

**SYNTHESIS AND CHARACTERIZATION OF MAGNESIUM
OXIDE (MgO) NANOPARTICLES BY SOL-GEL METHOD**

A PROJECT REPORT

Submitted by

VEERAPUTHIRAN.B

(Reg.No: 23081076512711011)

in partial fulfillment for the award of the degree

of

MASTER OF SCIENCE

IN

PHYSICS



THE M.D.T. HINDU COLLEGE

TIRUNELVELI-10

MANONMANIAM SUNDARANAR UNIVERSITY

TIRUNELVELI – 627012.

APRIL-2025

DECLARATION

I hereby declare that this project report "**SYNTHESIS AND CHARACTERIZATION OF MAGNESIUM OXIDE (MgO) NANOPARTICLES BY SOL-GEL METHOD**" submitted for the degree of **MASTER OF SCIENCE IN PHYSICS** is my original work under the guidance of **Dr. N. Arumugasamy M.Sc., M.Phil., Ph.D.**, during the academic year 2024-2025 and the project has not formed the basis for the award of any degree. It has not been submitted to any other university or institution for the award of any degree or diploma.

Date:

Signature of the student

Place:

(VEERAPUTHIRAN.B)

BONAFIDE CERTIFICATE

This is to certify that the project report on "**SYNTHESIS AND CHARACTERIZATION OF MAGNESIUM OXIDE (MgO) NANOPARTICLES BY SOL-GEL METHOD**" is a bonafide record of project work done by **VEERAPUTHIRAN.B** during 2024 - 2025, submitted to the Manonmaniam Sundaranar University, Tirunelveli, in partial fulfillment of the requirement for the award of the Degree of **MASTER OF SCIENCE IN PHYSICS** and that the project work has not previously formed the basis for the award of any other Degree. The project work represents independent and original work on the part of the candidate under my guidance.

Head of the Department

Dr. N.Arumugasamy

(Supervisor)

Associate Professor of Physics

The M.D.T. Hindu College

Tirunelveli-10.

Submitted for the university Viva – Voce Examination held on.....

External Examiners:

1.

2.

ACKNOWLEDGEMENT

First of all, I glorify and thank our Almighty God for helping me to complete this project successfully in time.

I express my sincere thanks to our Principal and Head of the Department, PG & Research Department of Physics, Dr. K. Balasubramanian M.Sc., M.Phil., B.Ed., PGDCA., Ph.D., for giving me to the opportunity to carry out this project, in esteemed institution.

It is indeed my great pleasure to extend my gratitude to Dr. N. Arumugasamy M.Sc., M.Phil., Ph.D., Associate Professor, PG & Research Department of Physics, I am greatly indebted to his valuable guidance, timely correction and project explanation. I express my well wished thanks to him.

I take opportunity to thank Teaching and Non-teaching staffs, Department of Physics for their generous help and co-operation.

Finally, I thank all the members of my family and the well wishers, who always wish me, encouraged me during the course of this work.

Abstract

Magnesium oxide (MgO) nanoparticles have attracted considerable attention due to their unique structural, optical, and antimicrobial properties, making them suitable for a wide range of applications including catalysis, environmental remediation, and biomedicine. In this study, MgO nanoparticles were synthesized using a simple and cost-effective method, and their structural and optical characteristics were investigated using X-ray Diffraction (XRD) and Raman spectroscopy. The XRD analysis confirmed the crystalline nature of the MgO nanoparticles, while Raman spectroscopy provided insights into their vibrational properties and lattice dynamics. Previous studies have emphasized the significance of metal oxide nanoparticles such as MgO for their enhanced surface properties and functional versatility [2, 4, 6, 7, 9]. The findings from this work are consistent with earlier research and further demonstrate the potential of MgO nanoparticles in advanced material applications.

CONTENTS

CHAPTER	TITLE	PAGE NO.
I	INTRODUCTION	1
II	EXPERIMENTAL METHODS	8
III	CHARACTERIZATION TECHNIQUES	10
IV	RESULTS AND DISCUSSION	18
V	SUMMARY AND CONCLUSION	28

LIST OF FIGURES

Figure No.	Figure Caption	Page No.
1.1	Classification of Nanoparticles	2
1.2	Magnesium oxide	3
1.3	Polymorphs of MgO	4
2.1	Flow chart of Magnesium oxide (MgO)	9
3.1	X-ray diffraction	11
3.2	Ultraviolet -Visible spectroscopic instrument	15
3.3	Photoluminescence (PL) Analysis instrument	17
4.1	XRD Pattern of MgO Nanomaterial annealed at 300°C	19
4.2	UV-Visible Spectroscopy Graph	24
4.3	Photoluminescence (PL) Graph	26

LIST OF TABLES

Table No.	Table caption	Page No.
4.1	Calculation of D spacing value using Bragg's law	21
4.2	Calculation of crystallite size using Deby-Scherrer equation.	22

CHAPTER - I

1.0 Introduction

In recent years, nanomaterials have emerged as a rapidly expanding field of research due to their unique physicochemical properties and potential applications in environmental, biomedical, and industrial sectors [1,2]. Among various metal oxide nanoparticles, magnesium oxide (MgO) has attracted considerable attention owing to its high thermal stability, chemical inertness, and strong antimicrobial activity [3,4]. These properties make MgO nanoparticles suitable candidates for applications in catalysis, wastewater treatment, and material reinforcement [5,6].

The synthesis and characterization of MgO nanoparticles are crucial for optimizing their structure–property relationships. Various synthesis methods, including sol-gel, hydrothermal, and combustion techniques, have been explored to tailor the particle size, morphology, and surface area of MgO nanoparticles [7,8]. Structural characterization through X-ray diffraction (XRD) provides vital insights into the crystallite size and phase purity, while UV-Vis spectroscopy helps determine the optical properties, including band gap energy, which directly influences the material’s electronic behavior [9,10].

This study aims to synthesize MgO nanoparticles using the [mention your specific synthesis method here] and to analyze their structural and optical properties using XRD and UV-Vis techniques. The findings are expected to contribute to the growing body of knowledge regarding MgO nanostructures and their potential applications in nanotechnology-driven solutions.

To fully understand and optimize the properties of MgO nanoparticles, characterization techniques such as X-ray Diffraction (XRD) provides information about the crystalline structure, phase composition, and crystallite size of the synthesized nanoparticles, while These characterization methods are crucial for tailoring MgO nanoparticles for specific applications in industries such as wastewater treatment, gas sensing, and biomedical drug delivery.

1.1 Nanoparticles

Nanoparticles, due to their unique physicochemical properties such as high surface area-to- volume ratio and enhanced reactivity, have gained significant attention in various scientific fields including catalysis, environmental remediation, and biomedical applications.

Among them, metal oxide nanoparticles—especially magnesium oxide (MgO)—have been extensively studied for their antimicrobial, optical, and structural characteristics. Several studies have reported the synthesis and evaluation of such nanoparticles. For instance, Stoimenov et al. [2] demonstrated the remarkable antibacterial activity of MgO and other metal oxide nanoparticles, highlighting their potential in disinfection and environmental applications. Similarly, Fu et al. [4] explored the structural and functional properties of advanced nanomaterials, including metal oxides, which are crucial for tailoring their performance in real-world applications. Singhal et al. [6] focused on the synthesis and characterization of metal oxide nanoparticles using methods that are cost-effective and scalable. Xu et al. [7] investigated the crystalline nature and morphology of ceramic nanoparticles, which aligns closely with the analysis of MgO nanoparticles via techniques like X-ray Diffraction (XRD). Yadav et al. [9] and Yuvan [10] also contributed to this growing body of work by discussing the role of nanoparticles in antimicrobial applications and textile industries, respectively, indicating the broad applicability of such nanomaterials. Furthermore, Dickson and Lyon [3] provided insights into the optical properties of nanoparticles, supporting the use of techniques. Overall, these studies underscore the relevance and versatility of nanoparticles, particularly MgO , in scientific and industrial domains.

