical Properties and Visible Light Photocatalytic Activity,” 2022)](https://paperpile.com/c/kLJ271/U2Ca). The Eu doping in the MnO2 grid acted as an electron scavenger, preventing electron-hole pairs from recombination on the surface of MnO2 nanoparticles and so increasing electron transport.“Effects of Co and Ni co-doping on the physicochemical characteristics of cryptomelane and its improved efficacy in photocatalytic phenol degradation demonstrated that while Co-doped cryptomelane has a similar micromorphology and crystal structure to un-doped cryptomelane, it is less crystalline and has a larger specific surface area.A controlled technique for creating nickel-doped Mn2O3 is solid-state synthesis, which uses the direct reactivity of solid precursor molecules. High-temperature annealing comes after combining sources of manganese and nickel. The crystal structure and phase purity of the generated material can be determined using X-ray diffraction (XRD) techniques. Its surface appearance can be seen using SEM, and its optical characteristics, such as band gap energy, can be found using UV-Vis spectroscopy. Diverse technological applications are advanced by the synthesis and characterisation of solid-state materials[(Keerthana & Ramesh, 2021; Murugesan, 2021; Tiwari & Jain, 2021)](https://paperpile.com/c/kLJ271/uPUKk+OEri5+abNGc)[(Keerthana & Ramesh, 2021; Murugesan, 2021; Subramanian et al., 2021; Tiwari & Jain, 2021)](https://paperpile.com/c/kLJ271/uPUKk+OEri5+abNGc+gPbm5). The creation of effective photocatalysts is crucial in the field of photocatalysis, which offers hope for sustainable energy and environmental solutions. As a possible photocatalyst, nickel-doped manganese dioxide (Mn203) is synthesized and characterized in this study. This research intends to shed light on the potential of this unique composite for harvesting solar energy and causing catalytic reactions with broad consequences by examining the delicate interplay between material composition, crystal structure, and photocatalytic activity.Here comes the aim of the study is to evaluate solid state synthesis and its characterisation of nickel doped Mn2O3 for the photocatalytic applications.

# MATERIAL AND METHODS

Ni-doped Mn2O3 nanomaterial is produced hydrothermally using KMnO4, Ni(NO3)2, and urea as precursors. The process is explained in detail here.

## Ni doped Mn2O3 nano material synthesis

**Precursor dissolution:** Start by combining 160 mL of distilled water with 0.2 M of KMnO4, 10 mol% of urea, and 0.2 M of Ni(NO3)2. The mixture is then aggressively agitated at room temperature to create a homogenous solution.

**Autoclave preparation:**Insert a 200 mL autoclave with the homogenous solution inside. A sealed high-pressure container known as an autoclave is used for chemical reactions at high temperatures.

**Heating and reaction:**Place the autoclave in a hot air oven and keep it there for 12 hours with a temperature of 160 °C. The chemical interaction between the precursors is aided by the high temperature and pressure of the environment, creating the required Ni-doped Mn2O3 nanocomposite.

**Cooling:**Allow the autoclave to naturally cool to ambient temperature when the reaction time has passed. This steady cooling reduces the possibility of product cracking and slows down sudden fluctuations in pressure.

**Impurity removal:**Use ethanol and distilled water to wash the synthesized product many times to get rid of contaminants. Washing the nanomaterial helps to purify it and get rid of any leftover reactants or byproducts.

