“EXPLORING PURE AND FE-DOPED ZnO NANOPARTICLES SYNTHESIZED via CHEMICAL PRECIPITATION”

Dissertation submitted to Department of Physics,
MES Asmabi College, P. Vemballur
(Affiliated to University of Calicut)

In partial fulfillment for the award of the degree of

MASTER OF SCIENCE
IN
PHYSICS

Submitted by

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CERTIFICATE ON PLAGIARISM CHECK

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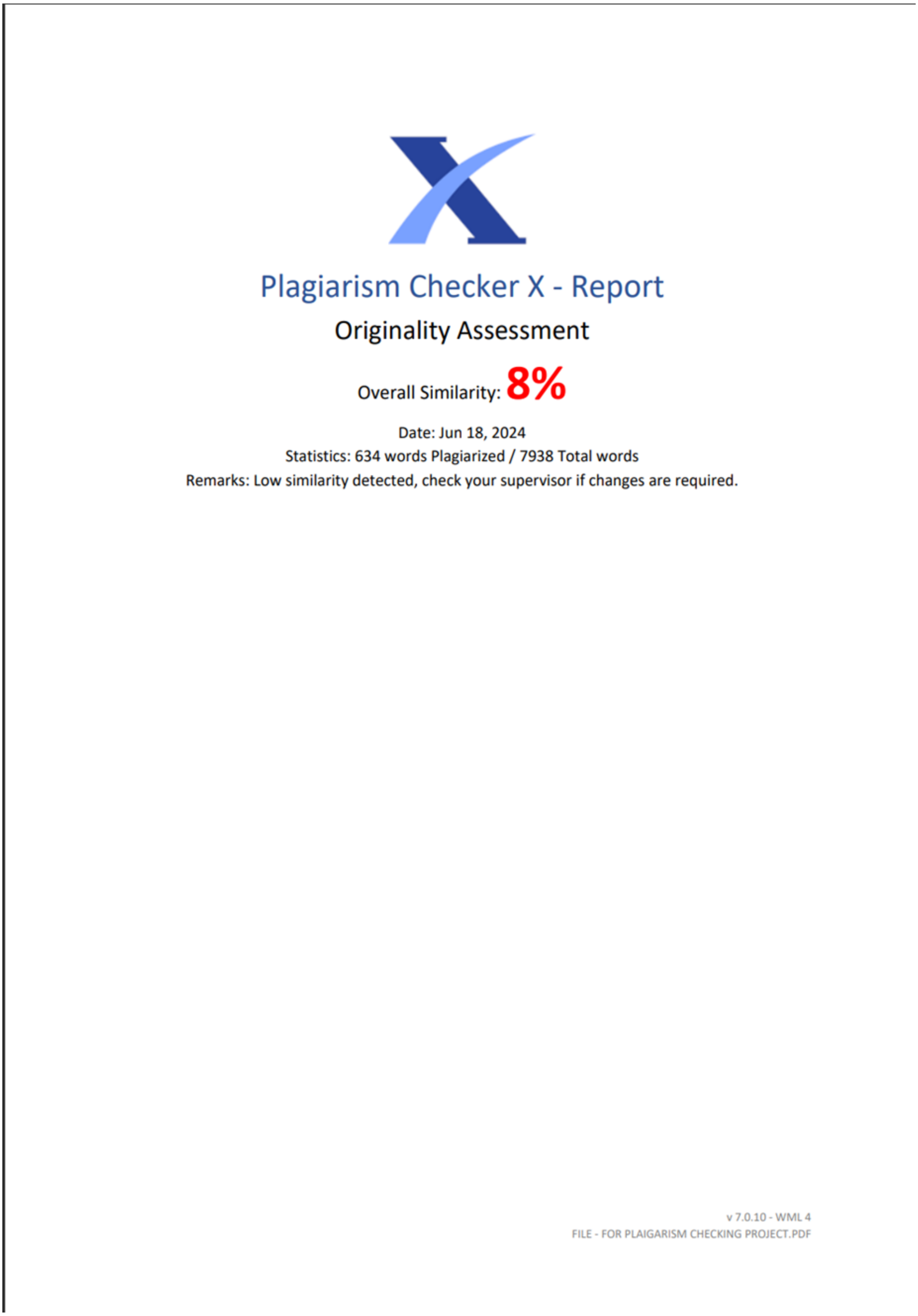

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EXPLORING PURE AND Fe DOPED ZnO NANOPARTICLES SYNTHESIZED via CHEMICAL PRECIPITATION

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DECLARATION

I hereby declare that this submission **"EXPLORING PURE AND Fe DOPED ZnO NANOPARTICLES SYNTHESIZED via CHEMICAL PRECIPITATION"** is a record of my project work carried out under the guidance of Dr. SAFEERA T A, Assistant Professor, Department of Physics, MES Asmabi College P Vemballur. I also declare that this project work has not been submitted previously for the award of any degree or diploma earlier. The findings in the project report are based on the information collected by me and not copied from elsewhere.

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ABSTRACT

In the present work, the zinc oxide nanoparticles are synthesized by chemical precipitation method. The first chapter gives a brief outline about nanotechnology. The second chapter deals with characterization techniques of nanoparticles. The third chapter contain the introduction about synthesized sample, zinc oxide. The methodology, results and discussions are in the fourth chapter. Zinc oxide nanoparticles (ZnO-NPs) are one of the metal oxide nanomaterials and a useful and versatile(flexible) inorganic substance because of its distinct physical and chemical properties. Strong chemical stability, a widening radiation absorption spectrum, a strong electrochemical coupling coefficient, and great photostability are all characteristics of zinc oxide nanoparticles. In our project we use the chemical precipitation method in order to create zinc oxide nanoparticles. This report presents a systematic study of ZnO and ZnO-Fe nanoparticles synthesised using the chemical precipitation method and their characterizations using XRD, UV-Vis spectroscopy, Photoluminescence and Sem.

CONTENTS

- RESULT AND DISCUSSION
    - XRD ANALYSIS
    - UV – VIS ANALYSIS
    - PHOTOLUMINESCENCE ANALYSIS
    - SEM ANALYSIS
- CONCLUSION

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CHAPTER 1

INTRODUCTION TO NANOSTRUCTURE

INTRODUCTION

Nanotechnology refers to the study, manipulation, and application of materials and devices on an extremely small scale, typically ranging from 1 to 100 nanometres. Nanotechnology and nanoscience are the name given to those branches of research and engineering that use phenomena occurring at nanoscale dimensions in the creation nanometre, characterisation, manufacturing and use of components, materials, gadgets, devices and systems.

In 1974 Tokyo Science University Professor Norio Taniguchi invented the term nanotechnology to describe precision machining of materials to within atomic-scale dimensional tolerances. The father of nanotechnology is considered to be American physicist Richard Feynman. In 1959, he introduced the concept, during his talk, “There’s Plenty of Room at the Bottom.” Nanoscience and nanotechnology are the study and application of extremely tiny things and can be used across all the other science fields, such as chemistry, biology, physics, materials science, and engineering etc. Prof. C.N.R. Rao is considered as the “Father of Indian Nanotechnology.” Nanoscience is a study of the fundamental principle of molecules and structures with at least one dimension roughly between 1 and 100 nanometres. “Nano” refers to a scale of science in the matrix system which denote one billion of base unit, i.e one nanometre is equal to 10 ^-9 m. The particle in size ranging from one to 100 nm are the building blocks of nanomaterials. Nano materials are created from blocks of nanoparticles, and thus they can be defined as a set of substance where at least one dimension is approximately less than 100 nm. Nano carbons such as fullerenes, carbon nanotubes, and graphene are excellent examples of nanomaterials. At the nanoscale, a particle’s size can affect characteristics like melting point, fluorescence, electricity conductivity, flexibility, and chemical reactivity. Gold on a scale demonstrate the special qualities that at the nanoscale. When particles size is reduced to nanoscale dimensions, properties such as fluorescence, electrical conductivity, melting point, chemical reactivity and the magnetic permeability will change. Solubility is an important property of nanomaterials. These materials have higher surface to volume ratio than bulk materials.

1.1. CLASSIFICATIONS OF NANOMATERIAL

1) ZERO -DIMENSIONAL NANOMATERIALS

Materials having all the dimensions within the nanoscale, i.e no dimensions are larger than 100 nm. zero dimensional materials means confinement is 3D and the degrees of freedom is zero. Some examples are Fullerene, Dentrimers and Quantum dots.