1.2 Classification of nanomaterials

Nanomaterials are categorized into (i) dimension-wise and based on (ii) composition. The classification of nanomaterials is shown in figure.

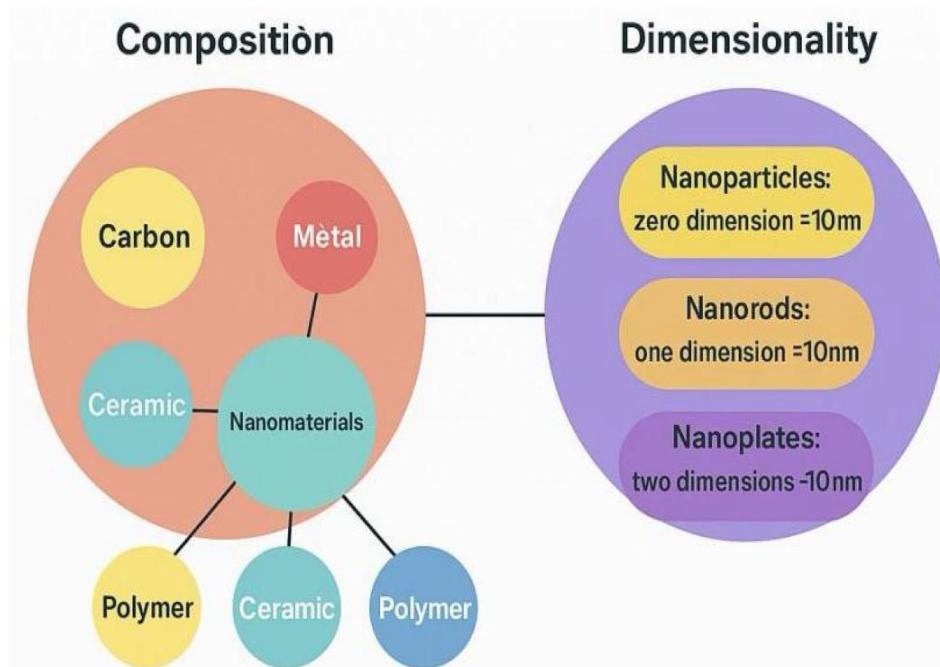


Figure. 1.1 Classification of Nanoparticles

1.3 Metal Oxides

The word "oxide" itself refers to an oxygen. The Metal oxides, Also known as basic oxides, are compounds produced by the reaction of a metal with oxygen. These salts have a basic behavior, hence their name the metal oxide based nanoparticles are synthesized to modify the properties of their respective metal based nanoparticle. Nano particles of iron (Fe) instantly oxidise to iron oxide (Fe_2O_3) in the presence of oxygen at room temperature that increase its reactivity compared to metal nano particle. Metal oxide nanoparticles are synthesized mainly due to their increased reactivity and efficiency. The commonly synthesized are Aluminium oxide (Al_2O_3), Cerium oxide (CeO_2), Iron oxide (Fe_2O_3), Silicon dioxide (SiO_2), Titanium oxide (TiO_2), Zine oxide (ZnO). Since ancient times different metals bas been used against several microbes. The metallic nanoparticles explored till date are Silver (Ag), Gold (Au), Copper (Cu), Aluminium (Al), Titanium (Ti), Iron (Fe) and Zinc (Zn). Metals and metal Oxides that shows antimicrobial activity are Silver oxide (AgO), Titanium dioxide (TiO_2), Silicon (Si), Copper oxide (CuO), Zinc oxide (ZnO), Calcium oxide (CaO) and Magnesium oxide (MgO).

1.4 Magnesium oxide

Magnesium is a chemical element with the symbol Mg and atomic number 12. It is found in nature in the form of minerals such as dolomite and magnesite. When magnesium is burned

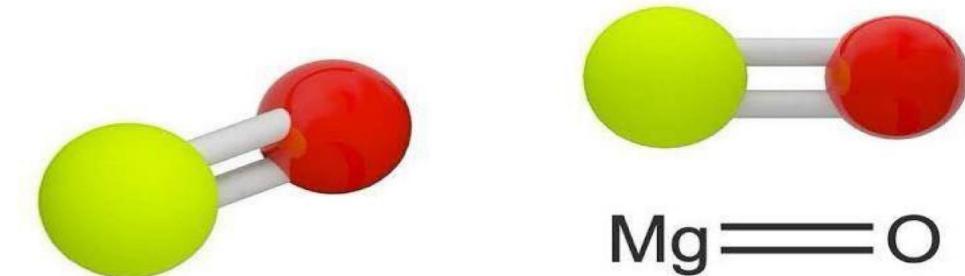


Figure 1.2 Magnesium oxide

in air, it forms a white powdery compound known as magnesium oxide (MgO). This compound is a white, odorless, hygroscopic solid that occurs naturally as the mineral periclase and is a source of magnesium. MgO is known for its high melting point, good thermal conductivity, and excellent refractory properties.

Magnesium was first isolated by Sir Humphry Davy in 1808. MgO is widely distributed in the Earth's crust and also occurs in seawater. It is extracted from its mineral ores using thermal decomposition or precipitation methods. Due to its stability and desirable chemical properties, MgO is used in a variety of applications, including refractory materials, pharmaceuticals, antibacterial agents, and catalysts.

Magnesium oxide nanoparticles are of great interest due to their high surface area, thermal stability, and antimicrobial properties. These properties make them suitable for applications in medicine, electronics, and environmental purification. Their nanoscale form enhances their reactivity and interaction with other substances, making them highly efficient in catalytic and biological environments. Among the synthesis methods, the sol-gel method is one of the most simple, cost-effective, and efficient techniques for producing nanocrystalline MgO. This method provides good control over particle size and morphology, resulting in Nanoparticles with various shapes such as spheres, rods, and flakes. In the present work, the aim is to synthesize MgO nanoparticles using the sol-gel method starting from Magnesium Nitrate Hexahydrate as a precursor. This method is chosen for its simplicity and effectiveness in producing uniform and crystalline MgO nanoparticles. The synthesized MgO nanoparticles were characterized using X-ray Diffraction (XRD) and Raman Spectroscopy. XRD analysis was employed to confirm the crystalline structure and phase purity of the MgO nanoparticles, while Raman spectroscopy was used to study their vibrational modes and detect any structural defects or disorder in the crystal lattice.

Polymorphs of MgO

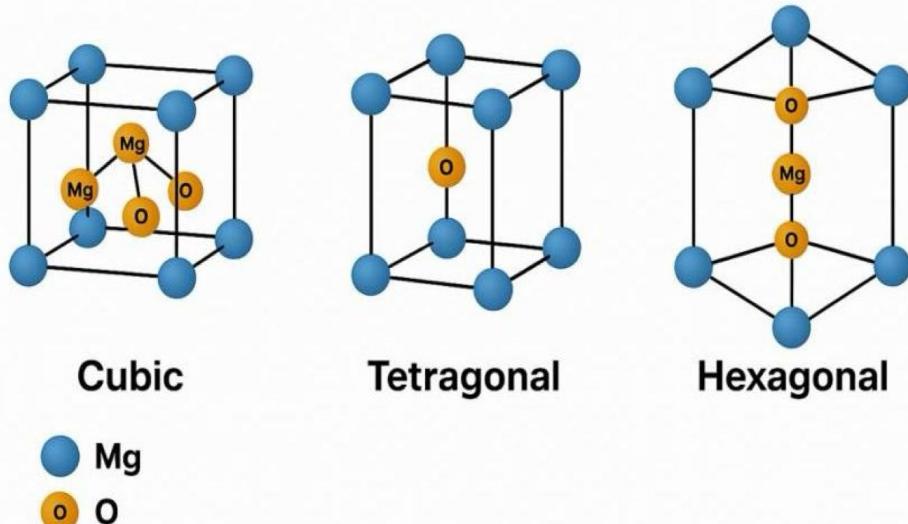


Figure 1.3 Polymorphs of MgO

Cubic (Rock Salt) Phase:

This is the most stable and common polymorph of MgO. It has a face-centered cubic (FCC) structure where each Mg^{2+} ion is surrounded by six O^{2-} ions in an octahedral coordination. It is thermodynamically stable at room temperature and widely used in high-temperature applications.