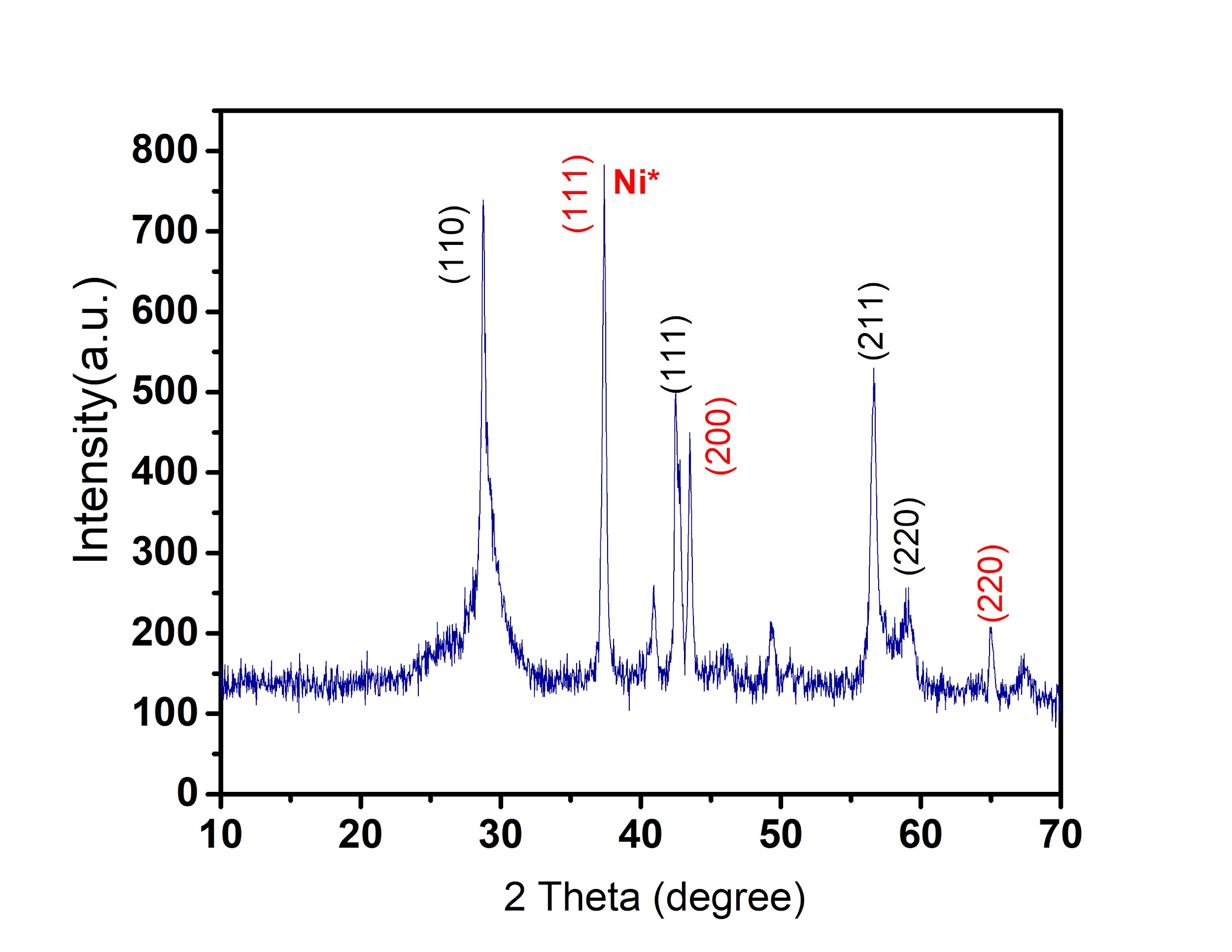
**Product collection:**The pure Ni-doped Mn2O3 nanomaterial should be collected once the contaminants have been adequately removed by washing. The intended nanocomposite that was created throughout the reaction is present in this product.

**Drying:**The collected material should be dried in an oven at 80 °C for 12 hours to get rid of any last-remaining solvent and guarantee the stability of the nanomaterial. This process aids in getting the nanomaterial ready for later characterisation and applications.

In order to create the final Ni-doped Mn2O3 nanomaterial, the procedure entails dissolving the precursors, for a reaction to happen under controlled circumstances, purifying the result, and then drying it. Depending on its unique features, this substance might have a variety of applications in areas like catalysis, energy storage, and sensing.

