International Islamic University Islamabad

Faculty of Basic And Applied Sciences

Department Of Physics

Synthesis, Characterization and Surface Modification Of Magnesium Oxide Nanoparticles

BY

Aqsa Rashid

(322-FBAS/MS PHY/F14)

Dr Shamaila Sajjad

Chairperson

Department Of Physics

Dr. Shamaila Sajjad
Chairperson
Department of Physics (FC, FBAS)
International Islamic University
Islamabad

Dean

International Islamic University Islamabad

Nanotechnology

Nanomaterials

X-ray diffraction

Spectroscopy

Certificate

It is certified that the work presented in this thesis entitled: “Synthesis, Characterization and Surface Modification Of Magnesium Oxide Nanoparticles” has been carried out by Aqsa Rashid (322-FBAS/MS PHY/F14) in laboratory of nanoscience and nanotechnology. This work is fully adequate in scope and quality for the degree of Ms Physics from international Islamic university of Islamabad.

COMMITTEE

External Examiner:

Dr. Nazar Abbas

Associate Professor

Comsats Institute of Information and Technology Islamabad.____________________

Internal Examiner:

Dr.WaqarAdil Syed

Associate Professor

International Islamic University Islamabad. ____________________

Supervisor:

Dr. ShamailaSajjad

Chairperson, Department of physics,

Assistant Professor

International Islamic University Islamabad.____________________

Dr. Shamaila Sajjad
Chairperson
Department of Physics (FC. FBAS)
International Islamic University
Islamabad

Co Supervisor:

Dr.Sajjad Ahmad Khan Lghari

Principle scientist

Pakistan Atomic Energy Commission.____________________

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MS in Physics

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Aqsa

Aqsa Rashid

(322-FBAS/MSPHY/F-14)

DEDICATION

The presented research is dedicated to

My loving parents

And

To the soul of my beloved brother

Bilal Rashid.

ACKNOWLEDGEMENT

I owe my gratefulness to Allah Almighty who has bestowed me with courage and strength for the successful completion of this work.

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Aqsa

Aqsa Rashid

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Abstract

In this work magnesium oxide nanoparticles were successfully prepared via co-precipitation method. Magnesium nitrate was used as a precursor. Different concentrations of silver were doped in MgO. Surface morphology, chemical composition and crystal structure were investigated by SEM, EDX, FTIR and XRD. XRD results showed cubic crystalline nature of prepared sample. SEM showed the rod like morphology of prepared MgO. These rods are agglomerated with each other. SEM showed that the doped silver lies on the surface of MgO in spherical pattern. Anti-bacterial activity of pure MgO and Ag-doped MgO was studied on E-Coli bacteria grown through streaking method. Many colonies of E-coli bacteria were killed by MgO which shows the antibacterial behavior of MgO nanoparticles. It was observed that this anti-bacterial behavior was enhanced by doping silver in MgO. So Ag-doped MgO nanoparticles have better anti-bacterial behavior than pure MgO nanoparticles.

Chapter 1

Introduction

1.1 Introduction to Nanotechnology

Nano comes from a Greek word “dwarf” or “little old man” who means “one billionth” (10^{-9}m)[1] . Nanotechnology may be defined as

“The design, characterization, production and application of structure and devices by controlling their shapes and size at nano meter scale.”

If we suppose that average length of people is 100 nano meters tall, the diameter of moon would be 8 inches and the earth would be 30 inches in diameter.As one nano meter is one billionth of meter. It is very difficult to imagine how small nano meter is. So here are some examples:

- Thickness of a sheet of paper is opproximately 100,000nm.
- One inch has 25,400,000 nanometers in it.
- Width of a human hair is approximately 80,000-100,000 nanometers.
- A human nail grows one nanometer in one second.

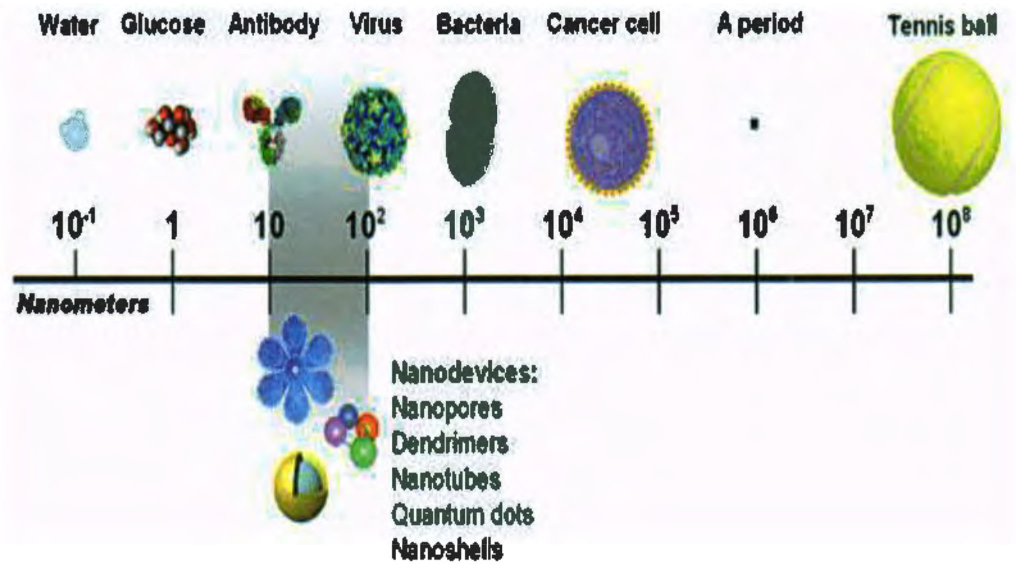


Fig 1.1: A comprison of nanomaterial sizes[2]

Nanotechnology is not new. It is the combination of already existing technologies. The new ability found in nanotechnology is to observe and menipulate at atomic level. This makes nanotechnology so promising from scientific point of view[3].

Range of nanometer scale is 1 to 100 nm , but nano science we study objects which are greater than 100 nm. In many fields of nanotechnology objects are studied in the range of 1-100 nm but in some fields like biochips objects are studied in the range of hundreds of nanometer.

Since 1981 it was not possible to see individual atom, but in 1981 when Scanning tunneling microscope developed we were able to see atom individually. The scanning tunneling microscope (STM) is among a number of instruments that allows scientist to view and manipulate nano scale particles, atoms and small molecules[4].

1.2 Nano material

A nano material is a material having at least one dimension in nanometer range. According to their dimensions nano materials are divided into different categories.

1. Zero dimensional nano materials

2. One dimensional nano materials

3. Two dimensional nano materials

4. Three dimensional nano materials

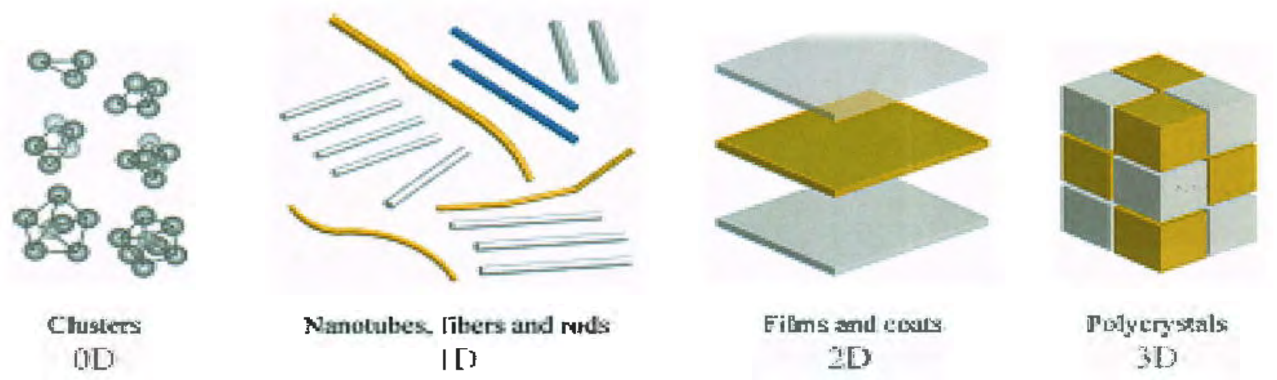


Figure 1.2: Shapes of nanomaterials in differnet dimensions

1.2.1 Zero dimensional nano materials

In these materials all three dimensions are less than 100 nanometers. The most common example of 0-D nano materials are nano particles. Nano particles can be amorphous and crystalline. They appear in many shapes and forms.

1.2.2 One dimensional nano materials

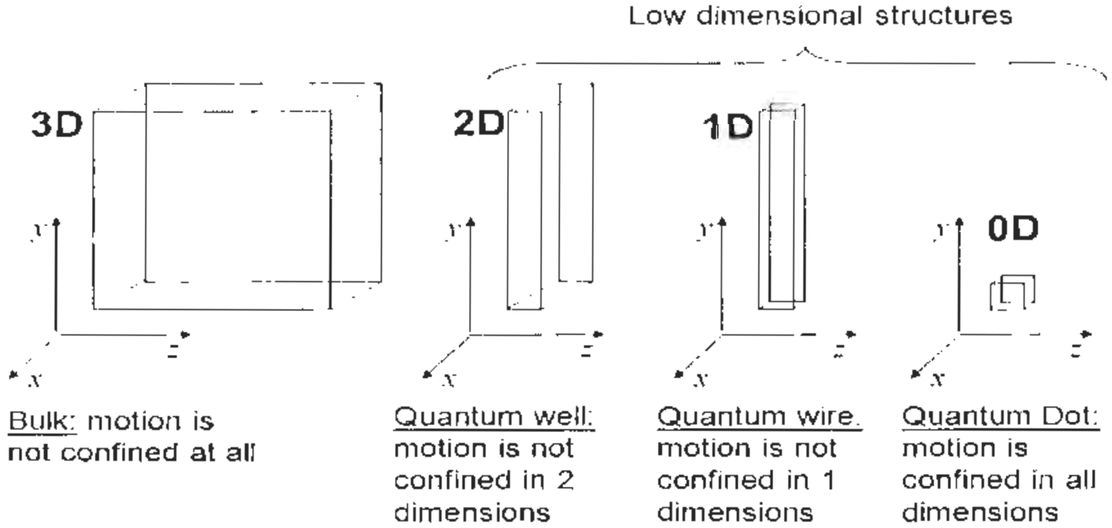
In these materials one dimension is outside the nano scale and two dimensions are less than 100 nm. This lead to needle like shape materials. Examples are nano tubes, nano rods and nano wires. 1-D nano materials can be amorphous or crystalline.They can be chemically pure or impure. They can be metallic, ceramic and polymeric.

1.2.3 Two dimensional nano materials

In these materials two dimensions are outside the nano scale and one dimension is less than 100 nm. Nano films are the examples of these materials. They can possess amorphous and crystalline nature.

1.2.4 Three dimensional nano materials

These materials are not confined to the nano scale in any dimension. 3-D nanomaterials have all three dimensions greater than 100 nanometeres.



When the nano level is reached, the properties like electrical conductivity, color, strength, weight change. When we reach at nano level,the material can change from conductor to semiconductor or insulator.Surface to volume ratio of nano materials is greater than bulk materials.Nano scale material has large surface area than larger scale materials of similar masses. As surface area increases, the number of atoms on the surface increases, large number of atoms can come in contact with surroundings and this increases the reactivity of a material. This increase in reactivity forms nanomatrials better catalyst.

1.3 Formation of Nanomaterials

Formation of nanomaterials at nano level is called nano manufacturing. There are two basic ways to synthesized nano materials.

1. Top down fabrication

2. Bottom up fabrication

1.3.1 Top down fabrication

In this fabrication large materials are break down into smaller material upto nano scale. In this process we require large amount of material because excess material is wasted in this process. This technique is applied for the manufacturing of computer chips.

1.3.2 Bottom up fabrication

In this fabrication we start with atoms and molecules and make larger structure, which can be time consuming. This fabrication can be linked to the building of a brick house. Instead of placing bricks one at a time, atoms or molecules are placed one at a time to build the desired nanostructures.

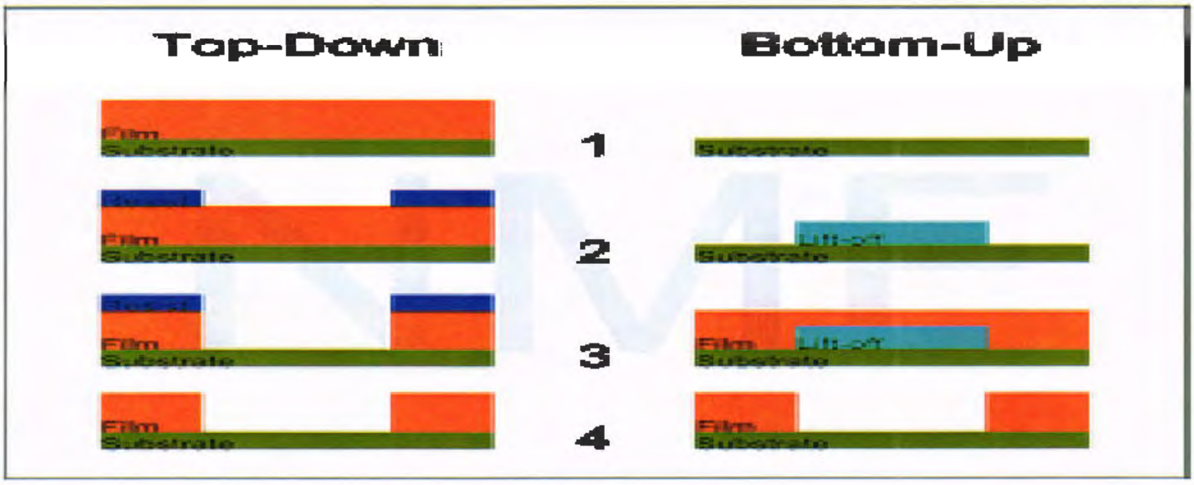


Figure 1.3: Fabrications of nano materials

1.4 Applications of nanotechnology

- In electronics, we can make transistors and integrated circuits by using semiconductor nano wires. Electrically conducting nano ceramics can also be made.
- In catalysis, nanotechnology is used in photo catalyst air and water purifiers. It is used for better activity, selectivity and life time in chemical transformations and fuel cells.
- Magnetic nano particles are used for hyperthermia. We can control drug delivery and drug release by using nanotechnology.
- By utilizing increased energy gap of nano materials we can make window layers in classical solar cells, rechargeable batteries and better thermal or electrical insulation materials. We can make magnetic refrigerators from super magnetic materials.

1.5 Issues and challenges to nanotechnology

• Difficulty in synthesis and isolation

It is very difficult to synthesis nanostructures, nano particles and nano materials as compared to their conventional bulk counterparts. Additionally, it is difficult to control size of the nano particles during synthesis in a solution. Moreover, in polycrystalline nano materials, grain growth may take place.