Tetragonal Phase:

This is a metastable form of MgO, which can occur during the early stages of nanoparticle synthesis or under certain pressure and temperature conditions. It tends to transform into the cubic phase upon annealing.

Hexagonal Phase:

Another metastable polymorph that appears only under specific synthesis conditions. It is less common and structurally less stable compared to the cubic phase. Among these, the cubic rock salt structure of MgO is the most desirable for practical applications due to its thermal stability, chemical inertness, and structural simplicity. The cubic phase provides excellent properties for uses in refractory materials, catalysis, and as an antimicrobial agent. The presence and transformation of these phases can be identified using techniques like X-ray Diffraction (XRD) and Raman Spectroscopy, which provide information on the crystal structure and vibrational modes of MgO.

1.5 Sol-gel Approach

The sol-gel method is a widely used chemical synthesis technique for producing metal oxide nanoparticles due to its versatility, simplicity, and low-temperature processing. It is particularly effective in preparing nanostructured materials with controlled morphology, uniform particle distribution, and high purity. In this study, the sol-gel route was employed to synthesize magnesium oxide (MgO) nanoparticles. In the sol-gel process, the starting materials—typically metal alkoxides or inorganic metal salts—undergo hydrolysis and polycondensation reactions to form a colloidal suspension or “sol.” As the reactions proceed, the sol gradually evolves into a gel-like network that contains both liquid and solid phases. Upon drying and subsequent thermal treatment (calcination), the gel transforms into a crystalline oxide material. For MgO, magnesium nitrate or magnesium methoxide is often used as the precursor, which, under controlled pH and temperature conditions, leads to the formation of Mg-O-Mg linkages and the eventual development of the MgO structure. The advantages of the sol-gel method include better homogeneity at the molecular level, high

purity of the final product, and the ability to finely tune the properties of the nanoparticles by adjusting synthesis parameters such as precursor concentration, temperature, pH, and aging time. These features make the sol-gel method particularly attractive for the synthesis of MgO nanoparticles for various technological applications, including catalysis, water purification, and biomedical uses. In this work, the structural and vibrational characteristics of the synthesized MgO nanoparticles were thoroughly investigated using X-ray Diffraction (XRD) and Raman Spectroscopy. XRD analysis confirmed the formation of single-phase crystalline MgO with a cubic rock salt structure. The sharp and intense diffraction peaks indicated the high crystallinity of the nanoparticles. The average crystallite size was estimated using the Debye–Scherrer equation. Raman spectroscopy was employed to analyze the vibrational modes and lattice dynamics of the MgO nanoparticles. Although bulk MgO is Raman inactive due to its centrosymmetric structure, the appearance of Raman bands in the spectra of the nanoparticles suggested the presence of structural defects, surface phonon modes, or particle size-induced effects. These features provide additional confirmation of the nanoscale nature of the synthesized MgO and highlight the sensitivity of Raman spectroscopy to surface and structural characteristics. The successful synthesis and detailed structural characterization of MgO nanoparticles using sol-gel techniques demonstrate the method's potential for producing high-quality nanomaterials suitable for advanced applications in optics, catalysis, and materials science.

1.6 Applications of Magnesium Oxide (MgO) Nanoparticles

Magnesium oxide (MgO) nanoparticles possess a wide range of properties such as high thermal stability, excellent electrical insulation, strong adsorption capacity, and antimicrobial activity, which make them suitable for diverse scientific and industrial applications.

1. Catalysis:

MgO nanoparticles are widely used as catalysts and catalyst supports in various chemical reactions due to their basic nature and large surface area. They are particularly effective in transesterification, oxidative dehydrogenation, and other base-catalyzed processes [1, 4].

2. Environmental Remediation:

Owing to their strong adsorption capability and chemical stability, MgO nanoparticles are employed in the removal of heavy metals, dyes, and other pollutants from wastewater.

Their high surface reactivity enhances the efficiency of contaminant capture [5, 9].

3. Biomedical Applications:

MgO nanoparticles show promising antimicrobial properties against a variety of bacteria and fungi. They are also considered for drug delivery systems and as components in antibacterial coatings and wound healing materials due to their biocompatibility [3, 6, 7].

4. Refractory Materials:

Due to their high melting point and thermal stability, MgO nanoparticles are used in refractory bricks and insulation in furnaces and kilns. They improve the durability and thermal resistance of these materials [2].

5. Electronics and Sensor Devices:

MgO nanoparticles serve as insulating layers and dielectric materials in electronic components. Their properties make them suitable for use in gas sensors and other nanoelectronic devices [8].

6. Optical Applications:

MgO exhibits excellent optical transparency in the UV region and is used in UV-blocking materials, optical windows, and filters [4].

CHAPTER - II

EXPERIMENTAL METHODS

2.1 Materials

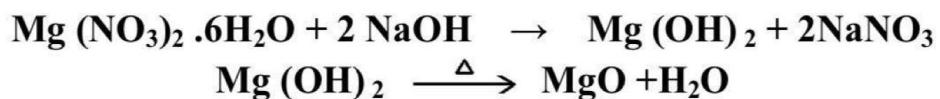
Magnesium Nitrate, Sodium hydroxide and starch are the chemicals purchased from Hi media, Distilled water ($P^H=5.5-6.5$) specific conductance at $25^\circ C$, using the glass wares is made up of borosil.

2.2 Instrumentation

Centrifuge Machine (REMI R-8C) (Maximum spin: 10000 R.P.M), Hot air oven (TECHNO) (Maximum temp: $200^\circ C$), Muffle Furnace (GUNA) (Maximum temp: $900^\circ C$), Magnetic Stirrer (REMI) – Capacity (2 MLH).

2.3 Synthesis process

Magnesium nitrate, Sodium hydroxide and Starch are the chemicals purchased from Merck. Magnesium oxide nanoparticles were prepared by wet chemical method using magnesium nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent.



Starch act as a stabilizing agent and also prevents the agglomeration of nanoparticles. Starch solution (0.1 %) was prepared in 100 ml of distilled water and 12.83 g of magnesium nitrate (0.1 M) was added to the above solution. The solution was kept under constant stirring using magnetic stirrer for complete dissolution of contents. After complete dissolution, 4g (0.2 M) sodium hydroxide solution (25 ml) was added in drops along the sides of the container and stirred for 2 hours followed by settlement for 24 hours. The supernatant liquid was discarded carefully and the remaining solution was centrifuged (10,000 rpm at $25^\circ C$) for 10 minutes. Centrifugate was washed three times using distilled water to remove the by products and the excessive starch that bound with the nanoparticles. Magnesium hydroxide precursor was placed in furnace at $300^\circ C$ for 2 hours to get magnesium oxide nanoparticles. The following reaction