2) ONE- DIMENSIONAL NANOMATERIALS

Materials having one dimension outside the nanoscale. It confinement is 2D, degrees of freedom is 1. This material exhibit elongated and needle like shapes. Examples are carbon nanotubes (single walled and multiwalled) nanohorns, inorganic nanotubes, nano ribbons, nanowires etc.1-D nanomaterials have thin films or surface coatings and are used in the circuitry of computer chips and for anti-reflective properties and hard coatings on eyeglasses. These have been used in electronics, chemistry, and engineering.

3) TWO-DIMENSIONAL NANOMATERIALS

Materials having two dimensions outside the nanoscale. This means confinement is 3D and the degrees of freedom is 2. These materials exhibit flat, plate like shaped. 2D nanomaterials are considered to be the thinnest nanomaterials. Due to their thickness and dimensions on nanoscale. These nanoparticles have a multi layered structure with strong in-plane bonding and weak van der Waals force intermediate layers. Some examples are metal nanosheets, graphene-based materials, transition metal oxides. The lengthy, permanent nanostructures of nanomaterials have thick membranes. These are utilized to create nanopore filters, which are used for filtering and separating small particles. Asbestos fibre is an example of 2D nanoparticles.

4) THREE-DIMENSIONAL NANOMATERIALS

Three-dimensional nanomaterials, that are not confined to the nanoscale in any dimensions. Above 100 nm these materials have three arbitrary dimensions. The bulk (3D) nanomaterials are made from multiple arrangement of nanosized crystals in different examples are nanoprisms and nanoflower.

5) HARD NANOPARTICLES

These nanoparticles give polymers access to their characteristics. The addition of clay nanoparticles to polymer matrix strengthens and increases reinforcement in the polymers. In order to make fashionable and useful apparel, hard nanoparticles have also been incorporated into textile fibers.

6) SEMI SOLID OR SOFT NANOPARTICLES

A bunch of the semi-solid and soft nanoparticles that have been created liposomes are of particular significance. Different forms of liposome nanoparticles are employed clinically such as delivery methods for anticancer medications, antibiotics, antifungal agents, and vaccines.

1.2. SIGNIFICANCE OF NANOMATERIAL

The basic properties of the nanometals are friction. Nanomaterials shows uniqueness property for several reasons. One which is their smaller size. Nanomaterials are ten thousand times smaller than the width of a human hair. Nanoparticle’s surface area to volume ratio is very high. Distinct property of a nanomaterials is attributed to quantum effects, larger surface area, and self- assembly. The properties of material become strongly dependent on the surface of the material which makes the uniqueness at nanoscale. The Nanoscale gold describes the unique properties of that take place at the nanoscale, depending on the size of the particle, it has appeared red or purple. Compared to bulk materials, nanoscale materials have a higher surface area to volume ratio. When the surface area per volume increases, materials can become more reactive.

A) QUANTUM CONFINEMENT EFFECT

Quantum confinement are the spatial confinement of electron hole pairs in one or more dimensions within a material.

Quantum wells – 1 D confinement

Quantum wires -2D confinement

Quantum dots -3D confinement

When the diameter of a particle approaches the De- Broglie wavelength of electrons in the conduction band the electron hole pairs spatially confined. Hence the energy difference between bands is increased with particle size decreasing. Then the size of the particle is too small to be comparable to the De - Broglie wavelength of the electron we can observe quantum confinement effect. Quantum confinement effect is observed only at dimensions below 2nm (metals do not have a band gap, so quantum size effects less prevalent). Quantum confinement is more noticeable in semiconductors because they have an energy gap in their electronic band structure.

1) Thermodynamic properties

Particle size is proportional to the melting point. They depend on the surface to volume ratio of the materials. That is size decrease surface to volume ratio increases melting point decreases. High bonding energy indicates high melting that means high stability.

2) Electronic properties

The optical properties of nanostructures are determined by the electronic structure of the materials. The electronic structure of the nanomaterials is very much dependent on the surface atom, the size and shape of nanoparticle. The properties like conductivity or resistivity are come under the category of electrical properties. In the case of carbon nanotubes conductivity changes with change in area of cross section. But, multiwalled carbon nanotube’s conductivity is different than that of single nanotube of same dimensions.

3) Optical properties

The optical measurements are helping to understand the bad gap of the materials. Band gap of the material which is inversely proportional to size of the particle. Nano wires and nanorods are optically active. The optical properties are related to electrical and electronic properties. The quantum confinement increases the energy required for absorption if the crystalline size of the nano crystalline material becomes comparable or smaller than the De Broglie Wavelength of the charge carriers created by the absorbed light. They give the absorption or photoluminescent spectra and their characterization is done using microscopic technique. The spectra obtained is shift towards the shorter wavelength region (blue).

4) Magnetic properties

Magnetization is observed to be decreasing with decreasing particle size. When the particle size reduced the surface contribution will increase. Because of the aberrations in interatomic spacing in the interfacial area, nanostructured materials exhibit a drop in saturation magnetization and ferromagnetic temperature, magnetic characteristic of nanomaterials are affected by grain size. Because of the their some properties such as low coercivity, strong electrical receptivity, good thermal stability. Nanocrystalline iron base alloys are employed for soft magnetic applications.

5) Mechanical properties

It has applications in nanodevices like nano reactors, nanogenerators etc. The mechanical property investigated are ductility, strain hardening, yield stress, brittleness, strength, rigidity. Different material exhibited different mechanical properties. Nano materials have excellent mechanical properties due to the surface to volume ratio and quantum effects. When ordinary materials mixed with nanoparticles, the grains become somewhat more refined. This results in the formation of either an intragranular or intergranular structure, which strengthens the grain boundary. The processing parameters, which include temperature technique, treatment time, and dosage of nanoparticles, are the major indicators of how the production process affect the mechanical properties of materials. The microstructure of nanomaterials reflects the effects of grain size on their mechanical characteristics. Nanoparticles have a strong surface activity and a notecible size effect. They contribute to the material's improved mechanical characteristics, decreased porosity, increased relative density and small size of the nanoparticles.

- **SURFACE TO VOLUME RATIO**

The characteristics of nanoparticles are significantly influenced by their surface area to volume ratio. When comparing the same volume of the materials to nanoparticles, the latter have a relatively larger surface area. Consider a sphere of radius r, for instance: The sphere will have a surface area of 4pir2. The sphere has a volume ratio will therefore be 4pir2/{4/3(pir3)}.

1.3. APPLICATIONS OF NANOTECNOLOGY

Nanotechnology alters the molecular structure of materials to craft smart objects. Nanotechnology and its microscopic universe offer wide variety of possibilities for contemporary science and industry. Nanoparticle, for example benefit from their much higher surface area to volume ratio. Their optical properties, such as fluorescence, change as a function of particle diameter. Numerous industrial areas can benefit from the application of nanotechnology and nanomaterials. Typically, they can be found in places like:

1) Electronics Devices

When it comes to creating stronger, lighter, more conductive quantum nanowires and smaller, faster, and more efficient microchips and devices, carbon nanotubes are almost certain to take the place of silicon. Because of its characteristics, graphene is ideal for use in the creation of flexible touch panels.

2) Energy

Solar panels that convert twice as much sunshine into power are technically achievable. Additionally, nanotechnology reduces costs, creates lighter and stronger wind turbines, increases fuel economy, and can save energy since some nanocomponents are thermally insulated.

3) Medicine

Certain nanomaterials characteristics makes them perfect for enhancing cancer or neurodegenerative disease early detection and treatment. They have the ability to specially target cancer cells without harming healthy cells. Additionally, certain nanoparticles have been added to pharmaceutical goods like sunscreen. The use of silver nanoparticles for use in medical devices is a hot topic. Nano silver kills a broad range of harmful microbes and has been shown to be effective against the Methicillin -resistant Staphylococcus aureus superbug and the HIV virus. Nanomedicine is not limited to simple single element nanoparticles such as silver. More complicated nanoparticles can perform certain task such as homing in on cancer cells to destroy them or drug delivery that can send drugs directly into cells.

4) Food

In this area of study, nanocomposites to improve food production by enhancing mechanical and thermal resistance and reducing oxygen transfer in packaged goods, or nano biosensors to identify the presence of pathogen in food. Silver has long been known as an effective antimicrobial agent and in its nanoform can now be easily impregnated invisibly into almost any product to aid in the destruction of bacteria and vires. This has important application in the food industry in terms of manufacturing, preserving, and storage.

5) Cosmetics

The fascinating group of nanoparticles known as fullerenes $C_{60,}$ they form which resemble small footballs of carbon atoms are being used in cosmetics in the form of face creams to remove other unwanted particles such as free radicles which are believed to cause damage to the body and skin. Sun creams are now available with titanium dioxide nanoparticles. The micro- sized particles are used as sunblock but are white in colour and are not used in sun creams that need to be invisible when applied. Then the Titanium oxide nanoparticles provide identical UV protection properties to the bulk material, but when particle size is reduced,the cosmetically unattractive whitening disappears.