• Instability of particles

Nano particles, especially metal nano particles are very reactive. They are thermodynamically metastable. The kinetics associated with nonmaterial is very rapid. Owing to high surface area, metal nano particles can even be explosive in direct contact with oxygen. They have poor corrosion resistance. Hence it is very challenging to retain the particle size in nano scale and to maintain the properties of nano materials during intended applications[3].

• Presence of impurities

Nano particles have very active surfaces, leading to absorption and adsorption. Once a species has been adsorbed on the surface of a nano particle the path length required for the diffusion to take the species to the center of the particle is very small. Hence the whole nano particle may easily be contaminated. Thus it is not challenging only to synthesis pure nano particles, but also to keep them pure during their services.

• Biologically harmful

As nano particles are very small so there are many chances that they can be easily anhaled by humans. Most of the nanomaterials are very toxic for humans so they attack on human cells.

Given the lack of data and ease with which nano particles can be redistributed in the environment,so we have to use precuations when we are dealing with nanoparticles.

1.6 Magnesium

Magnesium has atomic number 12. Magnesium has symbol Mg.It's color is grey. Magnesium is the ninth most abundant element in the universe [5]. It can easily dissolved in water [6]. The taste of magnesium ions is sour and in low concentration they help to impart tartness in fresh mineral water. Magnesium has lowest melting point (923K) and lowest boiling point (1363K) of any of the alkali earth metal[7].

Figure 1.4: Physical appearance of Magnesium

Magnesium is a metal. It conducts electricity and it can be bent to form different shapes. Magnesium burns in air. When magnesium burns it forms oxides.Magnesium is very essential element for human body. We use magnesium to produce energy. Magnesium is very necessory for the life of a human cell.

1.7 Oxygen

Oxygen is an element having atomic number 8. It has symbol "O". The name oxygen comes from a Greek word "oxy genes" which means "acid forming". French scientist Antoine Lavoisier[8].Oxygen is a non metal. It is very reactive. When it combines with other elements oxides are produced.[9]. Two atoms of oxygen are combined to form a coloress and odorless diatomic gas with formula O_2. Oxygen is a poor conductor of electricity and beat. It is denser than air. It is fairly soluble in water which makes aerobic life possible in rivers, lakes and oceans.

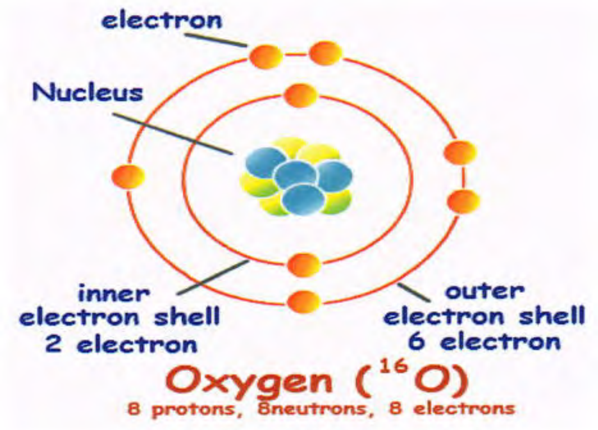


Figure 1.5: Atomic structure of Oxygen O_2

1.8 Oxides

An oxide is a chemical compound typically contains at least one atom of oxygen and one atom of other element [10]. Metal oxides typically contain an anion of oxygen in the oxidation state of -2. There are many metal oxide existed on earth because oxygen oxidize most of the elements in the presence of air and water. Due to its electro negativity, oxygen can form bonds with all elements in order to give oxide. Most metal oxides have polymeric structure.

The oxides typically link three metals or six metals because metal forms chemical bond with oxygen. This is very strong bond that's why oxides are insoluble in solvents.

Oxides can react with acids and bases.

- ### Basic oxides

The oxides that react with acids are called basic oxides. Metal oxides form basic oxides.If soluble in water, they react with water and give hydroxides e.g.

$$CaO + H_2O \text{ ---------}\rightarrow Ca(OH)_2$$

$$MgO + H_2O \text{ ---------}\rightarrow Mg(OH)_2$$

- ### Acidic oxides

Acidic oxides are formed when oxides react with bases. Acidic oxides are the oxides of non metals. They form acids with water e.g.

$$SO_3 + H_2O \text{ ---------}\rightarrow H_2SO_4$$

$$CO_2 + H_2O \text{ ---------}\rightarrow H_2CO_3$$

- ### Amphoteric oxides

Amphoteric oxides are the oxides that can form by the reaction of metals with either acids or bases. They are formed by metalloids (whose properties lie between metals and non metals). When amphoteric oxides react with an acid they form salt and water which is the property of a base. But when they react with alkalis they form salt and water, which is the property of an acid.

e.g.

$ZnO + 2HCl \dashrightarrow ZnCl_2 + H_2O$ (basic nature)

$ZnO + 2NaOH \dashrightarrow Na_2ZnO_2 + H_2O$ (acidic nature)

- **Neutral oxides**

Neutral oxides neither show acidic properties nor basic so do they not form salt when, they react with acids or bases.

Metal oxides have great importance in several fields of Physics, Chemistry and material sciences. Elements of metals combine with oxygen in order to give oxides of metals. Oxides nano particles have unique physical and chemical properties due to their limited size and high density of corner and edge surface sites. Among the metal oxides magnesium oxides (MgO) is widely used in chemical industry. It can be used as a scrubber for the gasses which pollute the air. It can also be used as catalyst. MgO exhibit a rock salt structure just like the oxides of alkaline earth metals. MgO particles have cubic shape. When we burn magnesium metal in the presence of air, cubic shape magnesium oxide smoke particles are formed [11].

1.9 MAGNESIUM OXIDE

Magnesium oxide or magnesia is a white solid mineral. Magnesium oxide is formed by the combination of magnesium and oxygen through ionic bonding. Magnesium gives two electrons to oxygen. Magnesium starts with 10 outermost electrons and oxygen starts with 6 outermost electrons. When magnesium gives 2 electrons to oxygen we end up with 8 electrons on each.

$$Mg + O_2 \longrightarrow MgO$$

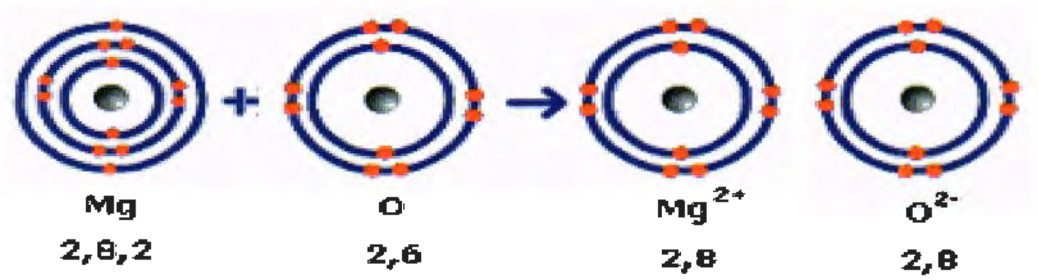


Figure 1.6: Electronic configuration of Magnesium oxide

It is an alkaline earth metal oxide. In MgO Mg^{2+} ions and O^{2-} ions are combined by ionic bondings. When magnesium oxide reacts with water magnesium hydroxide is formed. We can also produce magnesium oxide by heating magnesium hydroxide.

Magnesium oxide can also be synthesized by the calciming the magnesium oxide or magnesium carbonate. MgO can also be produced by the treatment of magnesium chloride with lime followed by heat. . Other important sources of MgO are seawater, underground deposits of brine and deep salt beds from which magnesium hydroxide $Mg(OH)_2$ are processed.

Figure 1.7: Crystal structure of MgO

1.10 Physical properties

- Magnesium oxide is hygroscopic. When hygroscopic substances are exposed to air, they absorb water molecules spontaneously.
- At room temperature magnesium oxide appears in white powder form.
- Magnesium oxide is very basic. It's PH is 10.3. If there is acidity in stomach, magnesium oxide neutralizes acid in stomach.
- Solubility of magnesium oxide in water is very low.
- Magnesium oxide has an extremely high melting point 5072 degrees Fahrenheit and high boiling point 6512 degrees Fahrenheit.
- Magnesium oxide has good corrosion resistance.
- It has high thermal conductivity and low electrical conductivity.
- It has transparency to infrared.

1.11 Magnesium oxide nano particles

Magnesium oxide nano particles appear in white powder form. Magnesium oxide is a combination of magnesium of block S, period 3 elements and oxygen of block P, period 2 elements. Magnesium oxide nano particles are non toxic. They are odorless. They are very hard. They have high melting point and high purity. They have high band gap. They are very corrosive. Chemically they are very reactive. Magnesium oxide nano particles have attracted great attention because of their remarkable properties such as good mechanical strength, high conductivity conductivity[12].

1.11.1 STRUCTURE

Magnesium oxide has the same structure as sodium chloride (NaCl). But sodium chloride is made up of single Na^+ and Cl^- ions and magnesium oxide is made of Mg^{2+} and O^{2-} ions. So there is a great attraction between these ions. This means to separate these ions we require a large amount of energy. Due to this reason magnesium oxide has high melting point.

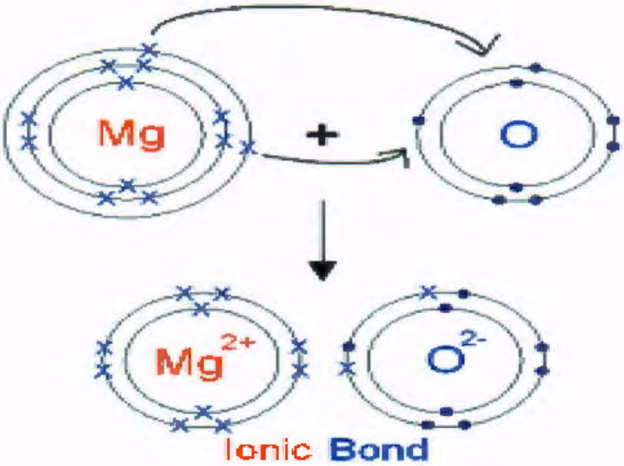


Figure 1.8:Atomic Structure of MgO

Nano crystalline magnesium oxide are highly reactive because of these two properties.

- Large surface area
- Large number of defects on the surface, these surface defects increase the reactivity[13].

1.12 Applications of magnesium oxide nano particles

Magnesium is an important inorganic material having large band gap. It can be used in electronics, heating apparatus, ceramics, refractory material and lots of other fields [14].

The potential applications of magnesium oxide nano particles are as follows:

⦿ High temperature dehydrating agent

As a high temperature dehydrating agent magnesium oxide is used for the production of silicon sheets and electronic industry materials.

⦿ Electric insulating material

As an electric insulating material magnesium oxide is used to make crucible, smelter, electrode bar and electrode sheet.

⦿ Fire retardant

As a fire retardant magnesium oxide nano particles are used for chemical fibers and plastic trades.

⦿ Refectories

Magnesium oxide nano particles are widely used in steel industry. It is used to make refectory materials, refectory coatings, electricity, cable, optical material, material for steel-smelting furnace and other high temperature furnaces.

⦿ Chemical industry

Magnesium oxide nano particles are widely used in chemical industry as a scrubber for air pollutant gasses (CO_2, NO_x, SO_x).

⦿ Medical field

As magnesium oxide is a base and it reacts with acid in stomach and nuetralizes it. SO magnesium oxide nanoparticles can be used for the treatment of acidity in stomach and heart burn. Acid present in pages of books rot the pages. MgO reacts with this acid and nutralizes it.

⦿ Catalyst

Magnesium oxide nano particles are used as catalysts or for catalytic support. As the particle size decreases, the relative number of surface atom increases and thus their activity increases[15].

1.13 Antibacterial activity of MgO nano particles

As we all know there are several diseases caused by bacteria.And these diseases causes millions of humans deaths. So we have to control bacterial population. To control this population of bacteria we have to produce an antibacterial agent. Antibacterial agent may be organic or inorganic. Inorganic anti bacterial agents are more important than organic antibacterial agents.Some of the antibacterial agents are metal oxides like TiO_2, ZnO, MgO and CaO. Among of them MgO is more important,because it remains stable during its production and it is safe for human body.It is also low cost and environmentally friendly material[16].

It is considered that oxide ions are the basic fector for the antibacterial behaviour of magnesium oxide nanoparticles. When surface area increase, quantity of oxide ions increases.So MgO nano particles exhibit stronger anti bacterial activity as compared to common MgO[17].

1.14 Silver nano particles

Silver nano particles are the nano particles of silver lies in the range of 1nm to 100nm. Silver nano particles exhibit numerous shapes depending on the application at hand. Spherical Silver nano particles are commonly used but diamond; octagonal thin sheets are also popular. These nano particles appear in different colored powder, depending on their size and morphology. Silver nano particles can be dissolved into different solvents like water and ethanol. Silver nano particles have unique optical, electrical and thermal properties. Silver nano particles also have anti-inflammatory properties. Silver nano particles are very effective antibacterial agents for both aerobic and anaerobic bacteria. In medicines Ag nano particles are used for diagnostic and therapeutic purposes. They can be used as air and water purification, food preservation chemical vapor sensors and catalysts.

1.15 Aims and objectives of the work

Aims and objectives of my MS work are:

- To synthesize Magnesium oxide nano particles by co-precipitation method.
- Doping of silver in MgO at different concentrations.
- To characterize the structure and morphology of as-prepared Magnesium oxide and Ag/MgO nano composites, with different characterization techniques such as Scanning Electron microscopy(SEM), Energy Dispersive Spectroscopy(EDS),Fourier Transform Spectroscopy(FTIR) and X ray diffraction(XRD).
- To examine the anti bacterial activity of as prepared Magnesium oxide nano particles and silver doped MgO nanoparticles.

Chapter 2

2.1 Literature review

D.Minakshi et al. Synthesized MgO nano particles by adopting quick precipitation technique assisted by PVP. They take 50ml 0.5M $Mg(NO_3)$ and PVP solution in 250ml flask and kept for constant stirring for 5-10 minutes. Then 50ml of 1M solution of $NaHCO_3$ was slowly added into the above solution the whole process was done under constant stirring. After the addition of surfectants and precipitating agent solution was allwed to stirrer for 3h without changing any parameter. After the completion of whole process white precipitates of MgO was settled at the bottom of the flask. Then MgO in powder form was carefully separated from surfectants liquid. The net substrate was kept under calcinations at 350°C, to obtain in the desired nano crystals of MgO at 20nm thickness. The XRD and TEM results indicate that the obtained MgO nano particles have crystalline structure. It can be seen from SEM and TEM images that the Size of MgO are 20nm thick and structure is non porous. It was also confirmed that use of magnesium nitrate with PVP is a good method for the fabrication of MgO nano particles [18].