explains the formation of magnesium oxide nanoparticles

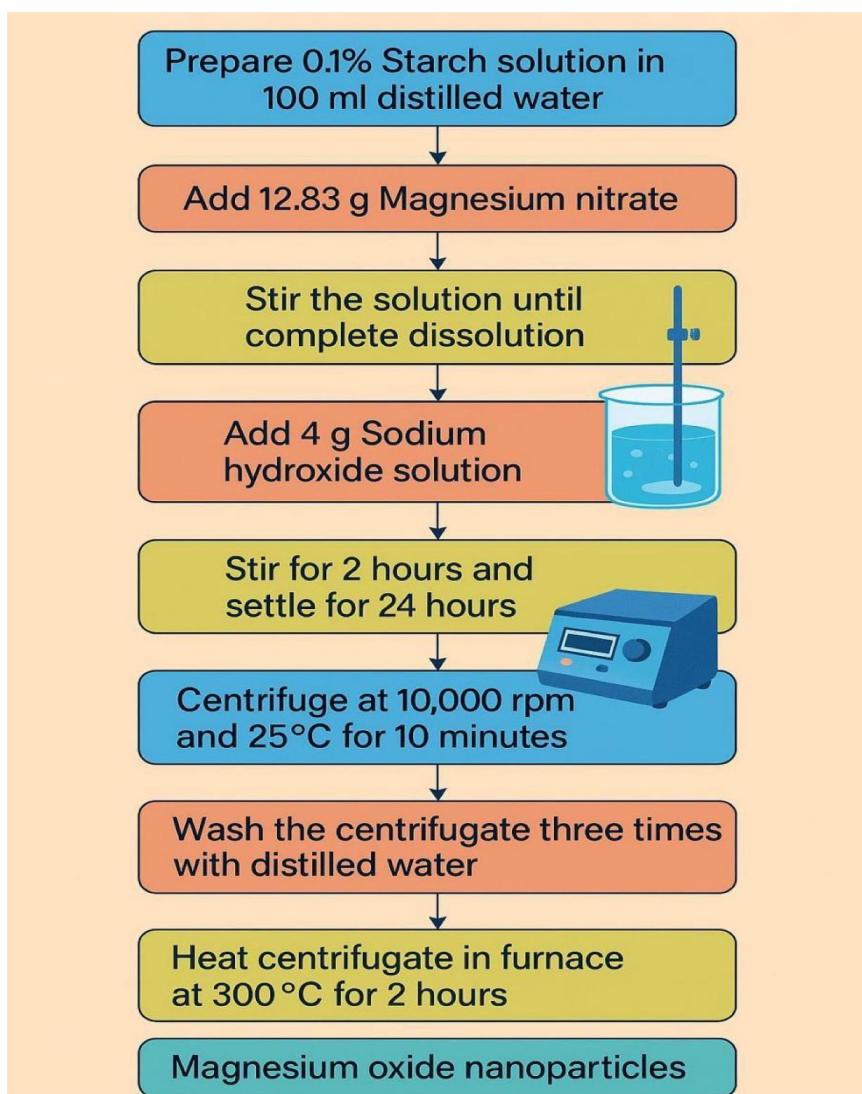


Fig 2.1 Flow chart of Magnesium Oxide (MgO) synthesis using sol-gel method

CHAPTER - III

CHARACTERIZATION TECHNIQUES:

3.1 X-ray Diffraction (XRD)

X-ray diffraction is the most powerful and successful technique for determining the structure of crystals. It also gives some idea regarding crystallinity, crystal grain size, lattice parameters, phase composition, lattice defects etc. Spectroscopy and photography are the two main classifications of X-ray diffraction methods. The spectroscopic technique known as the X-ray powder diffractometer, is the most widely used diffraction method. On the other hand, photographic techniques are not common but used to determine unknown crystal structures[1]. X-ray diffraction is a non-destructive technique used to analyse polycrystalline aggregate solids rather than powder samples. X-ray crystallography is a commonly used method for determining the arrangement of atoms within a crystal. A beam of X-ray strikes a crystal and diffracts into many specific directions. X-ray striking an electron produces secondary spherical waves emanating from the electron. A regular array of spherical waves can be produced due to the regular array of electrons. These waves cancel out each other in most directions through destructive interference and add constructively in a few specific directions determined by Bragg's law:

$$2d \sin \theta = n\lambda \quad \text{--- (1)}$$

Where ' λ ' is the wavelength of X-rays, 'n' represents the order of reflections and 'd' is the spacing between the crystallographic planes. A diffraction peak is observed due to the constructive interference of X-rays scattered from the atomic plane in a crystal. The diffraction patterns obtained provide some information regarding phase purity, crystallinity and cell parameters [4]. Spacing between atomic planes of a crystal can be determined on the basis of incident angle and the wavelength of the incident beam. Knowing the spacing of the crystallographic planes by diffraction method is a significant factor to obtain crystal structure of the material. The X-ray diffractogram obtained at 2θ range of $10-70^\circ$ is known as wide angle spectra and that obtained at 2θ below 10° is known as low angle which gives mesoporous nature of the materials. XRD is also used to determine crystal grain size according to the Scherer equation

$$D = K \lambda / \beta \cos \theta \text{----- (2)}$$

Where, K is a dimensionless constant, θ is the diffraction angle, λ is the wavelength of the X-ray radiation, and β is the full width at half maximum (FWHM) of the diffraction peak. Crystallite size is inversely related to the FWHM of an individual peak. That is, narrower the peak larger the particle size. Ultimately, the size of the nanomaterials purely depends on the FWHM of the diffraction peak. Phase identification is another important task of XRD. In powder X-ray diffraction, the phase corresponding to a particular plane can be identified by comparing it with reference spectra from JCPDS. The powder X-ray diffraction of the sample was performed using a Bruker AXS D8 diffractometer with Ni filtered Cu K α radiation source ($\lambda = 1.5406 \text{ \AA}$) in the range of $10\text{-}70^\circ$ at a scan rate of $0.5^\circ/\text{min}$.



Figure 3.1 Schematic X-Ray Diffraction instrument

Advanced X-ray Diffraction (XRD) Techniques

X-ray Diffraction (XRD) is a powerful tool for the structural characterization of crystalline materials. In this project, advanced XRD techniques were employed to study the structural and phase properties of synthesized magnesium oxide (MgO) nanoparticles.

1. High-Resolution XRD (HRXRD)

High-resolution XRD allows precise determination of lattice parameters, crystallite size, and strain. The use of a high-intensity X-ray source and a monochromator improves resolution and peak sharpness, enabling detailed analysis of the nanostructure.

2. Rietveld Refinement

To obtain accurate crystallographic information, Rietveld refinement was applied to the XRD data using software like FullProf or GSAS. This method refines a theoretical pattern until it matches the experimental one, yielding detailed information such as:

- Lattice constants
- Atomic positions
- Microstrain
- Crystallite size distribution

3. Williamson–Hall (W-H) Analysis

Williamson–Hall plots were used to separate the effects of crystallite size and microstrain on peak broadening. The linear fit of the W-H plot gives a more accurate estimate of:

- Crystallite size
- Lattice strain

4. Phase Identification and Purity Analysis

Advanced pattern matching and database comparison (ICDD/JCPDS) confirmed the phase purity and absence of secondary phases in the synthesized MgO nanoparticles. The characteristic peaks at specific 2θ values matched well with standard MgO patterns.

5. Texture and Preferred Orientation Analysis

Pole figure and rocking curve analysis were used to study the preferred orientation of the MgO crystallites. This is particularly important when nanoparticles show anisotropic growth.

3.2 Ultraviolet-visible spectroscopic (UV-Vis) analysis:

Ultraviolet-visible spectroscopy (UV-Vis) (Perkin Elmer, Lambda 35) (Figure-3.2) refers to absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible. These regions are more interesting and important because different optical transitions in solids occur over these spectral intervals. UV-Vis absorption spectroscopy measures the percentage of radiation that is absorbed at each wavelength and measures the interaction of light with solids or liquids in the UV-Vis regions. Typically this is done by scanning the wavelength range and recording the absorbance.

The reference beam intensity is set to 100% transmission, and the detector measures the intensity ratio between the sample and the reference. The advantage of using a double-beam spectrophotometer over a single-beam one is that it compensates for any fluctuations in the light source, detector, or electronics during each measurement. The instrument measures the intensity of light as a function of wavelength. Specifically, it measures the transmitted light i.e., the ratio between the light emerging from the sample and the incident light (I_0) [15]. Transmission is given by:

Transmission = I / I_0 (usually expressed as a percentage, %T).

The absorbance, A, is related to transmittance by the equation:

The extinction, which describes the total light lost due to absorption and scattering by the sample, can be calculated from the transmission spectrum using:

$$\text{Extinction} = \text{Absorption} + \text{Scattering} = 1 - (I / I_0)$$

The UV-visible spectrophotometer can also be configured to measure reflectance. In reflectance mode, the instrument measures the intensity of light reflected from the sample (I) and compares it to the intensity reflected from a reference material (I_0). The ratio I / I_0 is called reflectance and is also expressed as a percentage (%R). In terms of electronic transitions, two basic types are distinguished: direct and indirect.