6) Textile

Textile industry is making increasing use of nanomaterials to make them functional and smart. For example, nano silver is playing a lead role because of its antimicrobial properties. The Clothes can also be treated with nano film make them stain, water and static resistant. These films which are only a few atoms thick, could be in contact with skin over prolonged periods.

7) Agriculture

By using nanotechnology in agriculture, food output can be increased while maintaining or improving its safety, quality, and nutritional content. Utilizing fertilizers, insecticides, herbicides and plant growth factors/regulators efficiently are crucial methods for enhancing agricultural yields. Using nanocarriers allows for the controlled release of plant growth regulators, herbicides, and insecticides. In order to carry out pesticides in a controlled manner, improved release systems such as silica and polymeric nanoparticles have also been created. These species can be carefully delivered and released gradually by using Nanoscale carriers. These methods, which increase agriculture yields without harming the soil or water, are refered to as "precision farming". The most significant benefit is that using nanoencapsulation can reduce the herbicide dosage.

8) Environment

Some of its environmentally-friendly applications include heavy metal nanofiltration systems, ion-based air purification, and waste water treatment using nanobubbles. There are nano catalysts available to increase the efficiency and reduce the pollution of chemical reaction.

9) Tissue engineering

Tissue engineering is the study of the growth of new tissues and organs, starting from a base of cells and scaffolds. Because of their ability to generate crucial physical and chemical properties their improve their performance and make them useful for a variety of applications, nanoparticles are distinguished by their nanoscale dimension. These materials are highly useful in many TE applications because of their surface conjugation and conducting properties for gold nanoparticles, their antimicrobial properties for silver and other metallic nanoparticles and metal oxides, their surface conjugation and conducting properties of gold nanoparticles and the antimicrobial properties of silver and other metallic nanoparticles and metal oxides, the and metal oxides, their fluorescence properties for quantum dots, and their special electromechanical properties for carbon nanotubes. More over injured connective tissues has been replaced in TE using graphene based nanoparticles. Bio composite scaffolds containing nano silver could control infection caused by bacteria. The scaffolds' incorporated silver nanoparticles served as an adhesive layer to prevent infection, sepsis, and implant malfunction. Advanced tissue engineering technique may lead to life extension.

1.4. SYNTHESISE OF NANOPARTICLES

- There are two different approaches of material fabrications, one is the top-down approach and other is the bottom-up approach. In top-down technique, break-down of the bulk material into nanoscale structures. The methods utilized to create micron sized particles are extended by Synthesis techniques. These methods which are simpler by nature rely on either the division or removal of bulk material or the downsizing of bulk production procedures in order to create the desired structure with appropriate attributes. The primary issues with the hierarchical method is that the imperfections of the surface structures. Also introduces internal stress, surface defects, and contaminants. Some examples are ball milling, electro-spinning, lithography, sputtering, laser ablation. However, bottom-up strategy is an alternative approach that might result in less waste and be more cost effective. It describes the process of building a material up from the bottom up, either molecule by molecule, cluster by cluster, or atom by atom. The methods are either the early stages of development or are just stating to be applied in the commercial manufacturing of nano powders. Some examples are chemical vapor deposition, chemical vapour condensation, sol-gel, precipitation etc.

1.4.1. Top -down approaches

1) High energy ball milling

It is the simplest way of making nanoparticles of metal and alloy in the form of powder. The Mill is a device used for grainting (shapes can be planetary, rod, vibratory). The number of containers depends up on the quantity of resultant materials. Harden steel or tugsten carbide balls are put in a container along with the host powder or flakes and rotated at high speed. Container filled with inert gas. The ratio of balls and material is 2:1. The container is closed with tight lid and it should not be more than half filled. Large balls produce smaller grains but larger defects. The presence of impurities from and air in the container will affect the final product. Heat generates during the collision also lead to increase in reaction rate with impurities. To reduce this cryocooling can be used. In planetary ball milling the rotation is about some central axis. By controlling the speed of rotation and duration of milling is possible to ground the material to fine powder. Examples are cobalt, chromium etc.

2) Lithography

Using a greasy material, the image portions are worked on to flat stone or metal plate in the lithography printing process. In order to make the non- image parts ink-repellent, and the ink to stick to them. Alois Senefelder1796. Examples are Electron beam lithography and Photolithography. In Photolithography, it is used in microfabrication to pattern parts on a thin film or the bulk of a substrate (also called a wafer). It uses light to relocate a geometric pattern from a photomask also called an optical mask, to a photosensitive that is, light-sensitive, chemical photoresist on the substrate.

3) Sputtering

When high energy particles hitting a target sample, such as a silicon wafer, solar panel or optical device the process is known as Sputtering, occurs causing atoms to be expelled from the target or source material and deposit on the substrate. Some characteristics are the more pure and precise thin film deposition on the atomic level and Sputter yield can be controlled, Materials with high melting points can be easily sputtered also it shows better adhesion to substrate. A target source material that will be deposited onto the substrate and cause the plasma to glow is given a negative charge before the substrate is put in an inert gas-filled vacuum chamber. When free electrons from negatively charged target materials collide with the outer electronic shell of argon gas atoms in a plasma environment, their similar charges drive the electrons away. The inert gas atoms transform into positively charged ions due to the collisions' momentum, which attracts the negatively charged target material at a very high speed and "sputters off" atomic-size particles from the target source material. These minuscule particles pass through the sputter coater's vacuum deposition chamber and acquire.

1.4.2. Bottom -up approach

1) Chemical vapor deposition

It is a hybrid method using chemical in vapor phase to obtain coatings of variety of organic or inorganic materials. It is relatively simple economical easy processing and possible for depositing different types of materials. These are the Metallo-organic CVD, Atomic layer epitaxy, Vapor layer epitaxy, Plasma enhanced CVD. Technique to produce high- purity, high performance solid materials, usually μ thick with slow deposition rate. Apparatus includes, Gas delivery system Reactor chamber Substrate loading mechanism Energy source Vacuum system Exhaust system and exhaust treatment system Process control equipment. One or more volatile precursors are introduced to substrate like Halides, Hydrides and metal alkyls. The intended deposits are created on the substrate surface by the precursors reaction and/or breakdowns. The substrates are heated by resistive heating, radiant heating, radio frequency heating, lasers and UV-vis light. Volatile by-products if produced are eliminated by the reaction chamber’s gas flow.

2) Sol – gel method

In this chemical procedure, a "sol" (a colloidal solution) is formed that then gradually evolves towards the formation of a gel-like system containing both a liquid phase and solid phase. It might be necessary to remove a sizable amount of fluid at first in order to identify the gel-like characteristics. Numerous methods can be used to accomplish this. The simplest approach is to let sedimentation happen naturally and then drain the liquid that remains. Phase separation can also occur more quikely with centrifugation. Modifications made during this processing phase will undoubtedly have a significant impact on the finished component's microstructure characteristics. The precursor sol can be used in three different ways: it can be cast into suitable container with the desired shape (e.g., to obtain monolithic ceramics, glasses, fibers, membranes, aerogels), or it can be deposited on a substrate to form film amount of fluid may need to be removed initially for the gel-like properties to be recognized. This can be achieved in any number of ways. The easiest method is to allow time for sedimentation to occur, and then pour off the remaining liquid. Centrifugation can also be used to accelerate the process of phase separation. The microstructure of the final component will clearly be strongly influenced by changes imposed during this phase of processing. The forerunner sol can be either deposited on a substrate to form a film (e.g., by dip-coating or spin coating). It can also be used to synthesize powders e.g., microspheres, nanospheres). Among the benefits are that densification may be accomplished at a considerably lower temperature, making it an inexpensive low-temperature process that permits precise control over the chemical composition of the final product. It is possible to add tiny amounts of dopants, including organic dyes and rare-earth elements, to the "sol" and have them evenly distributed throughout the finished product.

3) Molecular beam epitaxy

Epitaxy method for thin-film deposition of single crystals. It is widely used in the manufacture of semiconductor devices. **Epitaxy** = Process of growing a crystal of a particular orientation on top of another crystal, where the orientation is determined by the underlying crystal. Requires high vacuum/ ultrahigh vacuum. Slow deposition rate (< 1000 nm per hr).