H mirzai and A davoodinia et al. used a sol gel process for the fabrication of magnesium oxide nanoparticles. The precursor used in this process was magnesium nitrate. By taking magnesium nitrate as a salt, ammonia solution was used as precipitating agent. The gel of magnesium hydroxide was kept in oven at 350w for 5 minutes. The temperature of precipitates was increased up to 80°C. Then resulting precipitates were cooled down at room temperature. Then they washed the sample with deionized water. Then they dried sample at 120°C. Morphology of magnesium oxide nanoparticles was studied by SEM. The crystal structure was analyzed by XRD. XRD revealed that prepared magnesium oxide has cubic shape with size 10nm[19].

Rizwan Wahab et al. prepared magnesium oxide nanoparticles having cubic shape. They used sol-gel process for this manufacturing. Magnesium nitrate was used as precursor. Magnesium nitrate was used with sodium hydroxide. they dissolved magnesium nitrate in deionized water. 0.5M NaOH was added drop by drop in above solution. After few minutes white precipitates of magnesium hydroxide were appeared at the bottom. Then this solution was washed with methanol more than three times. Hydrated magnesium oxide nanoparticles were dried at room temperature and calcined in air at 300 and 500^{0}C. SEM results showed that prepared magnesium oxide nanoparticles have size in the range 50nm to 70nm. XRD indicated that the obtained nanoparticles have good crystallinity. FTIR spectroscopy was used for analyzing the powder composition[20].

P.Ouraipryvan et al. synthesized highly crystalline MgO nanoparticles with mesoporoys-assembled structure by sol gel process. First a specific amount of analytical grade acetylacetone (ACA) was introduced into magnesium methoxypropylate in methoxypropanol (MMP). Then this solution was homogenized by shaking. Then 0.1 M laurylamine hydrochloride was mixed in the first solution. Then this solution was stirred for one day. This was done for the aging of particles. The gel was dried at a temperature of 80^{0}C. After dryig calcination was done at 500^{0}C. XRD, TEM and SAED analysis showed that the synthesized MgO possessed high crystallinity with particle size in nano size range. N_2 adsorption – desorption and pore size distribution was in narrow mesoporous range, which originated from an assembly of the MgO nanoparticles. The XRD and SAED showed that magnesium oxide nanoparticles was successfully prepared. Their size was in nano range[21].

Fares Khairallah et al. synthesized magnesium oxide nanoparticles using "aqueous wet chemical method". In this paper Interaction of MgO was studied with CO. XRD pattern showed the cubic structure of MgO nanoparticles. Crystalline sizes evaluated by Scherrer's formula were around13-18nm. XPS analysis showed the homogeneity of particles. DRIFT spectroscopy is used to study the reactivity of MgO nanoparticles. To study the catalytic activity of MgO nanoparticles they use CO and methanol.[22].

K.Ganapati Rao et al. prepared MgO nano particles by using magnesium nitrate as core precursors by co-precipitation method. Magnesium nitrate and sodium hydroxide soultion were prepared separately and then they are combined by adding drop wise sodium hydroxide solution in magnesium nitrate solution under constant stirring. Then white precipitates of MgO are appeared. After washing, drying and calcination MgO nano particles were obtained. These Nanoparticles have been characterized by XRD, PSA, SEM and TG-DTA for average crystalline size, average particle size morphology and thermal stability respectively. XRD showed that the obtained MgO nanoparticles are of cubic shape. Their size was about 18nm[23].

Yuanyuan Rao et al synthesized MgO nanoparticles by using sol gel method. Doping was done by using different ions lithium, zinc and titanium. doped with different ions.

Magnesium nitrate and dopant was dissolved in deionized water. At the same time gel of 0.02 mol of C6H8O7·H2O was made. Then this gel was kept in oven at 150°C.Then white precursor was obtained. Then precursor was crushed and heated at 600°C for 2h to attain doped MgO Nano powder. This sample was characterized by XRD, TEM and XPS. E. coli bacteria was used as a sample to study and compare the anti-bacterial behavior of different doping concentrations. The results show that Li-doped MgO exhibits better antibacterial activity[24].

Rajesh Kumar et al synthesized MgO nanocrystals by using co-precipitation method in which ammonia is used as co-precipitating agent. 0.1M of Mg(NO3) was dissolved in 100ml of distilled water. Then sodium hydroxide solution was added in drops into the first solution. Then solution was kept for 24 hours and then washed to remove impurities. After drying nanoparticles were calcinated at 600°C in air for 4h and 6h to transform into MgO. XRD showed that Mg $(OH)_2$ nanocrystals are polycrystalline in nature and after annealing at 600°C it transforms into MgO nanoparticles. TEM images of MgO nanoparticles show flake like structure. UV visible spectroscopy is used to estimate the optical band gap energy of MgO nanocrystals and found to be 5.92 ev[25].

Zhen-Xing et al studied the anti-bacterial behavior of MgO nanoparticles. As bacterial pollution is risk for human health. So antibacterial behaviour of magnesium oxide nanoparticles was studied in this paper and it was confirmed that MgO has good anti-bacterial behaviour[26].

Agrawal R.M et al prepared MgO nano particles by using $Mg(NO_3)_2$ and NaOH as precursors through liquid phase method. Firstly, they dissolved 5.2gm of Magnesium Nitrate Hexahydrade into 200ml of distilled water. Another solution of sodium hydroxide was made. Then this solution was added in the above solution in drops. After hat, solution was kept uder constant stirring and then kept for 2h on table gore aging of the particles. White precipitates were appeared at the bottom of the beaker. This solution was washed with distilled water and ethanol. Then the powder was dried and crushed and calcined at 400°C for 4h. XRD analysis showed the formation of FCC MgO nanoparticles [27].

Chandrappa K.G et al. synthesized disc like Nano sized MgO by solution phase method. Magnesium chloride and sodium hydroxide were used in ehanol medium. Prepared MgO nanopartilces were characterized by SEM,XRD,UV Visible and FTIR. It was found by XRD that prepared MgO nanoparticles were well crystalline. SEM shows granules, triangular, hexagonal and disc like shaped particles having 17-35nm size. It was observed that solution phase method was environmental friendly and efficieny method for genreation of MgO nanoparticles[28].

Hongmei Cui et al. synthesized Pure magnesium oxide nanoparticles and copper doped magnesium oxide nanoparticles by using co-precipitation method. A solution of copper acetate was made in ethanol. 0.2mol of Mg(NO3)2_6H2O was dissolved into ethanol. At the same time another solution of 0.2mol of (COOH)2_2H2O was made in ethanol. These three solution was mixed together in order to obtain a green precurser under constant stirring of 30 minutes.Then precurser was kept for 12h.After that this green precurser was washed with ethanol and then dried. And then by calcination at 500°C,nanosized MgO was prepared.TEM was used to examined the size and morphology of sample. Crystal structure was examined by XRD. XRD results revealed by doping copper the distance between the planes of magnesium oxide nanoparticles of (200) face is changed. XPS showes the perfect doping of copper into magnesium nitrate.The antibacterial property of MgO was investigated on E.Coli bacteria[29].

N. Badar et al. studied that how calcinations time effects on the band gap energies of MgO nanoparticles. MgO nanoparticles were synthesized via sol-gel method. Magnesium acetate tetrahydrate Mg(C2H3O2)2.4H2O was dissolved in Ethanol.Then to make solution gel like, tarteric acid was added into the solution until PH 6. Then after heating a white precurser of MgO was obtained. Prepared MgO nanoparticles were calcined at the temperature of 600°C for 24 hours. XRD is used for sample characterization.SEM is used for studying the morphology of sample. UV-Visible spectroscopy is used to study the band gap energy of the material. Results showed that band gap energy increases with longer heating time[30].

M.SUNDRARAJAN et al. synthesized MgO nanoparticles using wet chemical method under different calcination temperature. Magnesium nitrate,sodium hydroxide and starch were used for the preparation of magnesium oxide nanoparticles.starch was used as stabilizing agent. It also prevents from the agglomaration of particles. Starch solution was made in distilled water and then 12.83gm of magnesium nitrate was mixed into the first solution. Then solution was stirred untill the whole magnesium nitrate is dissolved. Then 4gm sodium hydroxide(25ml) soultion was added into the above soution drop wise under constant stirring. Then soution was kept for 24h for agging of the particles. Then the solution was centrifuged and washed to remove the impurities. The nanoparticles of magnesium hydroxide

Chapter 3

Experimental method

3.1 Chemicals

Table 3.1: Chemicals used in the synthesis

Names	Formulas	Molar mass
Magnesium nitrate	$Mg(NO_3)_2$	256.41 g/mole
P-123	$HO(CH_2CH_2O)_{20}(CH_2CH(CH_3)O)_{70}(CH_2CH_2O)_{20}H$	3809.77273 g/mole
Sodium hydroxide	NaOH	39.997 g/mole
Ethanol	C_2H_6O	46.06844 g/mole
Silver Nitrate	$AgNO_3$	169.872 g/mole

3.2 Instruments

The instruments used in the synthesis of silver doped magnesium oxide are:

- Beakers
- Measuring cylinder
- Conical flask
- Magnetic stirrer
- Stirring plate
- Hot plate
- Weighing balance
- Calcinations furnace
- Petri dishes

3.3: Synthesis method of MgO nano particles

- First of all I took 0.5gm of P-123 measuring in the weighing balance in 10ml of Ethanol and stir for 1 hour.

Figure 3.1: Solution of P123 in ethanol

- Then I add 10gm of Magnesium nitrate into the above solution under constant stirring until the whole Magnesium nitrate is dissolved into the solution and named this solution as sol1.

triangular, hexagonal and disc like shaped particles having 17-35nm size. It was observed that solution phase method was environmental friendly and efficieny method for genreation of MgO nanoparticles[28].

Hongmei Cui et al. synthesized Pure magnesium oxide nanoparticles and copper doped magnesium oxide nanoparticles by using co-precipitation method. A solution of copper acetate was made in ethanol. 0.2mol of Mg(NO3)2_6H2O was dissolved into ethanol. At the same time another solution of 0.2mol of (COOH)2_2H2O was made in ethanol. These three solution was mixed together in order to obtain a green precurser under constant strring of 30 minutes.Then precurser was kept for 12h.After that this green precurser was washed with ethanol and then dried. And then by calcination at $500^{o}C$,nanosized MgO was prepared.TEM was used to examined the size and morphology of sample. Crystal structure was examined by XRD. XRD results revealed by doping copper the distance between the planes of magnesium oxide nanoparticles of (200) face is changed. XPS showes the perfect doping of copper into magnesium nitrate.The antibacterial property of MgO was investigated on E.Coli bacteria[29].

N. Badar et al. studied that how calcinations time effects on the band gap energies of MgO nanoparticles. MgO nanoparticles were synthesized via sol-gel method. Magnesium acetate tetrahydrate Mg(C2H3O2)2.4H2O was dissolved in Ethanol.Then to make solution gel like, tarteric acid was added into the solution until PH 6. Then after heating a white precurser of MgO was obtained. Prepared MgO nanoparticles were calcined at the temperature of $600^{o}C$ for 24 hours. XRD is used for sample characterization.SEM is used for studying the morphology of sample. UV-Visible spectroscopy is used to study the band gap energy of the material. Results showed that band gap energy increases with longer heating time[30].

M.SUNDRARAJAN et al. synthesized MgO nanoparticles using wet chemical method under different calcination temperature. Magnesium nitrate,sodium hydroxide and starch were used for the preparation of magnesium oxide nanoparticles.starch was used as stabilizing agent. It also prevents from the agglomaration of particles. Starch solution was made in distilled water and then 12.83gm of magnesium nitrate was mixed into the first solution. Then solution was stirred untill the whole magnesium nitrate is dissolved. Then 4gm sodium hydroxide(25ml) soultion was added into the above soution drop wise under constant stirring. Then soution was kept for 24h for agging of the particles. Then the solution was centrifuged and washed to remove the impurities. The nanoparticles of magnesium hydroxide were calcined at 300°C,500°C and 700°C for 4h. SEM results showed that with increase in calcination temperature,particle size increases. It was observed from XRD that by increase in calcination time peaks becomes sharp which showes that crystallinity increase. The particle size was calculated as 4.6nm, 8.5nm and 13.3 nm w.r.t 300°C, 500°C and 700°C. Gram positive and gram negative bacteria were used to examine the anti bacterial behaviour of magnesium oxide nanoparticles. Antibacterial activity reduces with increase in calcinations temperature[31].

Alexander F.Bedilo et al. prepared magnesium oxide nanoparticles coated with carbon by usnig two different twchniques. A porous coating of carbon is formed on the surface of MgO nanoparticles.Resorcinol was found to be efficient agent. Inert gas was also used to prepare magnesium oxide nanoparticles.In this paper it was observed that overall stability of the material in the prescence of water is improved as compared to the non-coated noncrystalline MgO. With minor modification this method can also be used for the preparation of a wide veriety of carbon caoted metal oxides [32].

Chapter 3

Experimental method

3.1 Chemicals

Table 3.1: Chemicals used in the synthesis

Names	Formulas	Molar mass
Magnesium nitrate	$Mg(NO_3)_2$	256.41 g/mole
P-123	$HO(CH_2CH_2O)_{20}(CH_2CH(CH_3)O)_{70}(CH_2CH_2O)_{20}H$	3809.77273 g/mole
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- Petri dishes

3.3: Synthesis method of MgO nano particles

- First of all I took 0.5gm of P-123 measuring in the weighing balance in 10ml of Ethanol and stir for 1 hour.

Figure 3.1: Solution of P123 in ethanol

- Then I add 10gm of Magnesium nitrate into the above solution under constant stirring until the whole Magnesium nitrate is dissolved into the solution and named this solution as sol1.

Figure 3.2: Addition of Magnesium Nitrate

- Then in a separate beaker I mixed 2gm of NaOH in 25ml of Ethanol and stir this solution for 1 hour and named this solution as sol2.

Figure 3.3: Solution of NaOH in Ethanol

- After complete dissolution NaOH solution (sol2) was added in drops to the sol1 with constant stirring for 4 hours.

Figure 3.4 Mixing of Solution1 and solution2

- Then this solution was kept for 48 hours for the settlement of particles.
- Then I dried this solution. White powder form is appeared.
- Then solution was centrifuged and washed to remove by products. For washing I added this powder into the distilled water and stir then washed. I repeated this process 5 times. Then I gave washing to this powder 3 times using ethanol. So that all impurities were completely removed.