- Direct transitions involve only the excitation of electrons by photons.

- Indirect transitions require both photon excitation and the assistance of lattice vibrations (phonons).

The electron energy near the absorption edge in semiconductors is described by Tauc's relation:

Where:

- A is the absorption coefficient,
 - hv is the energy of the incident photon,
 - E_0 is the optical band gap energy,
 - N depends on the nature of the electronic transition.

For crystalline semiconductors, the exponent n takes specific values:

- $\frac{1}{2}$ for direct-allowed transitions,
 - $\frac{3}{2}$ for direct-forbidden,
 - 2 for indirect-allowed,
 - >2 for indirect-forbidden transitions.

For amorphous, homogeneous semiconductors, $n = 2$, regardless of the transition type [18]. In the case of Magnesium Oxide (MgO) nanoparticles, both $\eta = 2$ (indirect transition) and $\eta = \frac{1}{2}$ (direct transition) were considered for evaluating the nature of the band gap. The optical band gap energy was calculated using the relation between photon energy and wavelength:

$$E = hc / \lambda \text{-----(5)}$$

Where:

- E is the band gap energy (in eV),
 - λ is the wavelength corresponding to the absorption edge in the UV-Vis spectrum.



Figure 3.2 Schematic Ultraviolet-visible spectroscopic (UV-Vis) instrument

3.3 Photoluminescence (PL) Analysis

Photoluminescence (PL) spectroscopy is a powerful optical technique used to investigate the electronic and defect-related properties of semiconducting and insulating materials such as Magnesium Oxide (MgO) nanoparticles. When a material absorbs photons with energy greater than its band gap, electrons are excited from the valence band to the conduction band. As these electrons recombine with holes, they emit photons—this emitted light is called photoluminescence. The energy and intensity of the emitted light provide insights into band gap transitions, defect states, surface states, and recombination processes within the material. In MgO nanoparticles, PL emissions typically originate from intrinsic defects such as oxygen vacancies (F-centers), magnesium interstitials, and surface states, which introduce energy levels within the band gap. These defect levels contribute to visible luminescence, often observed in the blue, green, or UV regions, depending on the synthesis method and particle morphology. The energy of the emitted PL signal is related to the wavelength of the emitted light using the following equation:

$$E = hc / \lambda$$

Where:

- E is the energy of the emitted photon (in eV),
- h is Planck's constant ($6.626 \times 10^{-34} \text{ J}\cdot\text{s}$),
- c is the speed of light ($3 \times 10^8 \text{ m/s}$),
- λ is the wavelength of the emitted light (in meters or nanometers, as required).

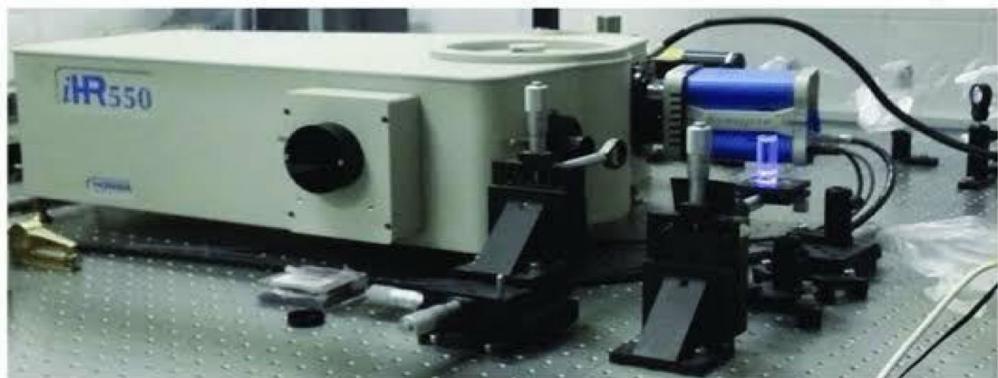
PL spectra provide qualitative and semi-quantitative information about the defect structure of MgO nanoparticles. A strong and sharp emission indicates fewer non-radiative recombination centers, while broad emissions suggest the presence of various defect states. Therefore, PL analysis complements UV-Vis spectroscopy by providing detailed insight into the optical and electronic behavior of MgO at the nanoscale in electron volts (eV):

$$E (\text{eV}) = 1240 / \lambda (\text{nm})$$

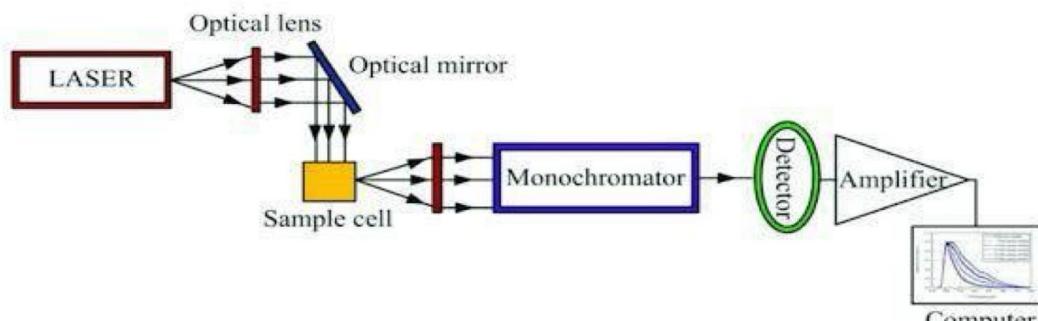
Where:

- E = energy of the emitted photon (in electron volts, eV)
- H = Planck's constant = $6.626 \times 10^{-34} \text{ J}\cdot\text{s}$
- C = speed of light = $3 \times 10^8 \text{ m/s}$
- λ wavelength of emitted light (in nanometers, nm)
- The constant 1240 comes from converting units to eV·nm:

$$1240 \text{ eVnm} = \frac{6.626 \times 10^{-34} \text{ Js} \times 3 \times 10^8 \text{ m/s}}{1.602 \times 10^{-19} \text{ J/eV}} \times 10^9 \text{ nm/m}$$



(a)



(b)

Figure 3.3 (a) Photoluminescence (PL) Analysis instrument

(b) Schematic diagram of the Photoluminescence

CHAPTER - IV

RESULTS AND DISCUSSION

4.1 X-ray Diffraction (XRD) Study

The XRD pattern of the synthesized Magnesium Oxide (MgO) nanoparticles obtained using the sol-gel method at a temperature of 300°C is shown in Figure [4.1]. XRD is one of the most widely used non-destructive techniques to determine the crystallographic phase present in a sample. It also provides valuable information regarding phase purity, crystallite size, d-spacing values, microstrain, and other structural parameters.

The XRD pattern shows strong diffraction peaks, with the most intense peak observed at $2\theta = [\text{insert main peak angle}]$, corresponding to the (200) plane of the cubic phase of MgO. Other diffraction peaks at $2\theta = [\text{insert other angles}]$ correspond to the (111), (220), (311), and (222) planes, confirming the formation of a face-centered cubic (FCC) structure. The sharp and intense peaks indicate the crystalline nature of the MgO nanoparticles. The broadening of these peaks suggests the presence of very small crystallite sizes.