Solid source Molecular beam epitaxy

In ultra-pure form elements like arsenic and gallium and, in ultra-pure form, are heated in separate evaporators until they slowly sublime. After that, the gas condensed on the wafer, where they it might interact with other molecules. Gallium and arsenic combine to generate single crystal gallium arsenide. The gaseous components contacting the surface could be desorbed if evaporation sources are utilized. The rate at which material impinges on the substrate can be controlled by adjusting the source's temperature, and the rate at which hopping or desorption occurs can be influenced by substrate's temperature. **Beam**: Because of their lengthy mean free pathways, evaporated atoms do not interact with vaccum-chamber gases or with each other until they reach the wafer. With the use of reflection high-energy electron diffraction, the growth of the crystal layers is seen (RHEED). The computer has the ability to manipulate the openings that lie front of every combustion chamber, allowing for exact control over the layer thickness. This control is so precise that it can even manage the thickness down to an individual atomic layer. Using this method, Intricate constructions made of layers of various materials can be created. The level of control has actually enabled the creation of structures where electron may become trapped in space. This resulting in quantum dots or even quantum wells.

When the substrate needs to be cooled, cryopumps and cryopanels are used to maintain an ultra-high vacuum environment inside the growth chamber. They use liquid nitrogen or cold nitrogen gas to reduce the substrate to a temperature of around 77 kelvins, or 196 degrees Celsius. In a vacuum, contaminants settle to cold surfaces.

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CHAPTER 2

CHARACTERIZATION TECHNIQUE

INTRODUCTION

Characterization methods in nanomaterials play a pivotal role in understanding their structural, morphological, and chemical properties, which are crucial for tailoring their behaviour and optimizing their applications across various fields including electronics, medicine, energy, and more. These methods encompass a wide range of techniques that offer insights into the intricate features of nanomaterials at the nanoscale level.

One of the fundamental techniques employed in nanomaterial characterization is microscopy, which includes scanning electron microscopy (SEM) and transmission electron microscopy (TEM). SEM provides high-resolution images of the surface morphology, while TEM offers detailed information about internal structures and particle sizes down to atomic levels. X-ray diffraction (XRD) is another indispensable method used to determine the crystal structure and phase purity of nanomaterials. By analysing the diffraction patterns generated when X-rays interact with the material's atomic lattice, researchers can discern its crystalline properties and identify specific phases present.

Both UV-visible spectroscopy and PL spectroscopy serve as powerful tools for characterizing nanomaterials, offering complementary information about their optical behaviour, electronic structure, and potential applications in fields ranging from optoelectronics to sensing and imaging.

In summary, UV-visible spectroscopy and photoluminescence spectroscopy are indispensable techniques in the characterization toolkit for nanomaterials, allowing researchers to probe their optical properties and electronic transitions, thus facilitating the design and optimization of nanomaterial-based devices and technologies

2.1 X-RAY DIFFRACTION METHOD

XRD is a non-destructive technique, it does not need elaborate sample preparation. Powder XRD method has been widely used to determine the crystal structure, lattice parameter, stresses, crystallite size of the nanoparticles, measure average spacing between layers or rows of atoms and also determine the orientation of simple crystal. It is the important indirect method for determining the crystallite size. X-rays are electromagnetic radiation with photon energies in the range of 100 eV to 100 keV. Only short wavelength x-rays between a few angstroms to 0.1 Å (1–120 keV) are used for is used for diffraction applications These x-rays with short wavelengths are comparable to the interplanar distances; hence, it is perfect for examining the atomic and molecular structure configurations in a variety of materials. Strong X-rays have the ability to pierce materials deeply and reveal details about their structures. In XRD, a monochromatic beam of x-rays with a typical wavelength ranging from 0.7 to 2 Å is incident on a sample material. The x-rays are diffracted by the crystalline phases in the sample, if Bragg's law is satisfied.

$$n\lambda = 2d\sin\Theta \qquad (2.1)$$

Where d is the spacing between atomic planes in the crystalline phase and λ is the x-ray wavelength and n is the order of diffraction. The XRD pattern is like a fingerprint, and the mixtures of various crystalline phases can be easily distinguished by comparing their XRD pattern with reference to data given in electronic databases such as the Inorganic Crystal Structure Database (ICSD) X-ray

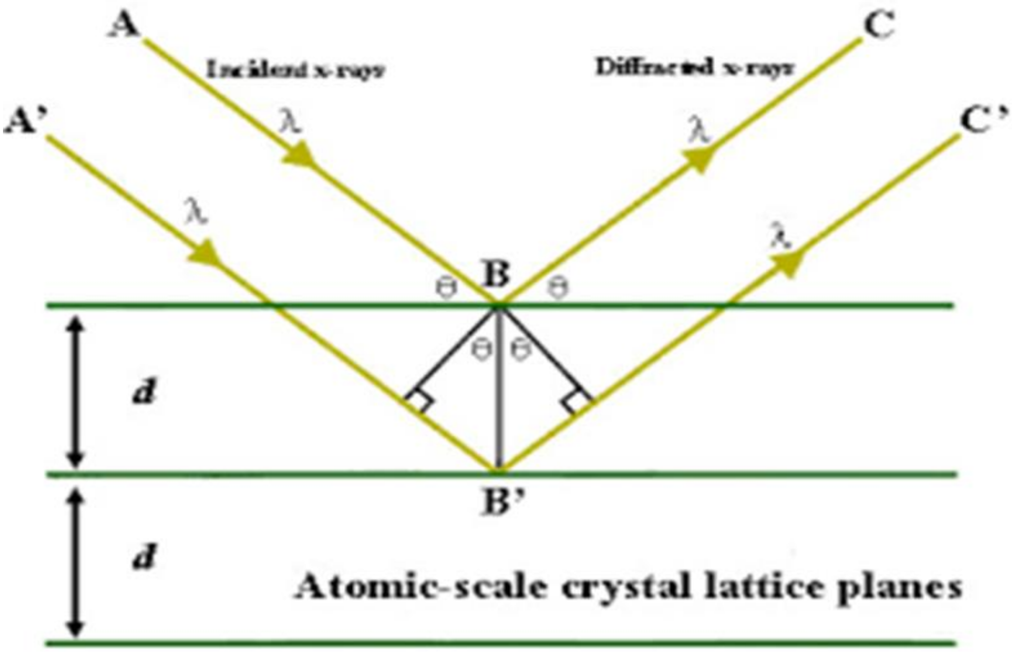

Fig. 2.1 X-ray diffraction from crystal lattice

X-rays are collimated and directed onto the specimen sample. As the sample and detector are rotated, then intensity of the reflected X-rays is recorded. This X-ray signal is captured by a detector, which subsequently processes it and outputs the count rate to a device like a printer or computer monitor. The X-ray detector is mounted on an arm to collect the diffracted X-rays and rotates at an angle of 2θ, while the specimen rotates at an angle θ along the direction of the collimated X-ray beam in a X-ray diffractometer. A goniometer is the tool used to spin the sample and maintain the angle. The detected output is fed to the recorder which gives the intensity variation of different diffracting planes. From which d-spacing can be determined.

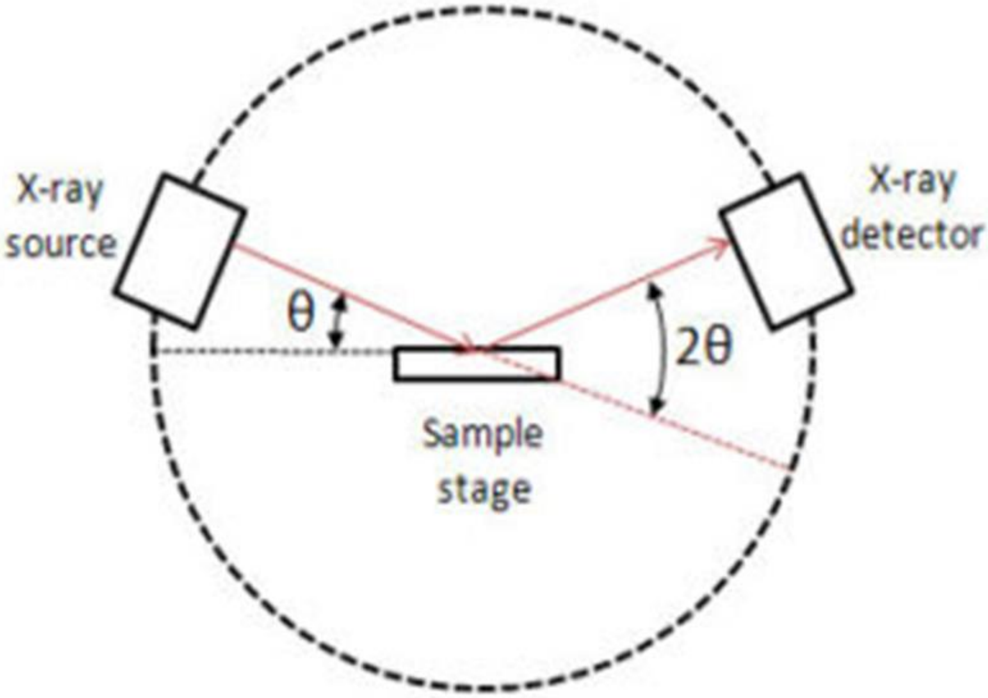

Fig. 2.1.1 Schematic diagram of x-ray diffractometer

X-ray tube, sample holder and x-ray detector are the major parts of the diffractometer. Williamson and Hall, Warren and Averbach, and Debye–Scherrer are the three methods used to calculate the crystallite size and strain of the sample. If there is no inhomogeneous strain, the average crystallite size is measured from the full width at half maximum (FWHM) of a diffraction peak broadening using the Debye–Scherrer equation.