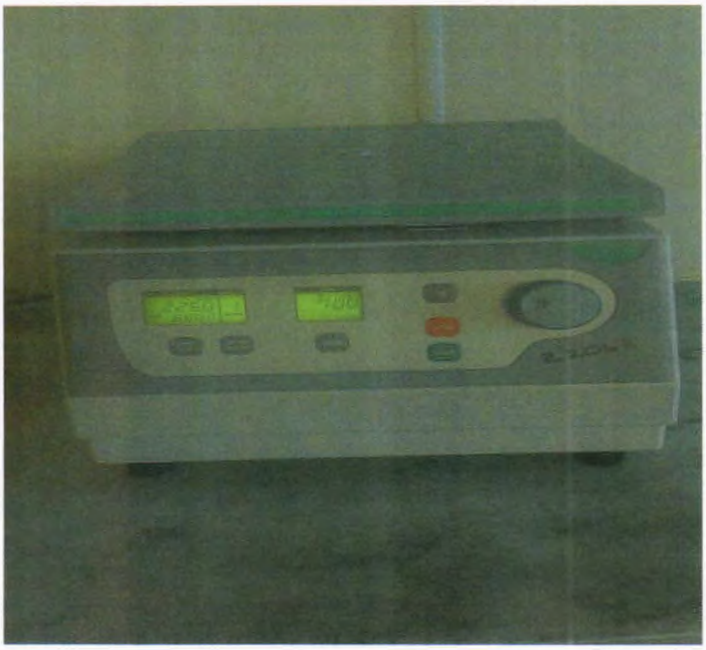


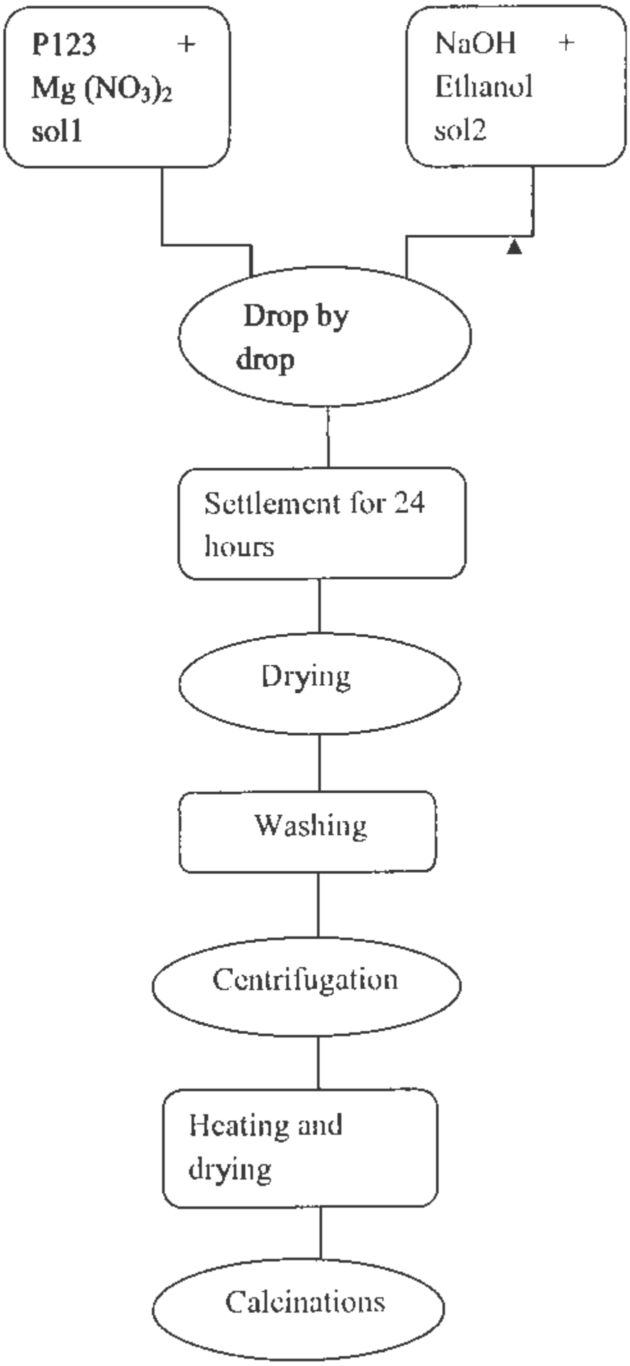
Figure 3.5: Centrifugation of sample

- Then this wet powder was kept on hot plate. When the moist was removed the sample appear in white powder form.

Figure 3.6 :Drying and crushing of sample

- Calcination was done at 500°C for 5 hours to obtain MgO nanoparticles.

3.3 Flow chart of synthesis method



3.4 Co precipitation method

In this thesis co-precipitation method is used. As co-precipitation method is simple and it has efficient applications, so it is used at large scale. Due to it's simplicity and better control on particle size co-precipitation method is usually preffered[33]. In this reaction concurrent existance of nucleation growth, agglomaration and coarsening is involved[34-35]. In co-precipitation method precipitates of metals are formed[36].These precipitates are dealed at suitable temperature in order to get nanoparticles.As every synthesis method require its own limitations.Co precipitation requires controlling PH, temperature, concentration of solution and stirring speed of the mixture[37].

3.5 Doping process

For doping process two concentrations of $AgNO_3$ were prepared in ethanol solution. First I take 0.05gm of silver in 10ml of ethanol and stir for one hour. Then add 1gm of MgO into this solution and stir for 1 hour. The whole process is repeated for 0.15 gm of $AgNO_3$. After washing these samples were heated and then dried.

Figure 3.7:Doping of $AgNo_3$ in MgO

3.6 Preparation of Bacteria

3.6.1 Preparation of media

First of all I weighed 6.3 gram nutrient agar in weighing balance and then added it into 100ml of distilled water and shake it well for 15 minutes so that all nutrient agar is dissolved into distilled water. Then this solution is covered carefully in order to avoid contaminations and placed it in an autoclave at 121^{0}C and 15lb pressure for 1 hour. Petri dishes and a loop is also placed in autoclave along with this solution. After autoclaving, media is cooled down.

3.6.2 Bacteria formation

For bacteria formation, I placed nutrient agar in two petri dishes. Then from another petri dish in which the media of E.Coli bacteria is present, I used this media with the help of loop and streak it into both petri dishes in which nutrient agar media is already present. This whole process is called streaking method for the growth of bacterial culture. After streaking media is cooled down and kept in an incubator for 48 hours. After 48 hours colonies of E. coli bacteria are grown and they were ready for experiment.

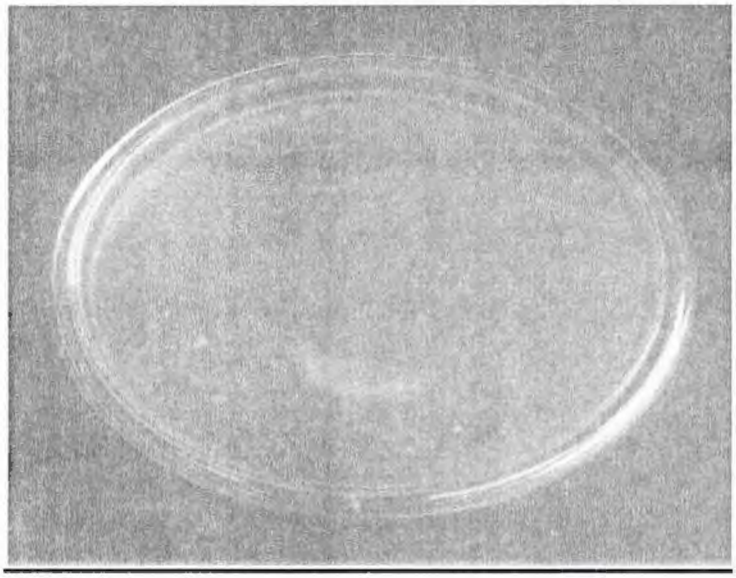


Figure 3.8 Sample 1 for Anti bacterial activity

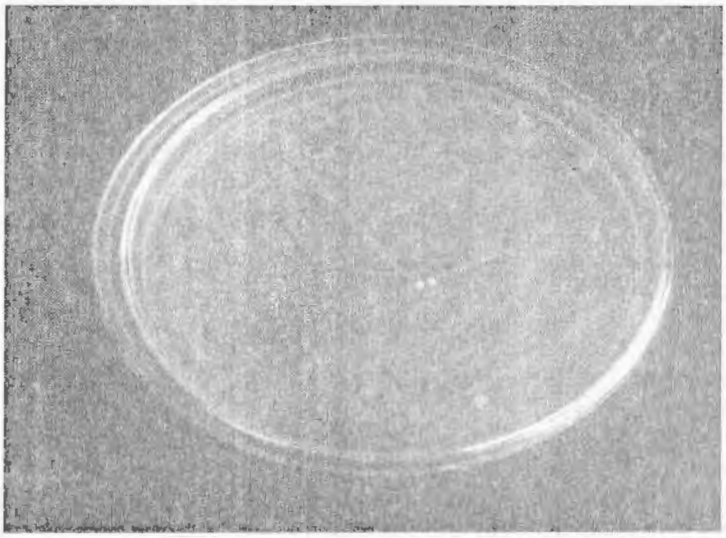


Figure 3.9 : Sample 2 for Anti becterial activity

3.7 Characterization techniques

In characterization techniques we study the material's features like its composition, structure and many physical properties and gain the knowledge that what is the distribution of elements in material. These techniques can be spectroscopic or microscopic. In microscopic techniques electrons, photons or ions can he used to gather data about the structure of sample. It also includes magnification of internal structure of material. Spectroscopic techniques involve interaction of ultraviolet, visible and infrared radiations with matter to study the chemical composition, composition variation, crystal structure and photoelectric properties of materials.

3.7.1 X ray Diffraction

X ray Diffraction is a characterization technique used for

- Determination of unit cell dimensions
- Measurement of sample purity
- Physical properties of material and thin films because the physical properties depends on the atomic arrangement within the matel.
- Measurement of average distance between atomic layers and particle size[38].
- Determination of structure of crystal and spacing between atoms.

Through this technique crystalline and non crystalline materials can be differentiated. The XRD pattern of each material is different and will never match with the XRD pattern of the other material just like the finger prints of humans. So XRD can be considered as the finger print of crystalline materials. The d spacing should be comparable to the wavelength of incoming x rays and structure of material should be geometrical in order to obtain the XRD pattern.

- **Fundamental principle of x ray diffraction**

In X ray diffraction constructive interference occure between monochromatic X rays and crystalline sample. These X rays are generated by cathode ray tube. Then filtaration of these X rays was done to obtain monochroamtic X rays. Cathode ray tube generates these x rays and then these X rays are filtered to produce monochromatic radiations and then they are concentrated by collimator and then finally directed towards the sample. These X rays interact with sample and produces constructive interference. This condition satidfies the Braggs Law.The mathematical form of Bragg's law is $n\lambda=2d\sin\theta$. These x rays which are diffracted are detected. The powder material is rendomly oriented and all diffrected directions are attained. As each materail has it's own petteren of d spacings. These d spacing is compared with standered referance patteren.

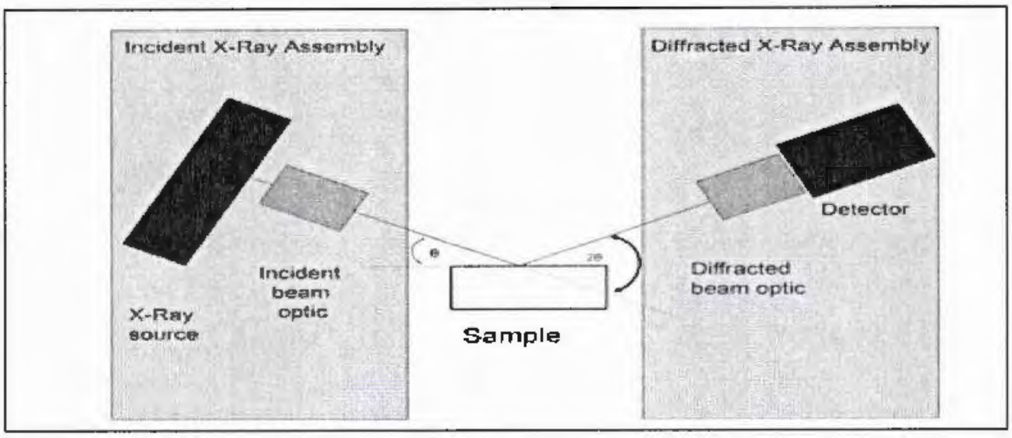


Figure 3.10 Illustration of XRD

- **Bragg's Law**

Bragg's law shows a relationship between incident wavelength, angle of incidence and inter planner spacing's. For X rays the following condition must be satisfied

$$n\lambda = 2d\sin\theta$$

Where

"n" is the number of integer, "λ" is the wavelength of incident x ray, "d" is the inter-planer spacing and "θ" is the scattering angle.

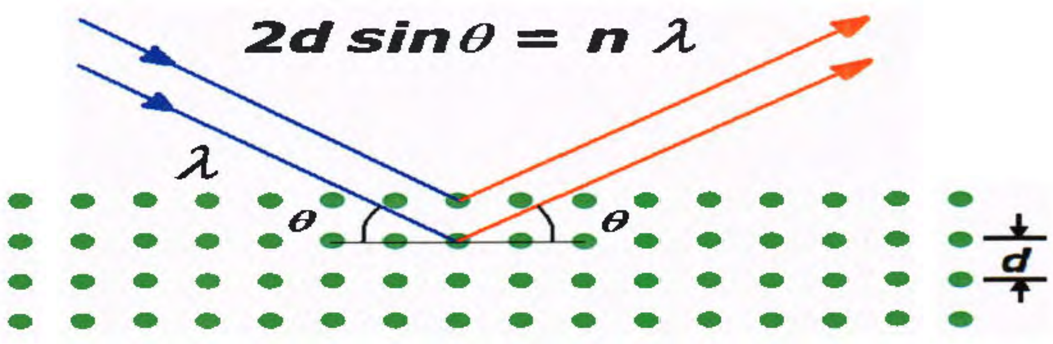


Figure 3.11: Bragg's law

3.7.2 Diffraction Methods

There are three special methods for the diffraction according to the arrangement of parameters

- Laue method
- Powder method
- Rotating crystal method

3.7.3 Laue Method

Orientation and nature of a material was investigated by using Laue method [40]. In this process sample was kept fixed by keeping the sample fixed wavelength of X rays is varied until the Bragg's condition for constructive interference is satisfied.

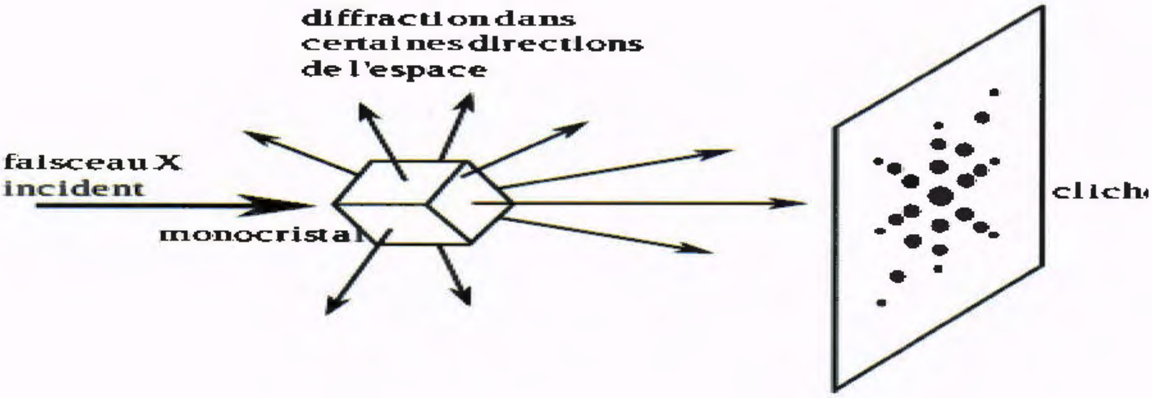


Figure 3.12: Laue method

3.7.4 Powder method

Powder method is used for analyzing the crystal structure. First of all the crystal which is to be examined is crushed into a fine powder and then an X ray beam is bomborded onto it. In this method each and every particle in powder behaves like a small crystal. In powder method, every set of planes have ability to show the reflection phenomena[40].