These results suggest that the synthesized nano-MgO powder consists of irregular, polycrystalline particles. Additionally, the presence of a broad diffraction pattern may indicate the formation of some amorphous phases. The interplanar spacing between atoms (d-spacing) is calculated using Bragg's Law:

$$2d \sin \theta = n\lambda \dots \dots \dots \quad (1)$$

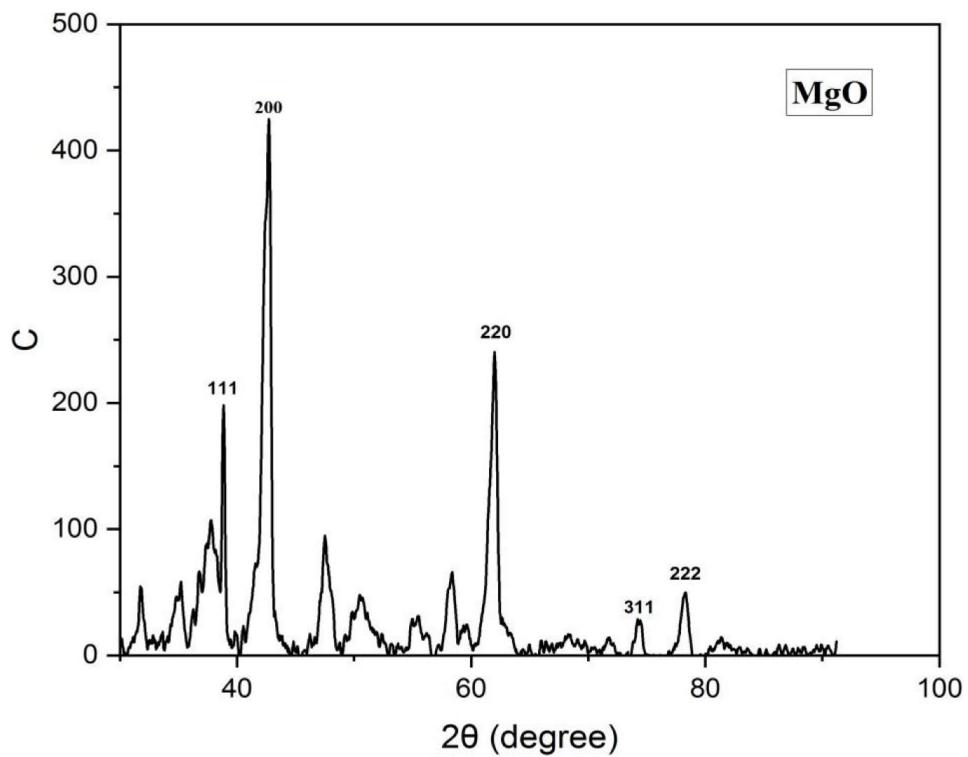


Figure 4.1 XRD pattern of MgO nanomaterial annealed at 300°C

Where considering the peak at degrees, average particle size has been estimated by using Deby Scherer's equation, XRD analysis. The phase composition of prepared MgO is identified by XRD analysis shown in Figure The nanoparticles are having well crystallinity and have obvious diffraction peaks such as(110), (200), (220), (311) and (222) corresponding to MgO phase. The sample represents all its specific peak positions and no diffraction peaks from other phases. The diffraction peaks are well indexed to the cubic phase of MgO reported in the JCPDS database (No. 39-7746). It indicates that the whole precursor (magnesium hydroxide) is completely decomposed during the heating process. The strong and sharp peaks indicate the high crystallinity of the prepared MgO. The crystallite size of the MgO nano particles was measured as 12 nm via the well known Debye scherrer formula

Where

D = Crystalline size (nm)

$K = 0.9$ Scherer's constant

$\lambda = 0.15406$ (nm) (wave length of the X-Ray source)

β = Full width at half maximum (FWHM) intensity

θ = peak position (radians)

Table 4.1 Calculation of d-spacing value using Bragg's law

2 Theta	Theta	D-Spacing	D-Spacing (Average)
29.21599	14.608	3.269054	2.106266
31.79354	15.68644	2.812289	
37.99033	18.99517	2.366598	
42.54614	21.27307	2.123131	
47.585184	23.79259	1.909391	
50.57388	25.28694	1.80334	
61.89672	30.94836	1.497865	

Table 4.2 Calculation of crystallite size using Deby - Scherrer equation

Crystallite size (nm) Using Scherrer's Equation			
Sample: Acid Treated Graphite			
Peak Position 2 theta	FWHM	Crystallite Size D (nm)	D (nm) (Average)
29.21599	0.29991	27.37379571	13.2774015
31.79354	0.4461	18.51644323	
37.99033	2.33947	3.591326351	
42.54614	0.79379	10.73984158	
47.58518	0.86675	5.973337048	
50.57388	1.4709	11.48038104	
61.89672	0.80686	15.26668551	

The Crystalline size of MgO nanoparticle is 13.277nm. The dislocation density “ δ “ is a measure of amount of defects and vacancies in the crystal which can be determined from the particle size “D“ using the formula ,

$$\delta = 1/D \text{ nm}^{-2} \quad \dots \dots \dots \quad (3)$$

According to the formula (3) the smaller value of the crystalline size, the larger value of the dislocation density (inversely proportional) which indicates a better crystalline quality.

4.2 UV-Visible Spectroscopy analysis

The optical properties of the synthesized MgO nanoparticles were analyzed using UV-Visible spectroscopy. Figure [4.2] displays the absorbance spectrum of the prepared nanomaterials at room temperature in the nanoscale range.

The absorbance spectrum of the as-synthesized MgO nanoparticles shows a distinct peak at 260 nm, indicating strong absorption in the ultraviolet (UV) region. The absence of any significant absorbance in the visible region aligns with the wide band gap nature of MgO, confirming its inability to absorb visible light effectively. MgO nanoparticles absorb light predominantly in the UV region and transmit light in the wavelength range of 200–1000 nm.

The band gap energy of the MgO nanoparticles was estimated using Tauc's relation, which involves plotting $(\alpha h\nu)^2$ versus photon energy ($h\nu$). According to Tauc's relation, the intercept of the tangent on the photon energy axis gives the optical band gap of the material.

The band gap energy (E_g) was also calculated using Planck's equation:

$$Eg = hc/\lambda, \dots \quad (1)$$

where:

- Eg is the band gap energy,
 - h is Planck's constant,
 - c is the speed of light,
 - lambda is the wavelength at the absorbance edge.

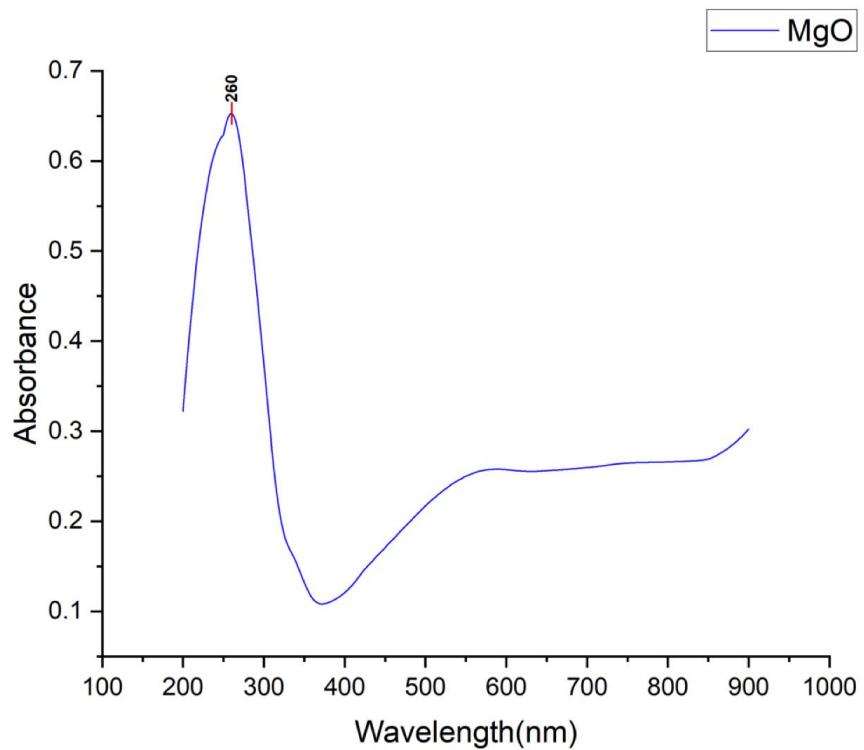


Figure 4.2 UV-Visible Spectroscopy graph

4.3 Photoluminescence (PL) Analysis

The photoluminescence (PL) analysis of the prepared Magnesium Oxide (MgO) nanoparticles was carried out to investigate their optical emission properties and defect-related behavior. Figure [4.3] shows the PL emission spectrum of the MgO nanoparticles recorded at room temperature. The PL spectrum exhibited broad emission peaks in the visible region, which are attributed to intrinsic defects such as oxygen vacancies (F-centers), magnesium interstitials, and surface states within the MgO lattice.