$$D = k\lambda / \beta c \cos\theta \qquad \textbf{(2.2)}$$

Where D is the crystallite size, βc is the FWHM, and theta is the glancing angle corresponding to the diffraction peak. The value of Scherrer's constant (k) varies between 0.9 and 1.15, and it is generally taken as 0.9 for a cubic material for simplicity. If the sample has strain because of crystal defect and or distortion, the strain can be able to compute using the equation

$$\epsilon = \beta c / 4\tan\theta$$

Total diffraction peak broadening due to smaller crystallites size and strain can be compute by combining the above equations

$\beta c = k\lambda / D\cos\theta + 4\epsilon\tan\theta$

Rearranging the equation above yields

$\beta\cos\theta = k\lambda / D + 4\epsilon\sin\theta$, which is called Williamson–Hall equation. From this equation we can plot with 2sinθ on x-axis and βcosθ on y-axis known as Williamson -hall plot. The crystallite size is determined from the y-intercept, while strain is calculated from the slope. Warren and Averbach's method take into account both the peak width and the shape of the peak which provides both the crystallite size distribution and lattice micro strain. From XRD diffraction pattern we can calculate important parameter is lattice constants. For a given plane with miller indices (h k l), using interplanar distance (d_{hkl}) we can calculate lattice constant. The equations are used

For cubic,

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \tag{2.3}$$

For hexagonal,

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2} \tag{2.4}$$

The corrected value of lattice parameter can be estimated from the Nelson-Riley (NR) plot. By extrapolating the linear fit of the graph between error function f(θ) with calculated lattice constants, to the y -axis corrected lattice constants can be obtained. The equation is given by

$$f(\theta)=\frac{1}{2}\left[\frac{\cos^2\theta}{\sin\theta}+\frac{\cos^2\theta}{\theta}\right] \tag{2.5}$$

Fig.2.1.2.XRD analysis instrument

2.2 UV VISIBLE SPECTROSCOPY

Optical spectroscopy technique can be generally classified into two groups: absorption and emission spectroscopy and vibrational spectroscopy. UV–vis spectroscopy is an representation of absorption and emission spectroscopy. Which involves wavelength from 300 to 800 nm to get the optical properties of the metallic or semiconducting nanoparticles with the size from ~2 to 100 nm. On the other words, Ultra-violet-visible (UV-vis) spectroscopy is used to obtain the absorbance spectra of a compound in solution or as a solid. The absorbance of electromagnetic radiation, or light energy, is essentially what is being seen spectroscopically. This radiation excites electrons in the compound or material from their ground state to their single excited state. The wavelength range of 800-200 nm corresponds to the 1.5 - 6.2 eV UV-vis area of energy in the electromagnetic spectrum. hence it is called UV-Vis spectroscopy. According to Beer-Lambert Law,

$A=\log I_0/I=€Lc$

This equation is the absorbance spectroscopy principle. Here A denotes the absorption (unitless), l0 denotes the intensity of light incident on the sample, l is the transmitted light intensity and c is the concentration of a solution (M). ε is the molar absorptivity of the compound or molecule in solution ($M^{-1}cm^{-1}$), L is the path length of the cuvette or sample holder (usually 1 cm).

Data from UV-vis spectroscopic can provide both qualitative and quantitative information about a particular molecule or substance. UV-vis spectroscopy operates well on liquids and solutions, however skewed if the sample is more like suspension of solid particles in liquid, as the sample will scatter light more than absorb it. Although it is uncommon, the majority of UV-vis devices can analyze solids samples or liquids using a diffraction apparatus. Liquids and solutions are typically the easiest to analyze with UV-Vis equipment.

One exciting use for this methodology is to calculate the band using several method; various software packages are available for this purpose. Mathematically,

E=hc/λ

Where, E=energy band gap

H= plancks constant

C=speed of light

Lambda=wavelength

The Tauc method is founded on the assumption that energy dependent absorption coefficient alpha can be expressed by the equation

$(\alpha h\nu)^{1/¥} = A\,(h\nu - Eg)$

The bandgap of the samples can be estimated by plotting $(\alpha h\nu)\ 1/n$ as a function of hν and extrapolating linear region of the curve to absorption equal to zero. Utilized is UV-vis spectroscopy to determine the concentration, size, purity, and aggregation state of the constituents, and functional groups in molecules.

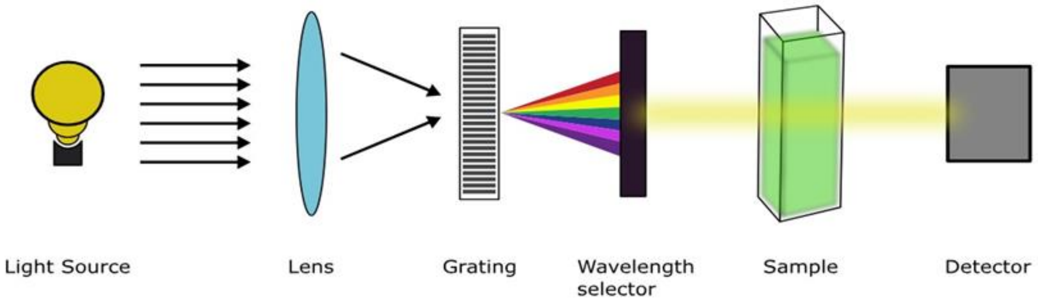

Fig.2.2. Schematic representation of UV-Visible spectroscopy

2.3 Photoluminescence (PL)

Photoluminescence spectroscopy is a very sensitive tool for the investigation of electronic states in the bandgap of semiconductors. It involves optical excitation of electrons from the valence band to the semiconductor’s conduction band. In other words, a type of light emission spectroscopy known as photoluminescence (PL) spectroscopy uses a mechanism known as photo-excitation to produce light emission. It’s utilizing photons with energy levels higher than the bandgap energy. This results in the creation of electron–hole pairs. After some time electrons and holes recombine, thereby releasing energy in the form of phonons, photons, or Auger electrons.

When a specimen is exposed to light, its constituent electrons undergo excited states transitions. (excitation) Light is one way that energy can be released when electrons descend from their excited states to their equilibrium states. (relaxation).

PL is a non-destructive and contactless optical techniques for examining a material’s electrical structure. In PL spectroscopy is an optical phenomenon a material is exposed to UV light at a wavelength around 254 nm from a Xenon lamp, which causes electrons to be excited to a higher energy level. After losing energy, these extremely energetic electrons return to the ground states by emitting photons into the visible spectrum, which the photomultiplier tube detect.