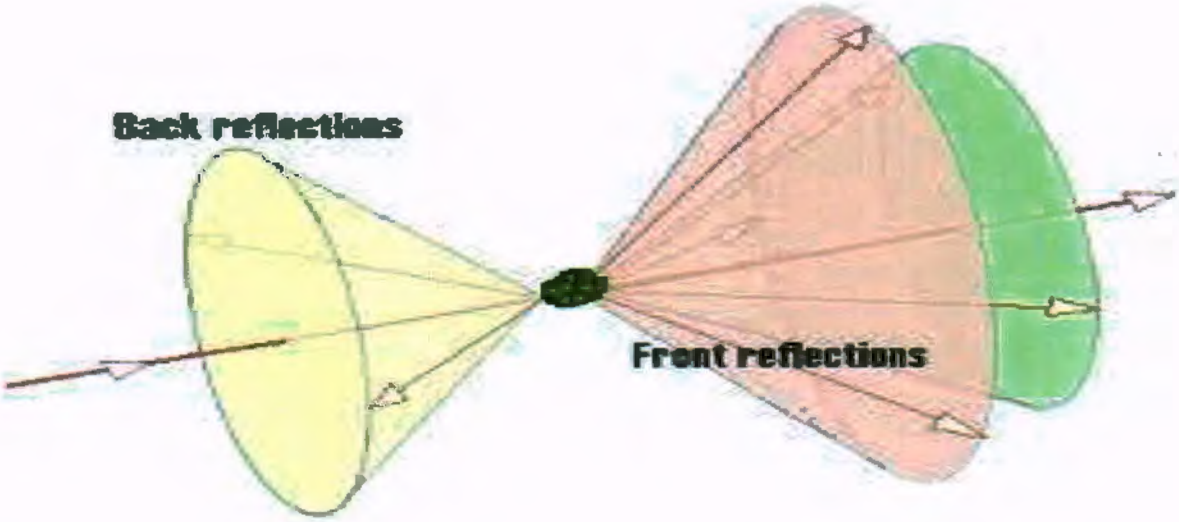


Figure 3.13 Powder method

3.7.5 Rotating crystal method

Crystal structure of a material is studied by using rotating crystal method. Wavelength of incident X rays is kept constant and incident angle is change by rotating the sample. Crystal which is to be examined is kept on one of it's axis.Film is placed around the sample. Then x ray beam is incidented perpendicularly on the sample. As the crystal rotates, sets of lattice planes will make correct Bragg's law at some point and at that point a diffracted beam will be formed.[41].

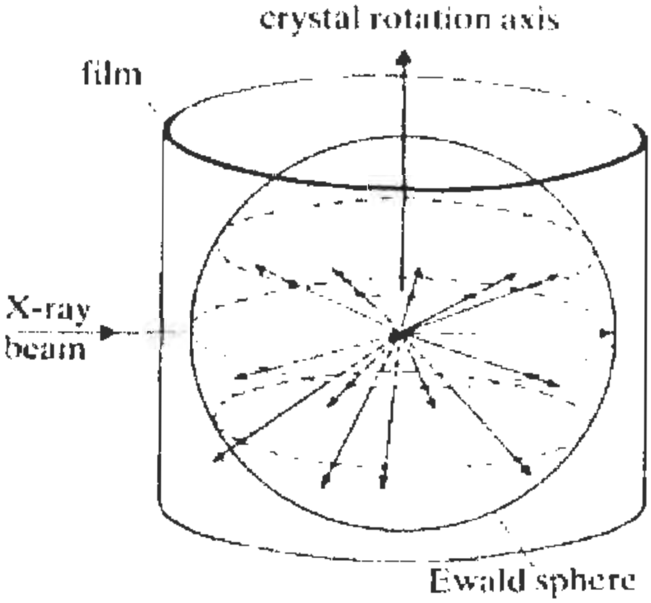


Figure 3.14 Rotating crystal method

3.7.6 Determination of particle size

Debye Scherrer formula is used for the determination of average particle size. Paul Scherrer introduces this formula in 1918. It's limitation for the measurement of grain size lies in the range 0.1 to 0.2μm. The formula is given by

$$D=0.9\lambda / (\beta\cos\theta)$$

Where β represents the full width half maxima and it is measured in radian, θ represents the Bragg's angle, D is the particle width and λ is the wavelength of Cupper Kα line. There is an inverse relation between diffraction curve width (B) and thickness of the crystal.When width of curve increases particle size decrease.

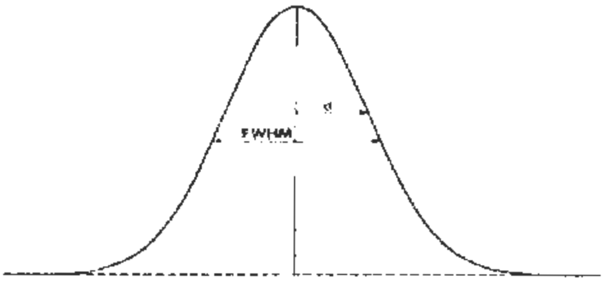


Figure 3.15: Full width half maxima

3.8 Scanning Electron Microscopy (SEM)

Scanning electron microscopy is an analytical technique, which is used to determine surface morphology, shape and size of the material[42]. SEM analysis is used to form a three dimensional image at varying magnification.[43].

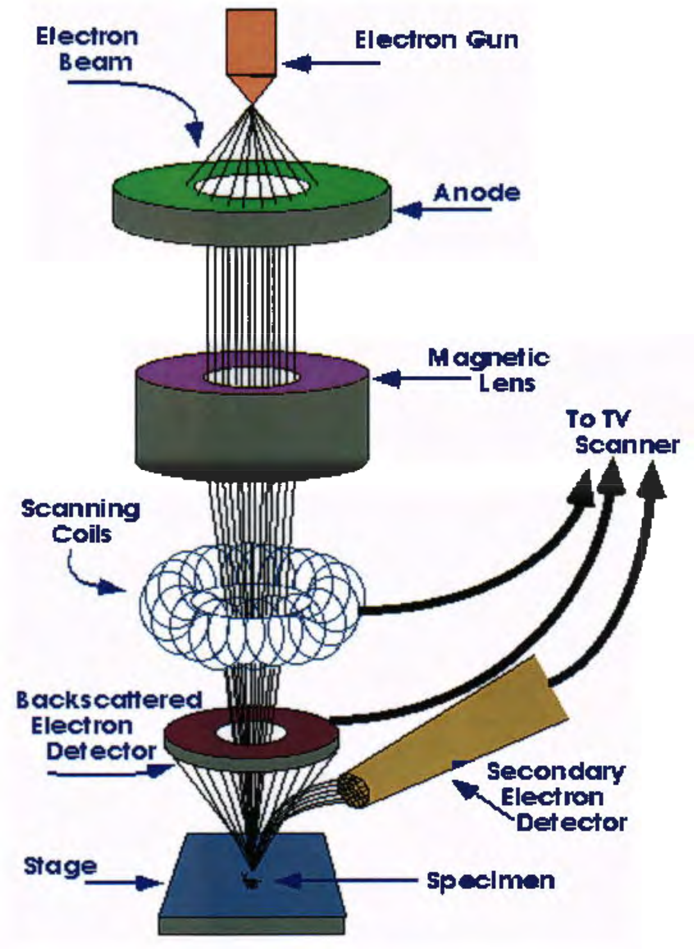


Figure 3.16: Different parts of SEM

3.8.1 Working principle of SEM

Electron gun produces primary electrons of high energy. These electrons incident on the surface of the sample. This produces secondary and back scattered electrons. These electrons are reflected from the surface of the sample. These electrons are collected on the detector in the form of electrical signals. These electrical signals form the image of sample on the screen. As secondary electrons produces image of the surface of the sample. The intensity of back scattered electrons is used for the determination of elements present in the sample. SEM gives the complete information about the morphology of sample by varying the intensity of secondary electrons[44].

3.9 EDX ANALYSIS

Energy dispersive X ray (EDX) analysis technique is used

- to find the percentage of element in the material
- product imperfections and defect analysis
- contamination detection

EDX becomes very powerful technique particularly in contamination analysis.The technique can be qualitative and quantitative and also provide information about percentage distribution of elements.

3.9.1 Fundamental principle of EDX analysis

As each element have a unique and special atomic structure so these elements allowes a special set of x rays to pass through them. To study the emission of x rays from a specimen, a high energy beam of charged particles like electrons or protons or a beam of x rays into the sample under studied. The energy of this beam varies from 10-20Kev[45]. At rest, an atom within the sample contains ground state. The incident beam excites this electron and ejects it from the shell. This creates an electron hole, where the electron was. An electron from the higher energy cell comes to fill this hole.Then a difference occures between a low energy level and a high energy level. This causes the emission of x rays. The energy of these emitted x rays is measures by EDX. This difference in energies examined the elemental composition of the material.

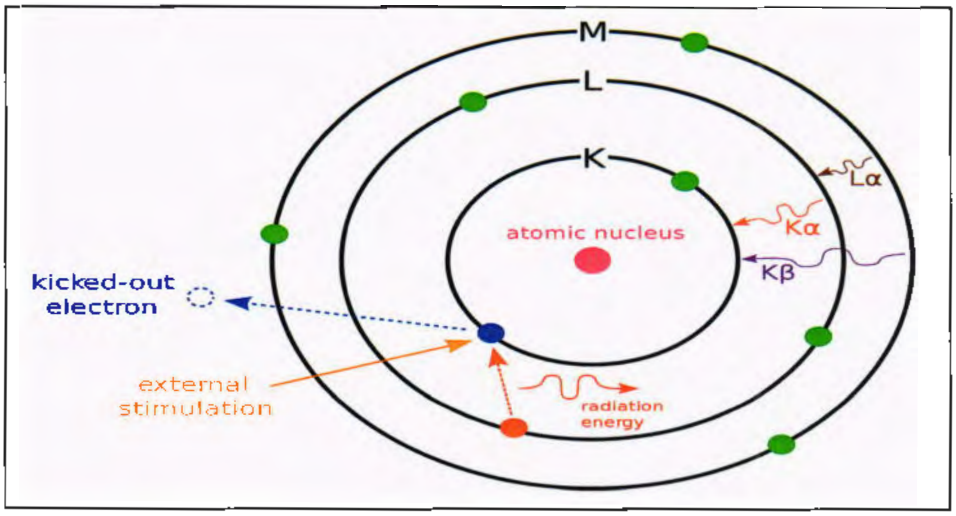


Figure 3.17 :Schematic illustration of EDX

3.10 Fourier Transfer Infrared Spectroscopy

Fourier Transfer Infrared spectroscopy is an analytical technique which is used to determine the organic, polymer and in some cases inorganic compounds. FTIR analysis uses infrared light and observes chemical properties of a sample[44]. In FTIR infra red radiations is used. These infra red radiation falls on the samples. A part of radiation is obsorbed by the sample.This absorbed radiation is compared with the reference light. This radiation is partially absorbed and partially transmitted. Both the reference beam and transmitted beam are combined together. By combining these beams a spectrum is obtained, which represent the molecular absorption thus infrared spectroscopy is used to recognize the unknown material. FTIR determine the amount of chemicals present in the material/ IR spectroscopy provides the detailed information about the internal structure of constituents of a sample.

3.10.1 Basic Components of FTIR

The unique part of FTIR spectrometer is Michelson's interferometer. Interferometer has following parts:

- Source
- Beam splitter
- Compensator
- Moveable mirror
- Fixed mirror
- Detector
- Screen

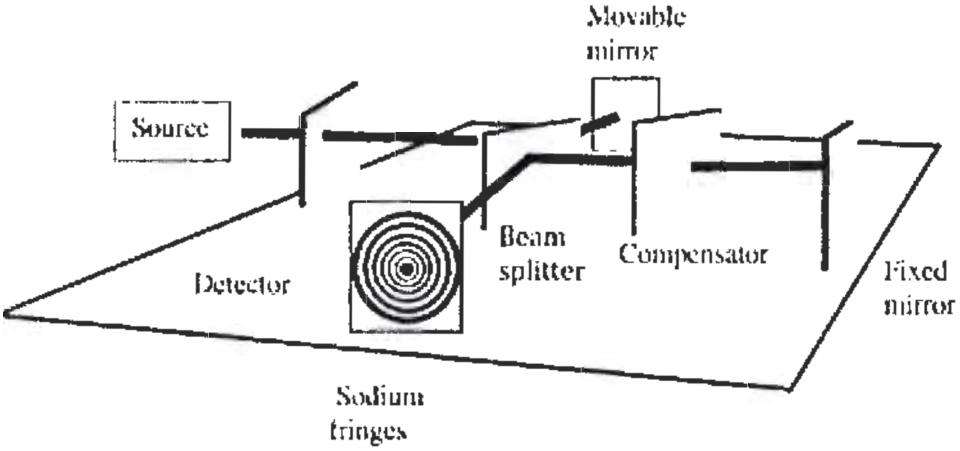


Figure 3.18: Different parts of FTIR

A source emits infrared light that hits the beam splitter. Beam splitter transmits half portion of light and half of light is reflected back. The reflected light strikes the fixed mirror and the transmitted light strikes the moveable mirror.Interferometer has mirrors at both arms. and oriented perpendicular to each other. Beam splitter is located at an angle of 45^0 relative to both mirrors. The lights beams passes through both of the mirrors are reflected back and recombine at the beam splitter to produce an interference pattern.

3.11 UV- Visible Spectroscopy

UV- Visible spectroscopy the charcterization techniques used for qualitative and quantitative analysis of a sample. It is also used to determine the purity of the material. This technique use visible light. In this region electronic transition of atoms and molecules. In this spectroscopy transition occurs from excited to grounded state while absorption deals with transition from grounded to excited state [45]. The absorption spectra show the impurity peaks contained by the material[46]. When UV visible light falls on the molecule having non bonding electron, then this electron absorb light in the form of energy and excite into higher anti bonding molecular orbital. If there is a small energy gap between highest occupied molecular orbital and the lowest occupied molecular orbital a longer wavelenth is absorbed[47]. This is very important technique which is used to characterize the absorption, reflection, transmission and band gap of different materials. This spectroscopy also measures the physicochemical properties of the material[48].