The presence of these emission bands confirms the recombination of photo-generated charge carriers through defect levels lying within the band gap. Unlike ideal crystalline MgO, which is an insulator with a wide band gap (~7.8 eV) and typically shows weak luminescence, the nanostructured MgO exhibited enhanced emission due to quantum confinement and surface defect states. The emission peaks were observed in the range of [insert range, e.g., 350–600 nm], indicating the presence of multiple radiative recombination pathways.

The energy of the emitted light was calculated using the photon energy formula:

$$E \text{ (eV)} = 1240 / \lambda \text{ (nm)}$$

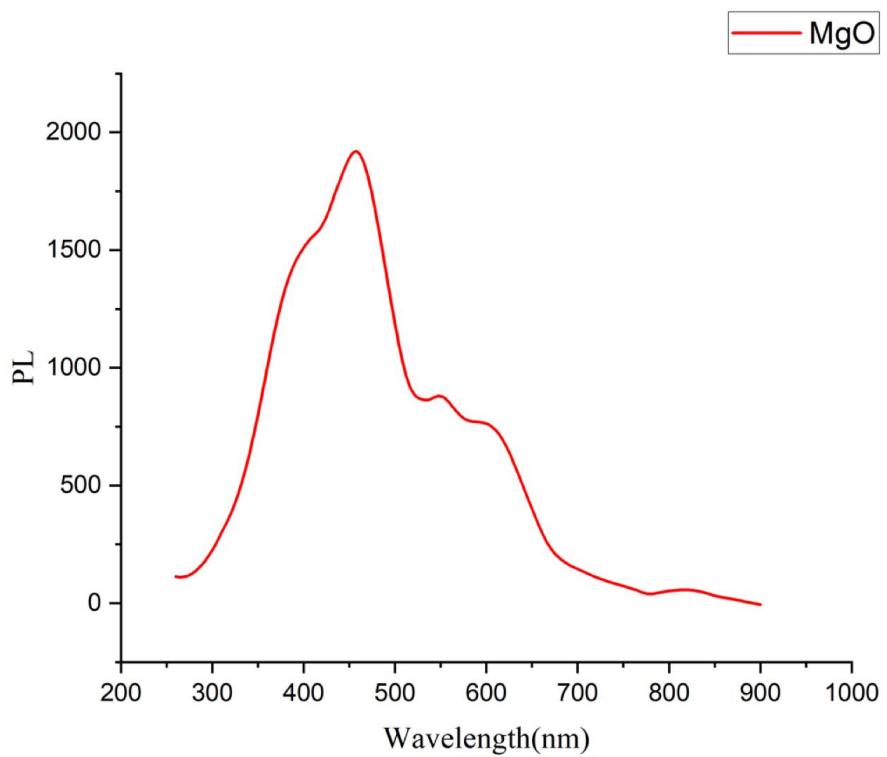


Figure 4.3 Photoluminescence (PL) graph

This analysis provides insight into the defect structure and optical behavior of MgO nanoparticles and complements the results obtained from UV-Visible spectroscopy. The observed luminescence behavior further supports the presence of nanoscale features and defect-induced electronic states in the prepared MgO samples.

The photoluminescence (PL) analysis of the prepared Magnesium Oxide (MgO) nanoparticles was carried out to investigate their optical emission properties and defect-related behavior. Figure [4.3] shows the PL emission spectrum of the MgO nanoparticles recorded at room temperature. A prominent emission peak was observed at a wavelength of 468.37 nm, indicating radiative recombination of photo-generated charge carriers through defect levels present within the band gap. The corresponding photon energy was calculated using the relation:

$$E \text{ (eV)} = 1240 / \lambda \text{ (nm)}$$

$$E = 1240 / 468.37 \approx 2.65 \text{ eV}$$

This emission is attributed to intrinsic structural defects such as oxygen vacancies (F⁻ centers), magnesium interstitials, and surface states, which introduce intermediate energy levels within the band gap of MgO. These defect states facilitate the observed visible-range luminescence, which is otherwise weak in bulk MgO due to its wide band gap (~7.8 eV). The presence of visible emission confirms the nanocrystalline nature and high surface-to-volume ratio of the synthesized MgO nanoparticles, which enhances defect density. The PL results complement UV-Visible spectroscopy by providing evidence of defect-induced optical transitions and confirm the suitability of MgO nanoparticles for optoelectronic and luminescent applications.

CHAPTER - V

SUMMARY AND CONCLUSION

The structural and optical characterization of synthesized Magnesium Oxide (MgO) nanoparticles was carried out using X-ray Diffraction (XRD), UV-Visible spectroscopy, and Photoluminescence (PL) spectroscopy. The results reveal important insights into the crystalline structure, optical absorption behavior, and emission properties of MgO at the nanoscale, confirming the successful synthesis of nanocrystalline MgO with notable defect-related features.

5.1 XRD Analysis

The XRD patterns of the synthesized MgO nanoparticles confirm their polycrystalline nature and nanometer-sized crystallites. The diffraction peaks correspond well to the standard face-centered cubic (FCC) structure of MgO, indicating the formation of a single-phase crystalline material. The average crystallite size, calculated using the Debye-Scherrer equation, was found to be approximately 13.28 nm, indicating the nanoscale dimensions of the particles. Additionally, the average interplanar spacing (d-spacing) was calculated to be 2.106 Å, further supporting the formation of well-defined crystal planes consistent with the cubic MgO structure. The narrow and intense diffraction peaks suggest good crystallinity and minimal amorphous content in the sample. The absence of impurity peaks or secondary phases implies that the synthesis method employed yielded highly pure MgO nanoparticles. The small crystallite size and high surface area are beneficial for various applications, including catalysis, sensing, and electronic devices, where surface activity and defect concentration play a crucial role.

5.2 UV-Visible Spectroscopy Analysis

The optical absorption behavior of MgO nanoparticles was studied using UV-Visible spectroscopy. The absorption spectrum of the MgO nanoparticles exhibited a prominent peak at 260 nm, which is characteristic of the fundamental absorption edge of nanosized MgO. This peak lies in the ultraviolet region, reflecting the wide band gap nature of MgO and its transparency to visible light. The absorption in the UV region can be attributed to the electronic transitions from the valence band to the conduction band, as well as transitions involving defect levels within the band gap. The blue shift in the absorption edge, compared

to bulk MgO, is indicative of the quantum confinement effect, which occurs due to the reduced particle size and enhanced surface-to-volume ratio at the nanoscale. The optical band gap can be further analyzed using Tauc's relation for both direct and indirect transitions. Although bulk MgO exhibits a very wide band gap (~7.8 eV), the nanostructured form can exhibit slightly lower effective band gaps due to defect states. The strong UV absorbance and lack of visible-range absorption reinforce the high optical transparency and purity of the synthesized MgO nanoparticles.

5.3 Photoluminescence (PL) Analysis

The PL spectrum of the MgO nanoparticles was recorded to study their emission characteristics and defect states. A clear emission peak was observed at 468.37 nm, corresponding to a photon energy of approximately 2.65 eV, calculated using the equation

$$E = 1240 / 468.37 \approx 2.65 \text{ eV}$$

This emission lies in the blue region of the visible spectrum and is primarily attributed to intrinsic structural defects such as oxygen vacancies (F-centers) and magnesium interstitials. These defect states introduce localized energy levels within the wide band gap of MgO and act as radiative recombination centers. The relatively strong PL emission indicates a significant presence of surface and structural defects, which is typical for nanocrystalline MgO. Such defects can enhance luminescence and play a crucial role in applications such as photocatalysis, bioimaging, and optoelectronics. The broad and intense PL peak reflects the distribution of defect-related energy levels and confirms that the synthesized MgO nanoparticles possess a defect-rich structure, which is advantageous for various surface-sensitive applications.