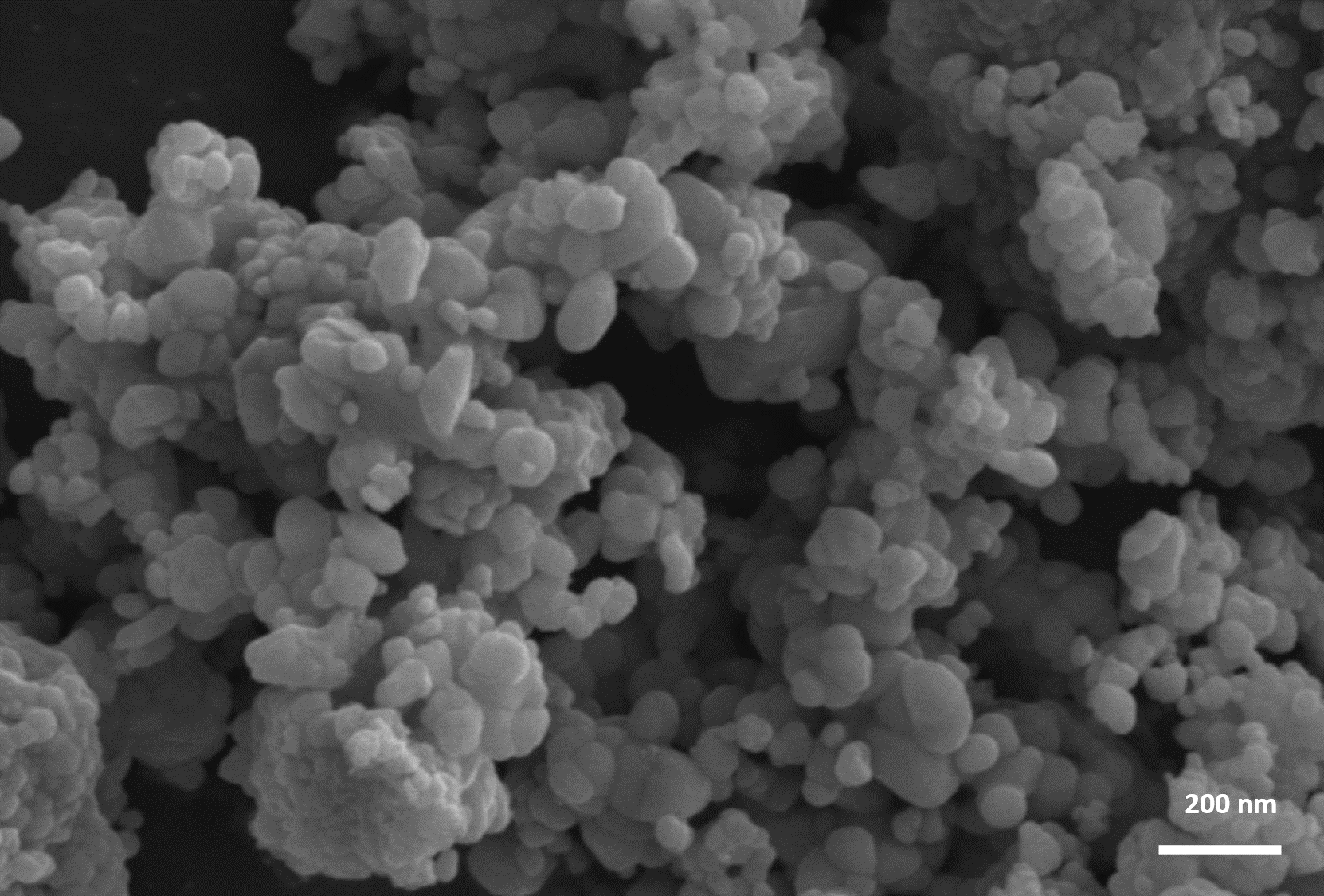
# RESULTS

## XRD ANALYSIS



**Fig 1:** An XRD pattern of the electrochemically grown Ni-Mn2O3 nanoflower after the hydrothermal treatment

## XRD: X-ray diffraction; JCPDS: Joint Committee on Powder Diffraction Standardsof NiMn2O3

Figure 1 shows peaks at 2θ angles (111), (110), (211), (200), and (220), indicating the presence of these crystallographic planes in the NiMn2O3 sample(Nikalje et al., 2024) (Chehelgerdi et al., 2023). The prominence of these peaks indicates the number or prominence of certain crystallographic planes in the material, as shown . A distinct and sharp peak, like (111), at a certain 2θ angle, for example, indicates a well-defined crystallographic plane and a high degree of crystallinity in that direction. Broader or less strong peaks, on the other hand, could indicate decreased crystallinity or the existence of contaminants or flaws in the crystal structure.