Figure 3.19: UV-Visible spectroscopy

3.11.1 working principle of uv visible spectroscopy:

UV visible spectrometer has following components.

1. Source
2. Dispersion devices
3. Sample holder
4. Detector
5. Signal processor

Source

Source produces a beam of light.

Dispersion devices

Dispersive devices disperse light of different wavelength at different angles. These devices are combined with an exit slit and used to select a particular wavelength of light from a continuous source. [49].

Sample holder

Sample holder contains sample. A variety of sample holders are available for uv visible spectroscopy. Mostly transparent sample holders are used.

Detector

A detector converts light signal into an electrical signal. Detector gives a linear response with low noise and high sensitivity. Normally a photomultiplier tube detector or photodiode detector is used in spectrometer. In order to determine that what amount of radiation is absorbed by the sample we compare the light passing through the sample and light reached the detector without passing through the sample [50].

Chapter 4

Results and Discussions

4.1 XRD ANALYSIS

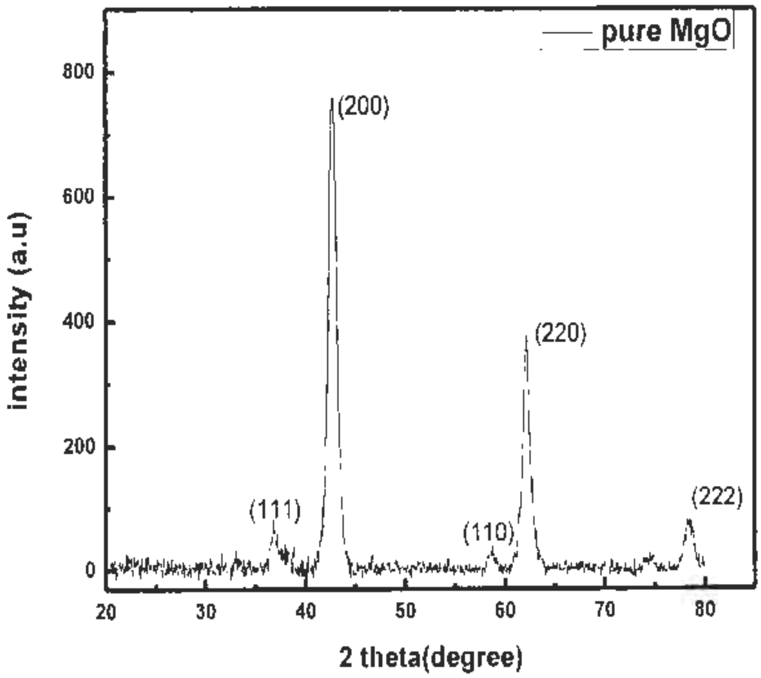


Figure 4.5 XRD Analysis of pure MgO

This figure shows the XRD spectra of pure and undoped MgO nano particles in the angle 2θ between 20° to 80°. The pattern shows sharp peaks at angles 36.75°(111), 42.6°(200), 58.7°(110), 62.1° (220), and 78.7°(222). The observed diffraction peaks are well matched with cubic MgO crystallites. These peaks are also in good agreement with JCPDS file of MgO (JCPDS Card NO. 45_946). The intense and sharp peak occures at 42.6°(200). This sharp peak shows the better crystallanity. No other peaks are detected revealing the purity of the sample.

By using information given in the following table and then by using Debye sherrer formula we can determine the particle size.

Table 4.3: Peak positions in XRD graph of pure MgO

Angle (2θ)	Height (cts)	FWHM	d spacing	Rel inten.[%]
36.7555	58.81	0.5904	2.44524	8.46
42.6073	695.27	0.7675	2.12198	100.00
58.7718	27.70	0.9446	1.57113	3.98
62.1892	371.68	0.5314	1.49275	53.46
74.3249	19.55	0.9446	1.27622	2.81
78.7085	56.64	1.0080	1.21477	8.15

The particle size determined by using sherrer equation is 22nm.

4.1.1 XRD Analysis of 5% Ag doped MgO

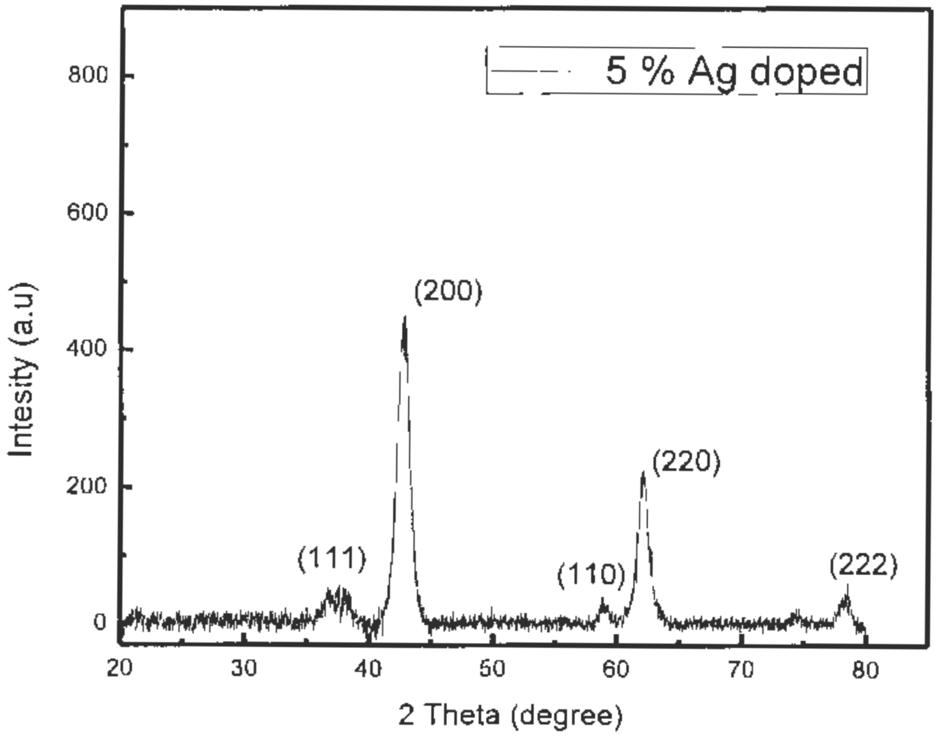


Figure 4.6 XRD analysis of 5 % Ag doped MgO

This figure shows XRD pattern of 5% Ag doped MgO nano particles. It is seen from the graph that peaks are compressed by doping the silver. Peaks are broader and less intense as compared to XRD graph of pure MgO nanoparticles. Broadening the peaks shows that by doping silver into MgO, particle size decreases.

Table 4.4: Peak positions in XRD graph of 5% Ag-doped MgO

Angle (2θ)	Height(cts)	FWHM	d spacing	Rel inten [%]
36.6944	41.33	0.5904	2.44917	10.50
38.2160	37.09	0.7872	2.35509	9.43
42.9341	393.49	0.9840	2.10658	100.00
58.8925	24.64	0.7872	1.56820	6.26
62.2900	174.28	0.2952	1.49058	44.29
74.4136	15.24	0.7872	1.27492	3.87
78.6299	29.78	1.2000	1.21578	7.57

4.1.2 XRD Pattern of 15% Ag doped MgO

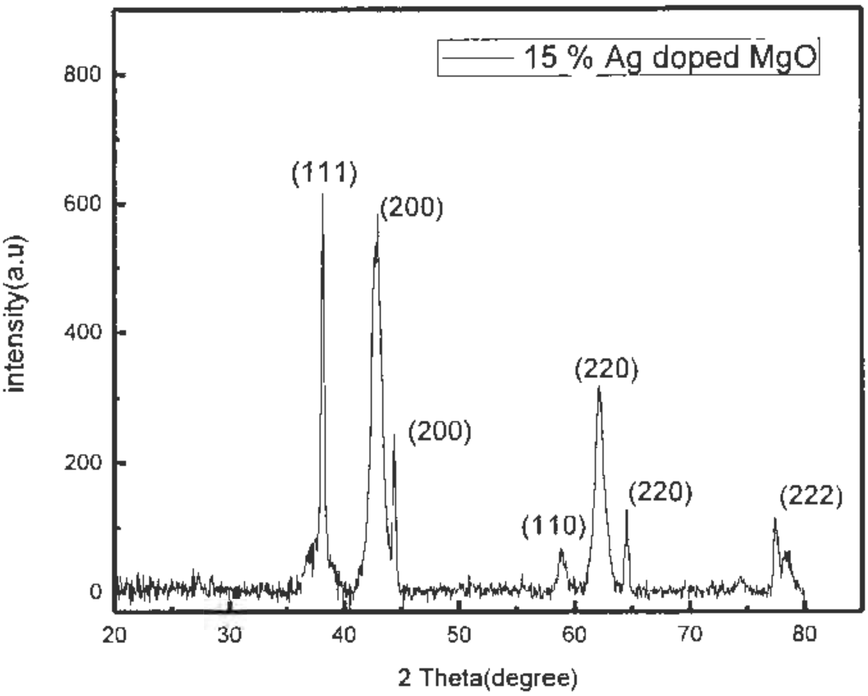


Figure 4.7:XRD pattern of 15% Ag doped MgO

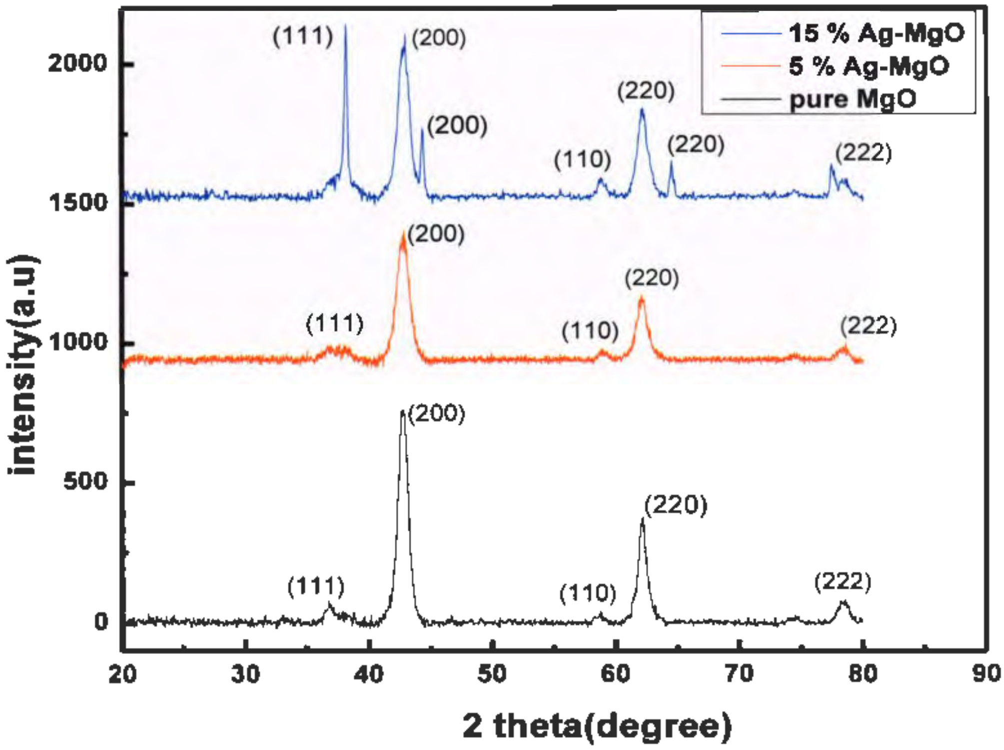


Figure 4.8: A comparative XRD graph of pure and silver doped MgO nanoparticles

Table 4.5: Peak positions of 15% Ag- doped MgO

Angle(2θ)	**Height[cts]**	**FWHM**	**d-spacing**	**Rel.int[%]**
38.169	584.11	0.2362	2.35786	100.00
42.5855	465.39	0.2952	2.12301	79.68
44.3462	252.60	0.2362	2.04273	43.25
58.7590	63.07	0.5904	1.57144	10.80
62.0352	317.11	0.4133	1.49609	54.29
64.4659	128.61	0.2362	1.44543	22.02
74.4478	20.35	0.9446	1.27442	3.48
77.3919	120.56	0.1771	1.23313	20.64
78.3623	54.72	0.7200	1.21926	9.37

It is seen from the graph by increasing concentration of silver, additional peaks of silver are obtained at 44.34°(200),64.46°(220) and 77.39° (222). There is a slight phase shift in the peaks towards a smaller angle by increasing the concentration of silver.

4.2 SEM Analysis

SEM Images of pure MgO

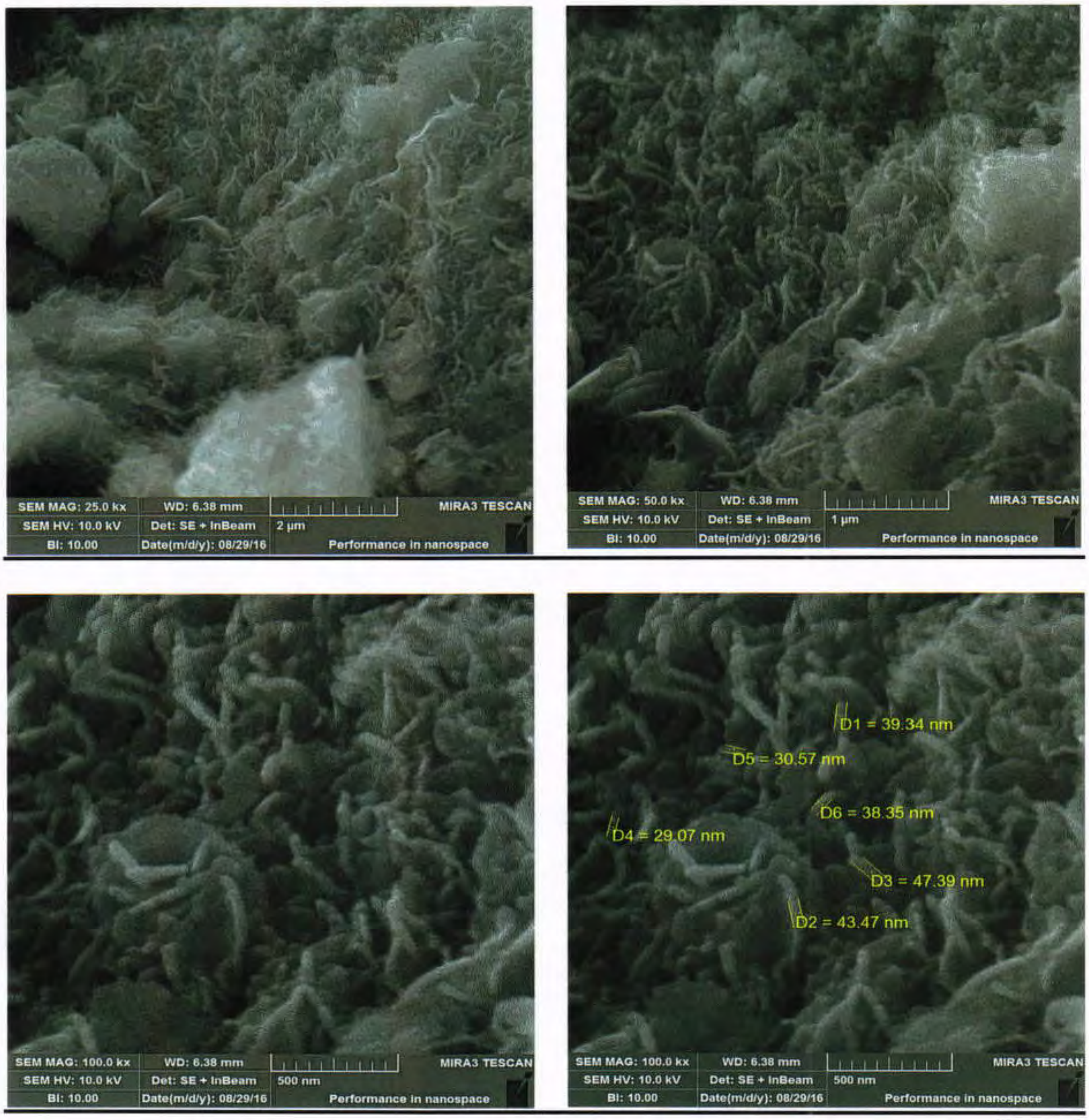


Figure 4.1: SEM images of pure MgO nano particles at different magnifications

The SEM (scanning electron microscopy) shows the morphology of nanoparticles. The SEM images of MgO nanoparticles are shown in figure. These figures show the rod like morphology. These rods are agglomerated with each other. The average diameter of these nano rods is 38nm.