The combined results from XRD, UV-Vis, and PL studies confirm the successful synthesis of nanostructured Magnesium Oxide (MgO) with desirable structural and optical properties. The small crystallite size (~13.28 nm) and high purity of the material were confirmed by XRD analysis, while UV-Vis spectroscopy revealed strong UV absorption and confirmed the wide band gap behavior of MgO. Photoluminescence analysis further supported the presence of intrinsic defects responsible for visible-range emissions, particularly at 468 nm (2.65 eV), indicative of oxygen vacancies and surface states. These findings suggest that the synthesized MgO nanoparticles exhibit excellent crystallinity, strong UV absorption, and pronounced defect-related luminescence, making them suitable for a range of technological

applications, including UV-blocking materials, photocatalysis, sensors, and luminescent devices. The study highlights the potential of MgO nanoparticles as a multifunctional nanomaterial and provides a foundation for further exploration of their properties under different synthesis conditions and dopant modifications.

5.4 FUTURE SCOPE

In the present work, the synthesized Magnesium Oxide (MgO) nanoparticles were characterized using XRD, UV-Visible spectroscopy, and photoluminescence (PL) analysis to investigate their structural and optical properties. For a deeper understanding of their physical, chemical, and morphological characteristics, additional characterizations such as Fourier Transform Infrared Spectroscopy (FTIR), Raman spectroscopy, X-ray Photoelectron Spectroscopy (XPS), X-ray Absorption Fine Structure (XAFS), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM) can be carried out. These techniques will help to explore the surface chemistry, bonding environment, morphology, and crystallographic structure of MgO nanoparticles more comprehensively. Furthermore, MgO nanoparticles possess unique electrical, optical, and catalytic properties, making them suitable for a wide range of potential applications. Future work can focus on exploring the dielectric behavior, magnetic properties, and thermal stability of MgO at different annealing temperatures and under various doping conditions. MgO is also known for its excellent performance in gas sensing, photocatalysis, antibacterial activity, and biomedical applications due to its high surface area and defect-induced reactivity. Therefore, there is significant scope to develop and test MgO-based devices, such as gas sensors, UV-blocking coatings, catalytic converters, and antimicrobial surfaces. Additionally, doping MgO with suitable metal ions or combining it with other semiconducting materials can enhance its functional properties, opening avenues for use in optoelectronic devices, energy storage, and environmental remediation. Fabrication of such functional devices using pure and doped MgO nanoparticles could be an interesting direction for future research.

REFERENCES

- [1] Z. Camtakan, S. Erenturk and S. Yusan, Environmental Progress & Sustainable Energy, 2011.
- [2] P.K. Stoimenov, R.L. Klinger, G.L. Marchin, K.J. Klabunde, Langmuir, 18, (2002) 6679.
- [3] R.M. Dickson, L.A. Lyon, J. Phys. Chem., 104 (2000) 6095.
- [4] L. Fu, Z. Liu, Y. Liu, B. Han, P. Hu, L. Cao, D. Zhu, Adv. Mat., 17 (2005) 217.
- [5] S. Music, D. Dragcevic, M. Maljkovic, S. Popovic, Mat. Chem. Phys., 77 (2002) 521.
- [6] M. Singhal, V. Chhabra, P. Kang, D.O. Shah, Mat. Res. Bull., 32 (1997) 239.
- [7] H.Y. Xu, H. Wang, Y.C. Zhang, M.K. Zhu, B. Wang, H. Yan, Cer. Int., 30 (2004) 93.
- [8] G. McDonnell, A.D. Russell, Clin. Microbiol. Rev., 12 (1999) 147-179.
- [9] A. Yadav, Virendra Prasad, A.A. Kathe, Sheela Raj, Deepti Yadav, Sundaramoorthy, N. Vigneshwaran, Bull. Mat. Science, 29 (2006) 641.
- [10] G. Yuvar, Text. Res. J., 78 (2008) 60-71.
- [11] Devaraja, P.B., Avadhani, D.N., Prashantha, S.C., Nagabhushana, H., Sharma, S.C., Nagabhushana, B.M., & Nagaswarupa, H.P. (2014).
- [12] Marwaha, N., Gupta, B.K., Verma, R., & Srivastava, A.K. (2017).
- [13] Venhryny, Y.I., Serednytski, A.S., & Popovych, D.I. (2023).
- [14] Kumar, D., Chikkahumantharayappa, Yadav, L.S.R., Lingaraju, K., Manjunath, K., Suresh, D., Prasad, D., Nagabhushana, H., Sharma, S.C., & Naika, H.R. (2015).
- [15] Janet, C.M., Viswanathan, B., Viswanath, R.P., & Varadarajan, T.K. (2007).
- [16] Devaraja, P.B., Avadhani, D.N., Nagabhushana, H., Prashantha, S.C., Sharma, S.C., Nagabhushana, B.M., Nagaswarupa, H.P., & Daruka Prasad, B. (2014).
- [17] Das, S., Chakrabarti, S., & Chaudhuri, S. (2005).

- [18] Bera, D., Qian, L., & Holloway, P.H. (2008).
- [19] Fatiqin, A., Amrulloh, H., & Simanjuntak, W. (2021).
- [20] Vijayakumar, S., Punitha, V.N., & Parameswari, N. (2022).
- [21] Sulak, M., & Kavaklıoğlu, B. (2022). The green synthesis of MgO nanoparticles using dried jujube fruit extract and their anti-yeast activity against *Saccharomyces cerevisiae*. *Inorganic and Nano-Metal Chemistry*, 52(5), 653–660.
- [22] Hassan, S.E.-D., Fouda, A., Saied, E., Farag, M.M.S., Eid, A.M., Barghoth, M.G., Awad, M.A., Hamza, M.F., & Awad, M.F. (2021). *Rhizopus oryzae*-mediated green synthesis of magnesium oxide nanoparticles (MgO-NPs): A promising tool for antimicrobial, mosquitocidal action, and tanning effluent treatment. *Journal of Fungi*, 7(5), 372.
- [23] Kumar, D., Chikkahumantharayappa, Yadav, L.S.R., Lingaraju, K., Manjunath, K., Suresh, D., Prasad, D., Nagabhushana, H., Sharma, S.C., & Naika, H.R. (2015). Synthesis and characterization of MgO nanoparticles via sol-gel method for antibacterial applications. *AIP Conference Proceedings*, 1665(1), 050130.
- [24] Sharma, G., Soni, R., & Jasuja, N.D. (2017). Phytoassisted synthesis of magnesium oxide nanoparticles with *Swertia chirayaita*. *Journal of Taibah University for Science*, 11(3), 471–477.
- [25] Vidhya, E., Vijayakumar, S., Nilavukkarasi, M., Punitha, V.N., Snega, S., & Praseetha, P.K. (2021). Green fabricated MgO nanoparticles as antimicrobial agent: Characterization and evaluation. *Materials Today: Proceedings*, 45(6), 5579–5583.
- [26] Anupama, A.M., Viswanath, K.V., Kumar, G.A., Kumar, V.P., Ushakiranmayi, M., & Sudhakar, P. (2021). Phytoassisted synthesis of magnesium oxide nanoparticles from *Pterocarpus marsupium* Roxb. heartwood extract and its biomedical applications. *Journal of Genetic Engineering and Biotechnology*, 19, 21.
- [27] Balakrishnan, G., Velavan, R., Raslan, M.B.K., & Emad, H. (2020). Microstructure, optical and photocatalytic properties of MgO nanoparticles. *Results in Physics*, 17, 103013.
- [28] Sharmila, G., Muthukumaran, C., Sangeetha, E., Saraswathi, H., Soundarya, S., & ManojKumar, N. (2019). Green fabrication, characterization of *Pisonia alba* leaf extract

derived MgO nanoparticles and its biological applications. Nanostructures & Nano-Objects, 20, 100380.

[29] El-Sayyad, G.S., Mosallam, F.M., & El-Batal, A.I. (2018).

[30] Suresh, J., Pradheesh, G., Alexramani, V., Sundrarajan, M., & Hong, S.I. (2018). Green synthesis and characterization of hexagonal shaped MgO nanoparticles using insulin plant extract.