**Fig 2:** The FE-SEM images of the NiMn2O3 prepared by hydrothermal treatment.

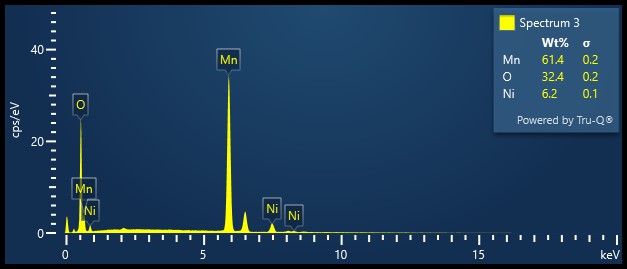
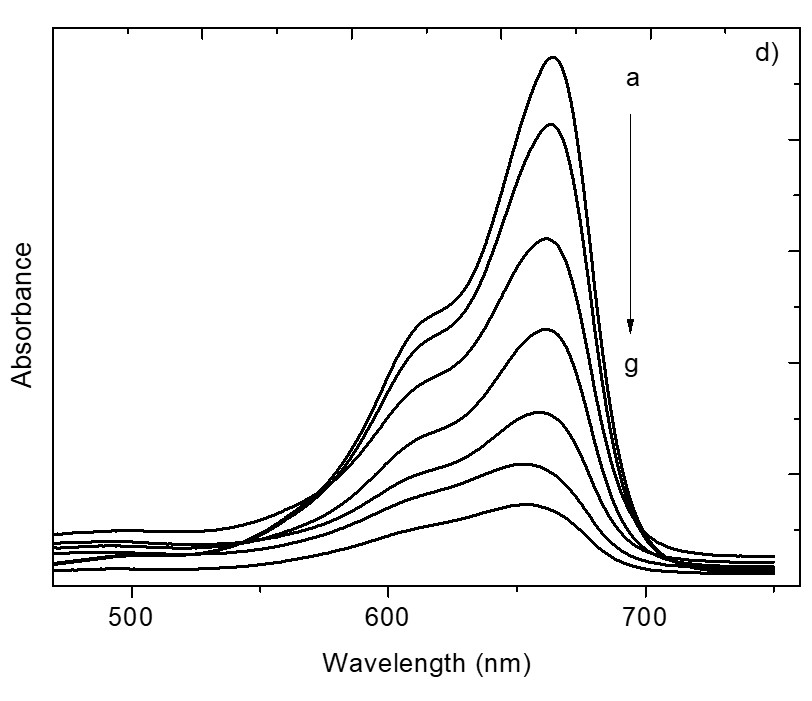
 The topography and surface morphology of materials can be effectively seen at high magnifications using Scanning Electron Microscopy (SEM).A focused electron beam is used by SEM to scan the surface of the sample. The interaction between the high-energy electrons in the beam and the atoms on the surface results in different signals.SEMs can also use detectors for Energy Dispersive X-ray Spectroscopy (EDS). The high-energy electrons may knock out electrons in the inner shell when they hit the sample. Higher energy electrons descend to fill these vacancies, emitting X-rays that correspond to the elements present. Applying EDS to a sample at certain spots on its surface can provide details about its elemental makeup.

Fig 3 :EDX Analysis of Ni doped Mn2O3

Ni-doped Mn2O3 is examined using EDX (Energy-Dispersive X-ray Spectroscopy) SEM in Fig. 3, which examines the elemental composition of the sample. It can demonstrate the presence and distribution of elements such as Ni, Mn, and O in the material. Researching material qualities and potential uses requires an understanding of how Ni doping affects the composition of Mn2O3, and the findings corroborate this understanding. The data in Figure 4 suggests that manganese and oxygen are the main constituents of the sample, as Mn2O3 is composed of these atoms. The occurrence of contaminants or other elements in tiny levels is indicated by the presence of trace elements (0.2%, 0.2%, and 0.1%).The energy of the X-rays employed in the analysis is represented by the energy level (15 keV). With the use of EDX analysis, a potent method, one can ascertain a material's elemental composition by analyzing the distinctive X-rays released when high-energy electrons bombard the sample.



**Fig 4:**Photocatalytic application of Ni doped Mn2O3

Photocatalytic degradation of Ni-doped Mn2O3 is a technique that uses light energy to propel chemical reactions that decompose organic pollutants or other substances. In this case, the material being studied is Ni-doped Mn2O3, manganese oxide doped with nickel atoms. The absorbance readings at specific wavelengths (500 nm to 700 nm) reveal the material's absorbance properties across this range.The absorbance values at different wavelengths suggest that Ni-doped Mn2O3 may absorb visible spectrum light, which is between 500 and 700 nm in wavelength. Because the material may employ solar energy for catalytic reactions by absorbing visible light, this is a crucial part of photocatalysis.