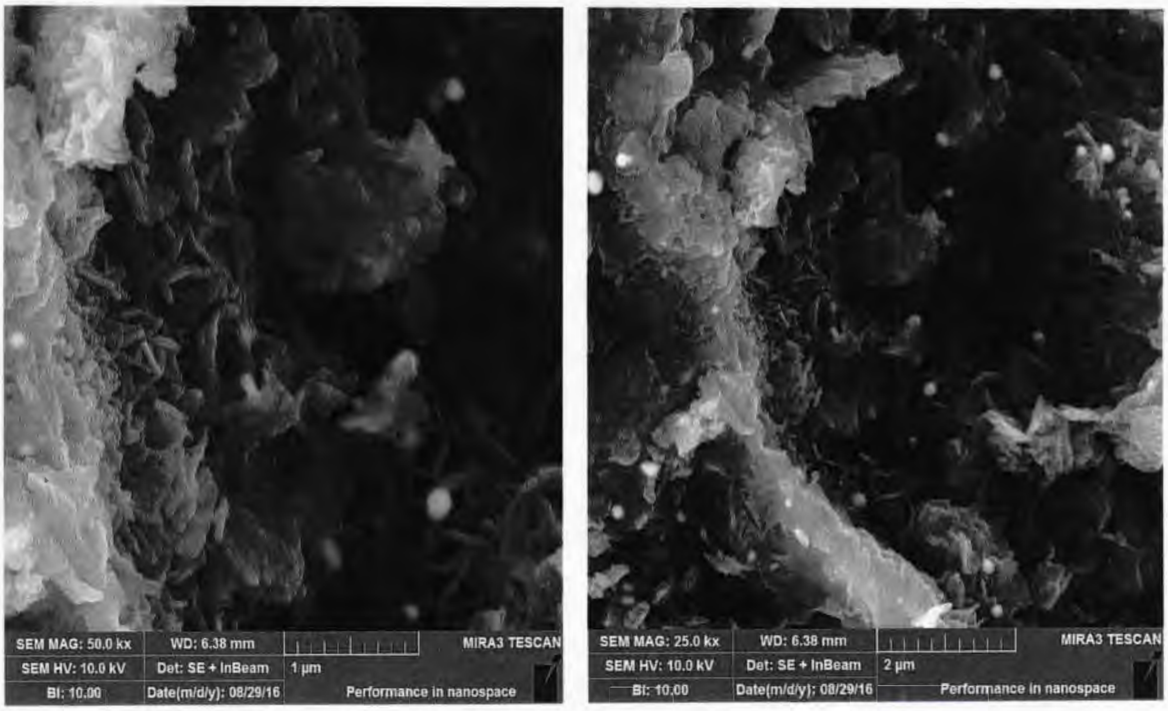


Figure 4.2 SEM images of Ag-doped MgO nano particles at different magnifications

This SEM (scanning electron microscopy) of silver doped Magnesium oxide nano particles clearly shows that silver element lies on magnesium oxide nano particles in spherical pattern at different ranges.

4.3 EDX Analysis

4.3.1 EDX Analysis of pure MgO

Table 4.1 : chemical composition of MgO

Element	Weight %	Atomic %
OK	51.29	61.54
Mg K	48.71	38.46
Total	100.00	

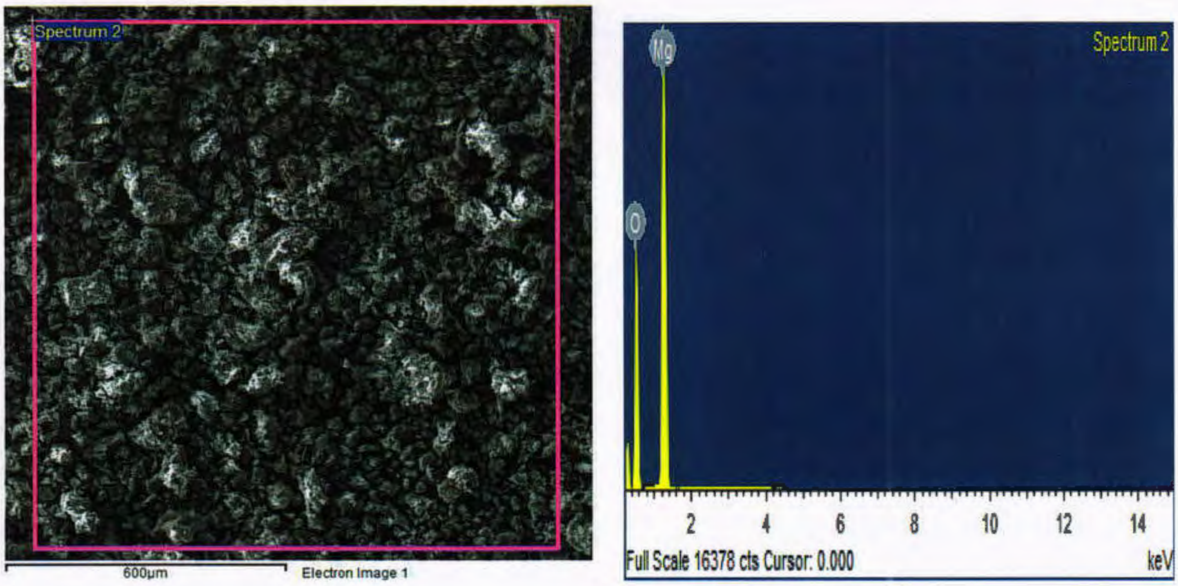


Figure 4.3: EDX Analysis of pure MgO

The EDX (Energy dispersive X-ray) analysis is performed to determine the elemental compositions. The EDX spectrum of pure MgO nanoparticles is shown in figure. The peaks of oxygen and magnesium are clearly seen. There are no other peaks showes the purity of prepared sample.

4.3.2 EDX Analysis of Ag doped MgO

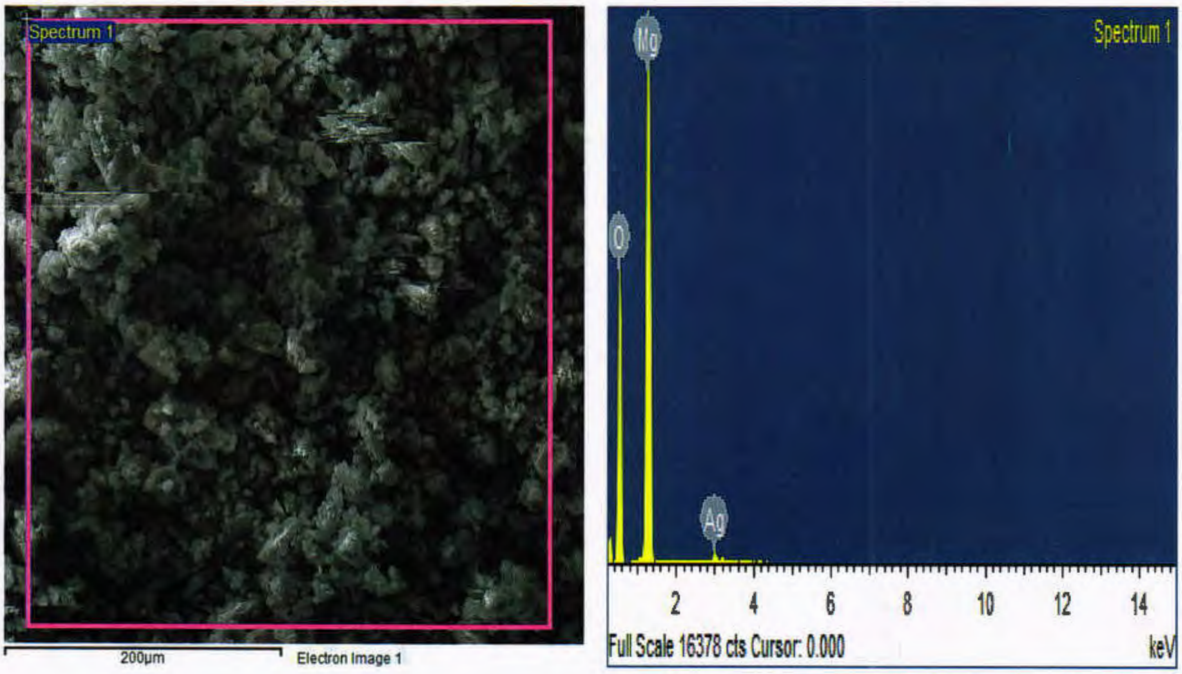


Figure 4.4:EDX Analysis of Ag-doped MgO

Table 4.2: chemical composition of Ag-doped MgO

Element	Weight %	Atomic %
OK	52.69	63.85
Mg K	44.76	35.69
Ag L	2.56	0.46
Total	100.00	

This figure shows the EDX spectra of Ag doped magnesium oxide nano particles. It gives information that which element is present in the sample. Peaks of oxygen, Magnesium and silver are clearly seen. No other peak is observed, which shows that there is no impurity in the sample.

4.4 FTIR ANALYSIS

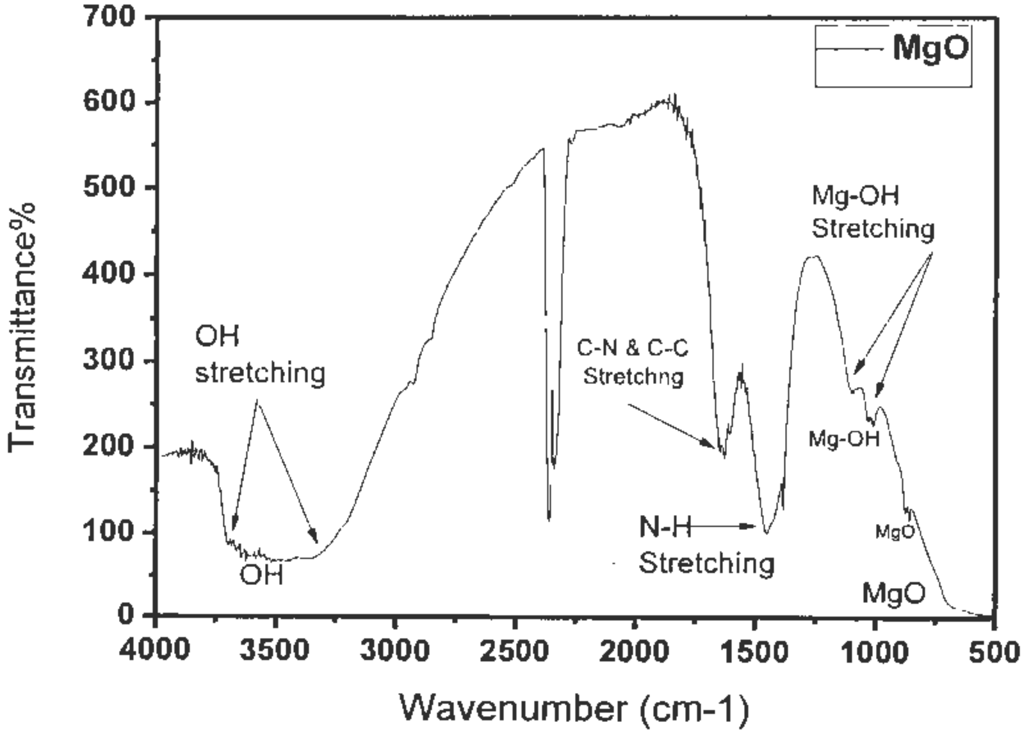


Figure 4.9: FTIR spectrum of pure MgO

The composition of MgO sample was analyzed by FTIR spectroscopy. MgO nanoparticles were mixed with KBr and pallet of mixture was used for infra red spectroscopic measurement at room temperature. The broad spectrum between 3308 and 3688 cm^{-1} shows stretching mode of vibration in hydroxyl group (O-H). The band at 1635 is assigned to C-N and C-C stretching. The peak at 1453 is due to stretching vibration of N-H bond. The bands at 1031 and 1090 correspond to the Mg-OH stretching vibrations. The peak at 666 and 864 shows the formation of MgO.

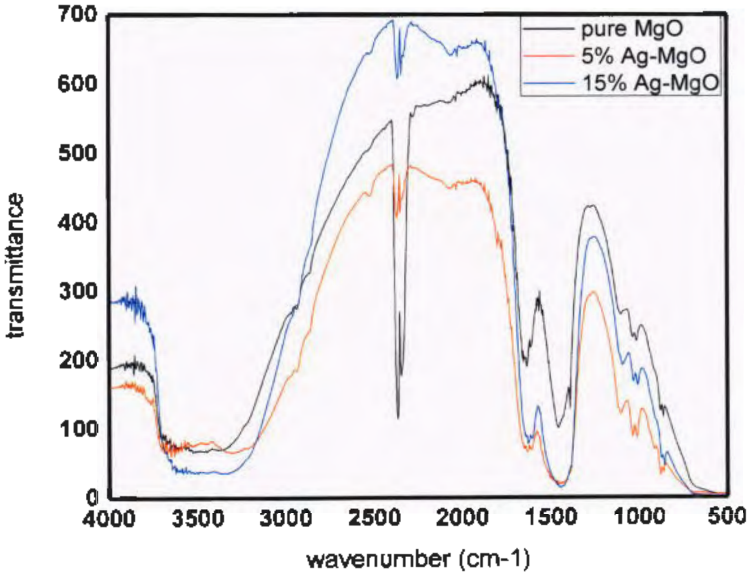


Figure 4.10 :A comparative FTIR graph of pure and silver doped MgO

A close comparison of FTIR graphs of pure MgO and Ag doped MgO was done. It shows some minor structural variations between pure MgO and Ag doped MgO. The study of these graphs shows that by doping crystal undergoes some lattice stress.