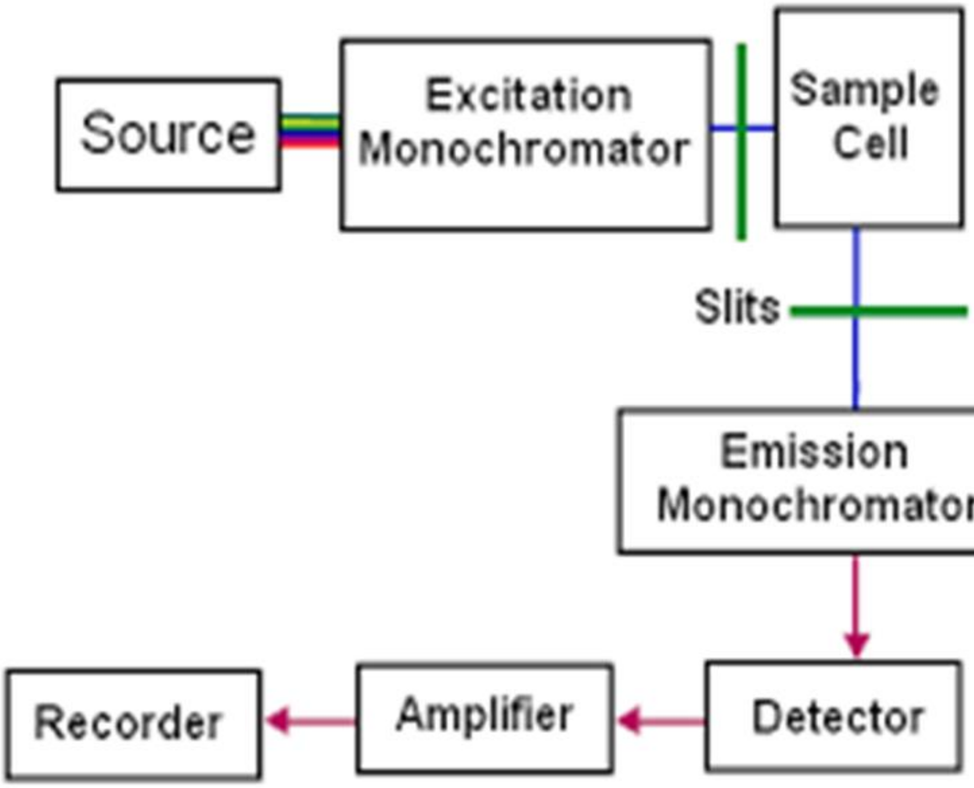

Fig. 2.3. Schematic representation of photoluminescence

Intensity, emission wavelength, bandwidth of the emission peak, and the emission stability are the four parameters characterize the PL emission properties of a sample. As the size of a semiconductor nanoparticle decreases, the bandgap increases, resulting in shorter wavelengths (blueshift) of light emission. This shift in wavelength of PL spectra can be utilized to study the bandgap and impurity levels of the nanomaterials.

2.4 Scanning Electron Microscopy (SEM)

Electron microscopy is the most important techniques to characterize the material's morphology on the nano meter to atomic scale. There are two main types of electron microscopes: SEM and TEM. When a high-energy primary electron beam passes through a specimen, electrons undergo elastic scattering and inelastic scattering along with transmitted beam (in case of thin foil). The interactions of primary electrons with a specimen's atoms generate several signals such as secondary electrons (SEs), backscattered electrons (BSEs), Auger electrons, Bremsstrahlung (continuous) x-rays, characteristic x-rays, etc. High-energy electrons go forward and form the transmitted beam. Some of the electrons are diffracted or scattered. Both transmitted beams and diffracted beams are elastically scattered and coherent.

The basic Principle of the scanning electron microscopy is, when the electron beam (energy 5–30 keV) impinges on the specimen, many types of signals are generated including SEs and BSEs. When the electrons contact with the positively charged nucleus, the majority of them scatter at huge angles (ranging from 0° to 180°). Typically, BSE is used to refer to these elastically scattered electrons.

When electrons in orbital shells interact with each other, some of the electrons lose kinetic energy and scatter inelastically. Conduction electrons that are weakly bound may be expelled from the sample by incident electrons.
These electrons are called SEs. The SEs as well as BSEs are widely used for SEM topographical imaging. The interaction of primary electron beam with a specimen generates SEs, BSEs, and characteristic x-rays, which are used to obtain 3- D image, elemental contrast and elemental composition or elemental mapping, respectively.

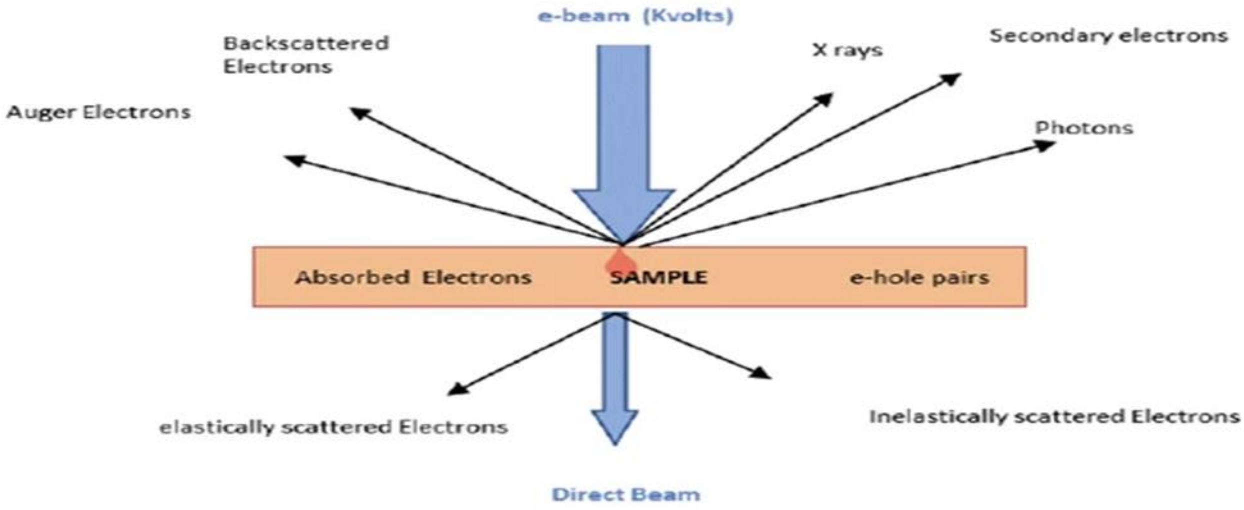

Fig .2.4. Electron interaction with the atoms on the sample surface

The lateral spatial resolution of a SEM image is affected as per the dimension of the volume from where the signal electrons escape. Thus the images formed by SE's have a better spatial resolution than that formed by BSE's. A typical SEM has an image resolution of 5–10 nm, but a modern state-of-the-art SEM like field emission SEM is capable of providing an image resolution of 1 nm. In an electron microscope, electrons emitted from a hot filament are usually used. Sometimes cold cathode (a cathode that emits electrons without heating it) is also used. Under the application of a very high electric field a cold cathode emits electrons. It is also known as a field emitter. Such SEMs are known as FE-SEM and are able to give better images than hot filament SEM. In an electron microscope, the electron beam can be focussed to a very small spot size using electrostatic or magnetic lenses. generally, lenses are used is the electrostatic. The fine beam is scanned or rastered on the sample surface using a scan generator and back scattered electrons are collected by a specified detector.

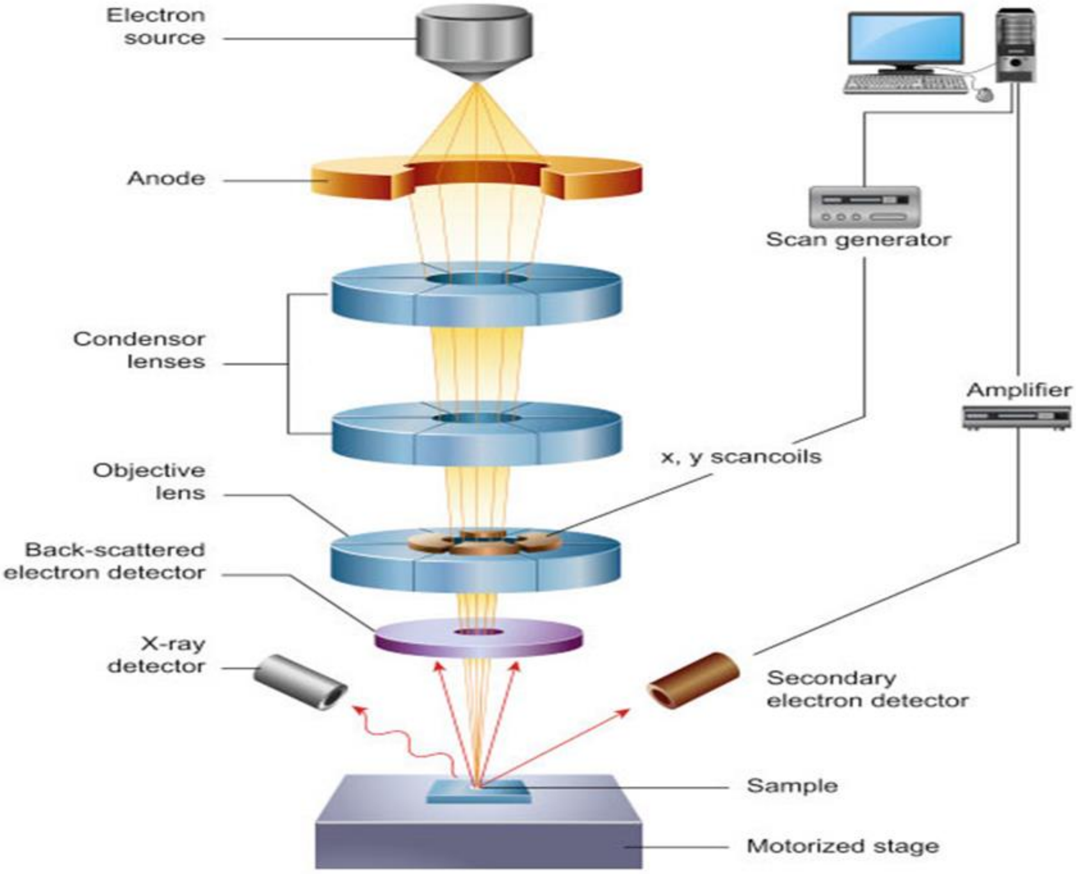

Fig. 2.4.1 Schematic diagram of SEM

In generally two types of microscopy are available one is optical microscopy and other is scanning electron microscopy. The former is oldest one. It is also called light microscopy. It is differs from SEM,