# DISCUSSION

Nickel dopants can be added to Mn₂O₃ to boost its catalytic activity. By altering a material's electrical structure, doping can enhance surface area, charge separation, and absorption capabilities. These components increase the efficiency of the photocatalytic process [(Jenke, 2022)](https://paperpile.com/c/kLJ271/HZcm). Ni-doped Mn₂O₃ can be utilized in environmental remediation procedures such as the cleaning of air and wastewater due to its photocatalytic properties. Additionally, it can be researched for usage in solar fuel manufacturing and other sustainable energy applications [(Jaroszewski et al., 2018)](https://paperpile.com/c/kLJ271/Kkcp).When light is absorbed by Ni-doped Mn₂O₃, excited electrons create electron-hole pairs. These high-energy electron-hole pairs may participate in redox reactions on the material's surface [(Serp & Minh, 2022)](https://paperpile.com/c/kLJ271/X3BS). A photocatalytic degradation process occurs when pollutants deposited on the catalyst's surface interact with excited electrons and holes. This process breaks down the pollutants into smaller, less harmful molecules, or they may even mineralize completely into water and carbon dioxide [(Moura et al., 2016)](https://paperpile.com/c/kLJ271/PA2L)The XRD analysis plays a major role in determining the manufactured material's crystal structure and phase purity. The peaks in the XRD pattern that correspond to specific crystallographic planes verify the existence of the Mn₂O₃ phase [(Kadish & Ruoff, 2000)](https://paperpile.com/c/kLJ271/obbG). A detailed analysis of peak positions, intensities, and widths provides information about the substance's crystalline makeup and crystallite sizes[(Pranati et al., 2021; Sakthi et al., 2021)](https://paperpile.com/c/kLJ271/mZbhN+QMpcl). Wide peaks may indicate a small crystallite size or an amorphous phase. When compared to pure Mn₂O₃, the XRD pattern exhibits additional peaks or shifts, indicating the successful integration of nickel dopants[(G. & Ganapathy, 2022; Kumar & Ramesh, 2021)](https://paperpile.com/c/kLJ271/MfdCm+2DHDP)). These differences, which reflect changes in the lattice properties caused by nickel's different atomic size from manganese's, support the doping effect [(Sitharaman, 2016)](https://paperpile.com/c/kLJ271/zvMt).Zn-doped MnO has been demonstrated to be the ideal sensing material for xylene vapors due to its low-temperature selectivity and activity . Manganese oxide catalysts that were both undoped and zinc doped were successfully produced using a straightforward wet impregnation technique. XRD analysis indicates that the catalyst's components are α-MnO₂ structures. However, due to the low zinc content in the Zn-doped MnO, the zinc peaks in the catalyst's diffraction patterns cannot be distinguished[(Wang et al., 2025)](https://paperpile.com/c/kLJ271/xuyt). The SEM analysis indicates that the catalyst is composed of aggregated particles.Based on the XRD pattern, the FCC structure of CuMn₂O₄ NPs is used to calculate the particle size, dislocation density, microstrain, microstress, and lattice parameters. The functional groups that are present in the generated nanoparticles are identified using FTIR spectroscopy [(Wang et al., 2025)](https://paperpile.com/c/kLJ271/xuyt).To evaluate and compare pure and doped nanofibers' properties for potential optoelectronic applications, the physical properties of the nanofibers fluctuate with the concentration of Ni [10]. Using PDDA, a straightforward self-assembly method is proposed for GO/Mn₂O₃ nanosheet deposition on NiF for supercapacitor applications. TEM, FE-SEM, XRD, and XPS investigations were used for comprehensive characterization. To show that pure Mn₂O₃ was present, the electrode's different Mn valencies were compared . The resulting Ni-doped Mn₂O₃ exhibited higher photocatalytic activity in comparison to pure Mn₂O₃. These components contributed to the material's remarkable ability to decompose organic pollutants when exposed to light [(Tsang & Wang, 2021)](https://paperpile.com/c/kLJ271/akZq).

# CONCLUSION

The work successfully employed the solid-state synthesis strategy by verifying the fabrication of nickel-doped Mn2O3 using X-ray diffraction analysis. The addition of nickel dopants to the Mn2O3 crystal structure, as demonstrated by the synthesis process.Advanced characterization techniques like Ni SEM analysis and EDX SEM analysis were used to extensively examine the surface morphology and elemental content of the synthesized material. These studies confirmed the synthesis process by verifying the presence of nickel dopants and the structural integrity.Determining the material's long-term durability and scalability for large-scale applications will also be crucial to its actual implementation.

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