4.5 Anti-bacterial activity

For checking the anti-bacterial activity of pure MgO and Ag doped MgO I took 0.06gm of pure MgO and 0.06gm of 15% Ag doped MgO. Then I make a solution of 0.06gm MgO in 10ml of ethanol and shake it well until the MgO nanoparticles are fully dissolved in ethanol. The same solution is made for 15% Ag doped MgO nanoparticles. Then I spread both these solutions in two different petri dishes in which E.Coli bacterial culture is already present. Then I placed these petri dishes into sunshine for testing the anti-bacterial behavior of MgO nanoparticles and Ag doped MgO nanoparticles.

Figure 4.11 Sample for antibacterial activity of pure MgO

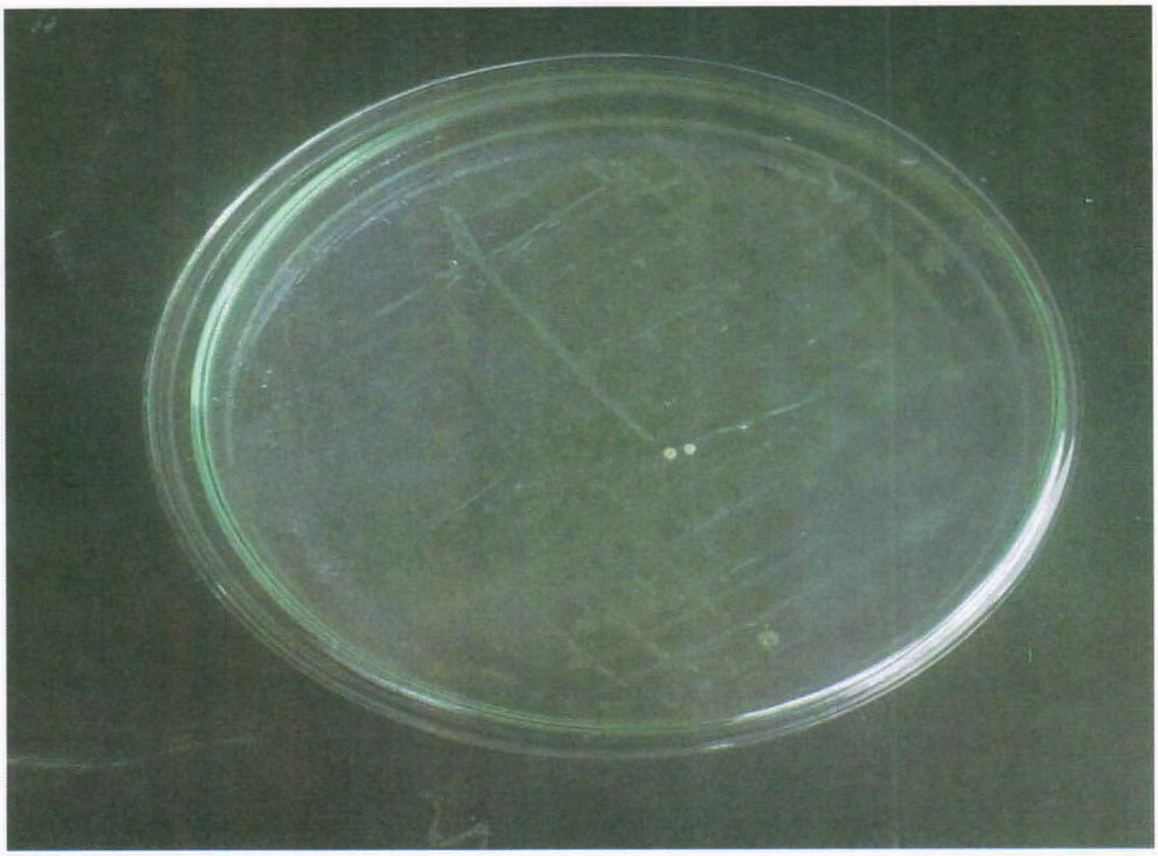


Figure 4.12: Sample for antibacterial activity of Ag doped MgO

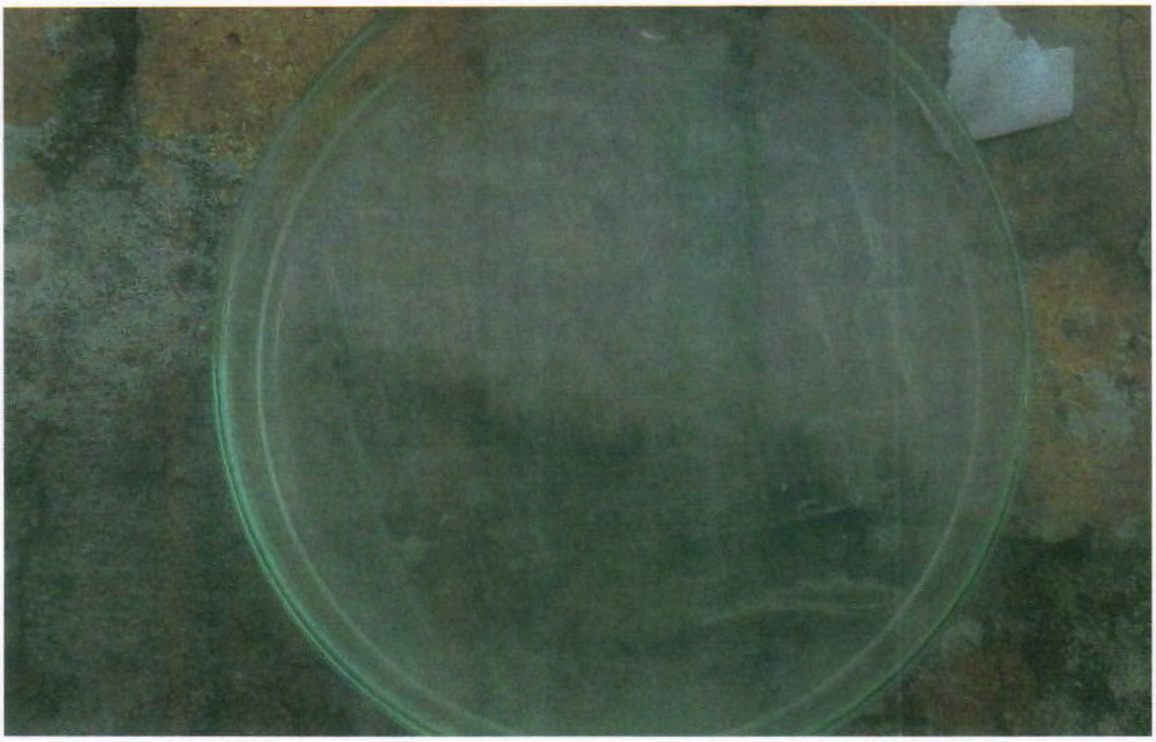


Figure 4.13: Effect of pure MgO nanoparticles after 3 hours

Figure 4.14: Effect of Ag doped MgO nanoparticles after 3 hours

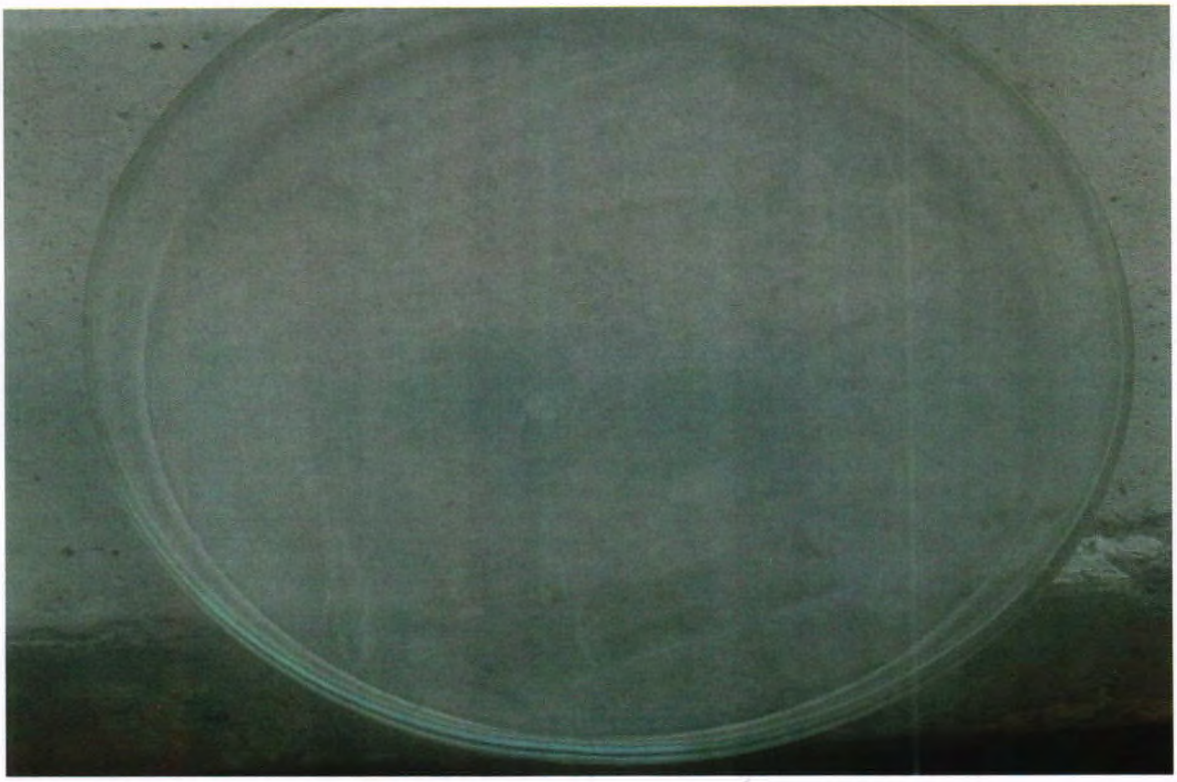


Figure 4.15:Effect of pure MgO nanoparticles after 6 hours

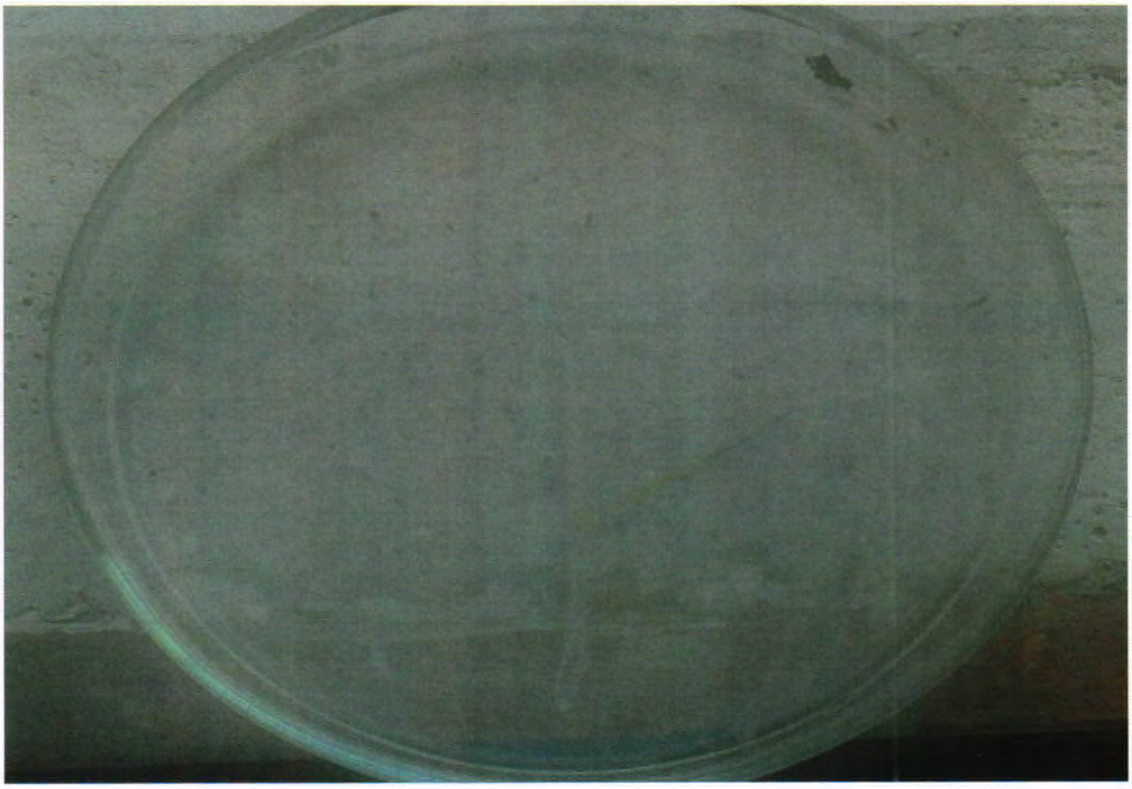


Figure 4.16: Effect of Ag doped MgO nanoparticles after 6 hours

It is seen from above pictures that many of the bacterial colonies were killed which shows the anti-bacterial behavior of MgO nanoparticles. But when the effect of pure MgO and silver doped MgO nanoparticles was compared that it was concluded that Ag doped MgO has better anti-bacterial behavior than pure MgO. So we can enhance the anti-bacterial activity of MgO nanoparticles by doping silver into it.

4.6 Conclusion

Magnesium oxide nano particles were succesfully prepared by co precipitation method. Magnesium nitrate was used as precurser and sodium hydroxide as a precipitating agent to synthesis MgO nanoparticles. It was observed that this method of preparing MgO nanoparticles is very simple and cheap. Different concentrations of silver was doped in MgO. Structural characterization of MgO nanoparticles were carried out by X-ray differection(XRD) technique. Surface morpholgy was studied by using Scanning electron microscopy(SEM). Chemical composition was studied by using Fourier transform infra red(FTIR) spectroscopy. It was observed by SEM results that prepared MgO has rod like morphology. These rods are agglomerated with each other.XRD revealed that MgO has cubic structure and they posses better crystallinity. XRD and EDX results showed that pure samples of MgO are prepared.

Magnesium oxide nanopartilces and silver doped MgO were applied to investigate the anti bacterial activity. E-Coli bacteria were used for experiment. These bacteria were prepared in biotechnology lab through streaking method. Anti becterial behaviour of pure MgO and silver doped MgO was studied under sun light. MgO nanoparticles killed many colonies of bacteria,which showes anti bacterial behaviour of MgO. But this behaviour was enhanced by applying Ag-doped MgO nanopartilces on Bacteria. So it was concluded that Ag-doped MgO have batter anti-bacterial activity than pure MgO nanopartilces.

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