1. The main principle of work in OM is the light, unlike SEM which depend up on electron emission.
2. Simple OM has only one lens while compound OM has two lenses.
3. Magnification of modern OM reaches to the range between 400-1000 times the original sizes which is very low compared to the SEM.
4. SEM more expensive than OM and cannot be easily maintained

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CHAPTER 3

INTRODUCTION TO ZINC OXIDE NANOPARTICLES

3.1. ZnO NANOPARTICLE

- Chemical formula: ZnO
- Group : Zinc 12 and Oxygen 16
- Elecrtonic Configuration: Zinc [Ar]$3d^{10}4s$
- : Oxygen [He]$2s^{2}2p^{4}$
- Shape :Hexagonal
- Density :5.61g/cm^{3}

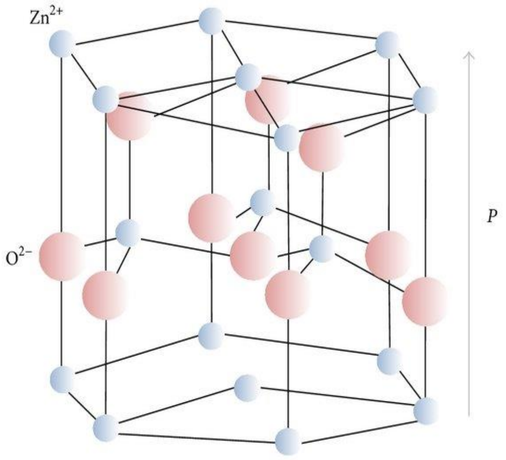

Fig.3.1. Hexagonal structure of zinc oxide

Zinc oxide nanoparticles (ZnO-NPs) are one of the metal oxide nanomaterials and a useful and versatile(flexible) inorganic substance because of its distinct physical and chemical properties. Strong chemical stability, a widening radiation absorption spectrum, a strong electrochemical coupling coefficient, and great photostability are all characteristics of zinc oxide nanoparticles. Zinc oxide is a formula of an inorganic compound its formula is ZnO. It is a whitish powder and it is not soluble in water. Zinc oxide nanoparticles or ZnO-NPs, are extensively produced and used as a zinc nutrient in metals and a variety of commercial and industrial products, such as cement, plastics, glass, ointments, lubricants, batteries, ferrites, fire retardants, and sunscreens.

3.2. Properties Of Zinc Oxide Nanoparticles

Zinc oxide is the fifth most common element in the crust of the earth. Comparatively, Zinc oxide is less toxic and inexpensive than other nanoparticles of metal oxide. Zinc oxide nanoparticles are a perfect applicant for applications of research in biomedicine, seeing their antifungal, antibacterial, and anticancer activities that influence their capabilities. zinc oxides do not catch fire when they are nanoscale particles. The key information regarding zinc oxide nanoparticle is that their characteristics and attributes vary depending on how they are made.

The majority of zinc oxide is created artificially, however it does occur as the mineral 'zincite'. Zinc oxide nanoparticles are lower than 100 nm in size because their diameter is less than 100 nm. Zinc oxide nanoparticles are sufficiently large on the surface relative to their size and strong catalytic activity. The precise chemical and physical characteristics of zinc oxide nanoparticles are altered by the method of synthesis.

Because of their exceptional ransparency to visible light and ultraviolet (UV) absorption properties, zinc oxide (ZnO) nanoparticles make excellent sunscreens. Humans come into contact with Zinc oxide on a regular basis via a variety of sources, including cosmetics, food, and other products. Zinc oxide is the fifth frequent element in the earth’s crust and an important one, yet it can be harmful to humans in excessive concentrations. Zinc oxide dissolves in human body just once. Foods are enriched with zinc acetate (E650), a type of zinc, to help avoid human zinc deficiencies. Compared to other metal oxide nanoparticles, zinc oxide (ZnO) is less costly, less poisonous, and more biocompatible. It is demonstrated that their photocatalytic activity is high.

Zinc oxide (ZnO) is a broad band gap semiconductor with a 3.37 eV energy gap at room temperature. These wide-bandgap characteristics of a semiconductor ZnO produce ROS to kill the cells, and their inherent photoluminescence properties are appropriate for applications of biosensing. At temperatures above 1300 °C evaporation occurs, and at 1800 °C sublimation occurs. Therefore it does not melt because there is no liquid state; instead, it transitions directly from the solid state into the gas state.

Zinc oxide nanoparticles shape is solely determined by their synthesis method. They might be anything, including nanotubes or nanorods. Against gram +Ve bacteria, they are more active. ZnO nanoparticles has all of the qualities that make an antibacterial chemical suitable for human consumption, including nontoxicity, the ability to not react with food or containers, and tasteless or nice taste. Thus, these nanoparticles have use as a preservative in packaging, a medication delivery system, and an antibacterial agent. They disperse more readily into food, eradicate inside microbes, and shield people from disease. Out of all the metal oxide nanoparticles, zinc oxide nanoparticles exhibited the highest level of toxicity against various bacteria.

3.3. APPLICATIONS OF ZnO NANOPARTICLES

In terms of technical uses, zinc oxide is a versatile material. They are used in a variety of industries, including pharmaceutical and cosmetics. When an additive is put to rubber, it promotes the vulcanization process, which is used to make tires. ZnO nanoparticles have high conductivity, heat is therefore produced as the tires churn, but it is better removed thanks to the excellent conductivity of ZnO nanoparticles. Additionally, they serve as a catalyst in the chemical industry’s production of cosmetics.. ZnO nano particles are safe, affordable, and simple to make. Ceramics, glass, cement, rubber etc have all are made extensive use of ZnO powder as an in gradient.

1) Sunscreen

The most popular use of ZnO nanoparticle is sunscreen because of their excellent UV light-absorbing capacity and complete transparency to visiblr light because to a sufficiently broad band gap. It has been shown that they eliminate harmful germs from packaging and UV-protective materials like textiles. Zinc oxide tendency to absorb UV photons, which are subsequently reflected from tiny mirrors, makes it a fascinating choice for use as a physical UV filter in sunscreen. Sunscreen should have a sun protection factor (SPF) of more than 25 to provide effective protection against the sun’s rays.

2) Anti-Cancer

They have been demonstrated to be the most toxic to cancer cells and the least damaging to human cells. The cytotoxicity of NP is influenced by Surface capping and selective toxicity. Different stress mechanisms were demonstrated against cancer cell lines, irrespective of the size and surface chemistry of ZnO nanoparticles. They are utilized to load activate medicines because they exhibit innate anticancer properties. Because ZnO Nanoparticles contain certain moieties, they can target and select cancer cells more effectively. The transcription level confirms cellular apoptosis. Combination therapy has been shown to be more cytotoxic against tumor cells.

3) Antidiabetic Activity

Insulin production, secretion and storage all depend on zinc. The ultrastructural and structural improvements demonstrate that ZnO nanoparticles can cure pancreatic injury brought on by diabetics. The biochemical normalization of blood and serum insulin serves as confirmation.

4) Antibacterial Activity

ZnO Nanoparticles are effective against various foodborne pathogens and food pollutants due to their string antibacterial properties. The degree to which they can cause oxidative stress determines these outcomes. ZnO releases Zn+ ions, which bind to the thiol group of the respiratory enzyme and therefore stop it from working. ZnO Nanoparticles have an impact on the cell membrane and cause ROS to develop. Thus, Zn+ is absorbed by bacterial cells upon coming into contact with ZnO nanoparticles. This limits the respiratory enzyme’s activity, produces ROS, and subsequently releases free radicles that results in depolarization and protein leakage. ROS generation and DNA damage were also discovered. These findings suggest that ZnO nanoparticles and antibiotics work cooperatively to cure bacterial diseases.

5) **Antifungal Activity**

ZnO nanoparticles from M. oleifera shown toxicity against isolates of Alternaria saloni and Sclerrotium rolfii two plant pathogens. Zinc oxide nanoparticles were shown to be toxic against plant and food diseases and to exhibit antifungal activity against a variety of fungal species. So, ZnO Nanoparticles can therefore be applied to the food safety and agriculture sectors. It has been noted that the inhibition of fungal energy is influenced by ZnO Nanoparticles concentration. If ZnO Nanoparticles are excited by visible light, yeast cells may be destroyed.

6) Anti-Inflammatory Activity

ZnO that is nanosized can only penetrate the deep layer of sensitive skin by reducing localized skin irritation and stimulating the formation of IgE antibodies throughout the body. Zinc's potent antibacterial qualities and ability to promote epithelialization have made wound-healing ZnO nanoparticles an efficient addition to wound dressings. ZnO nanoparticles were added, and an increase in the diameter of the inhibitory zone was seen for both S. aureus and E. coli.

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CHAPTER 4

METHODOLOGY, RESULTS AND DISCUSSIONS

4.1. SYNTHESIZE OF ZINC OXIDE NANOPARTICLE

4.1.1. CHEMICAL PRECIPITATION METHOD

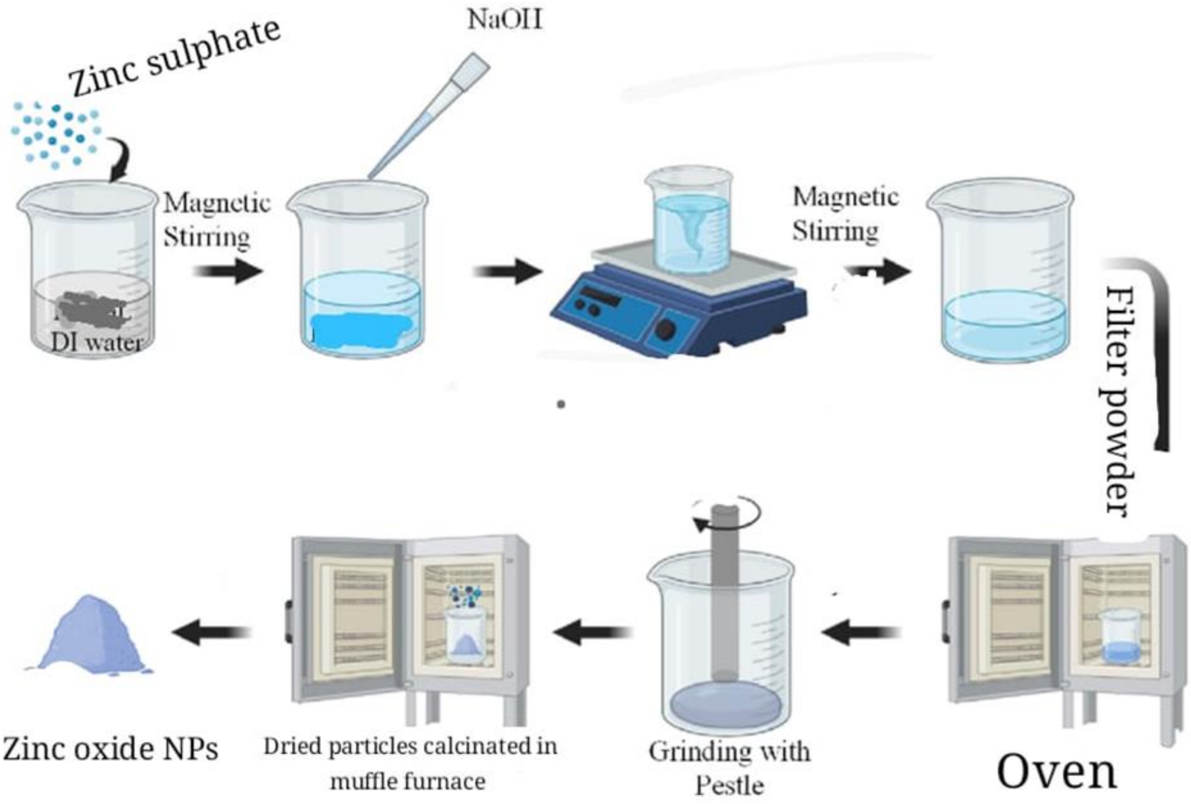

Figure 4.1 Synthesis of ZnO nanoparticle using chemical precipitation method

Zinc Oxide nanoparticles can be synthesized by a many of physical, chemical, and biological methods including vapor deposition, arc plasma method, thermal evaporation method etc. While sol-gel process, precipitation and hydrothermal technique are examples of some chemical methods to synthesize of ZnO nanoparticles.

In our project we use the chemical precipitation method in order to create zinc oxide nanoparticles. The structural and optical characteristics of zinc oxide nanoparticles were observed by X-ray diffraction analysis, photoluminescence and absorption.

4.1.2. Preparation of Pure Zinc Oxide

.2M zinc sulphate (2.9g) was made in 50 milliliters of distilled water while stirring. .4M sodium hydroxide solutions were made by dissolving weighed NaOH pellets in 50ml distilled water while stirring. Adding NaOH solution into former solution drop by drop while continuously stirring. After, filter the solution then washed two times using distilled water and acetone alternatively. Then the precipitate will be dried in a hot air oven for overnight at temperature 90^0C. The white product crushed to obtain fine powder. The Zinc oxide nanoparticles powder then treated by calcinations in the furnace at a temperature of 500^0 C for 3 hr.

4.1.3. Preparation of Fe Doped Zinc Oxide

2.85g zinc sulphate was made in 50 milliliter of distilled water while stirring. 0.016g iron chloride added into the solution of zinc sulphate, 8g of NaOH solutions were made by dissolving NaOH pellets in in 50 ml distilled water while stirring. By adding NaOH solution into former solution drop by drop while continuously stirring. Do the same procedure above mentioned. Then finally we get the Fe doped zinc oxide nano powder.

4.2. RESULTS AND DISCUSSIONS

4.2.1. Structural X-ray Diffraction (XRD)

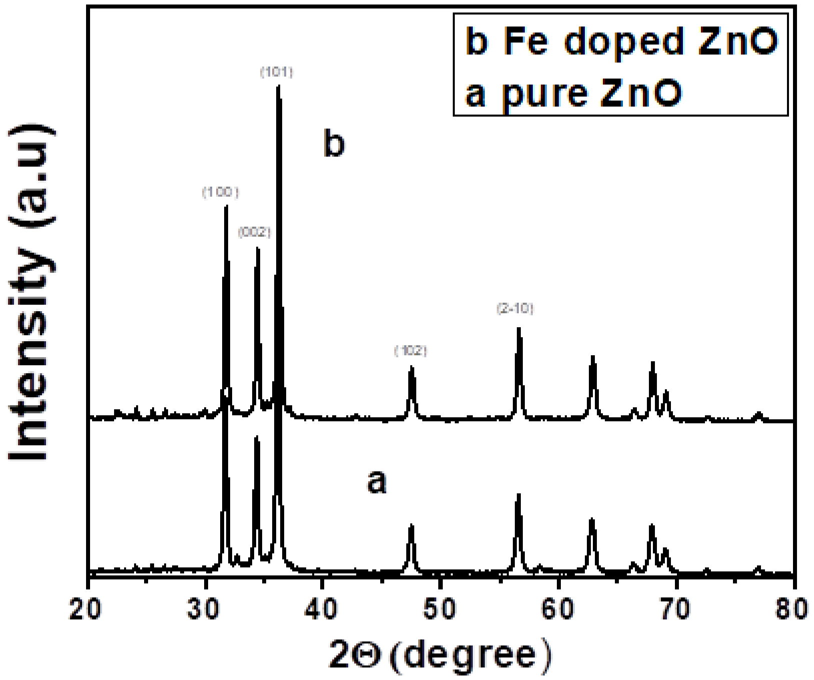

Fig.4.2.1 XRD peak of ZnO and ZnO-Fe

The crystallographic structure of the synthesized sample was analysed using X-ray diffraction (XRD) method and it as shown in Fig. 3.1. From XRD we can determining the crystal structure, lattice parameter, stress, and grain size of the Zinc oxide nanoparticles. The phase purity of synthesized zinc oxide nanoparticles was confirmed by XRD studies of the nanoparticles.

The XRD pattern of pure ZnO shows diffraction peaks at values 31.73^0,34.39^0,36.21^0,47.51^0,56.56^0 and 62.81^0 Corresponding to the lattice planes (100), (002), (101), (102), (2-1 0) and (103). In case of Fe doped ZnO, the diffractions occur from 31.78^0,34.43^o,36.26^0,47.51^0,56.60^0 and 62.90^0 respectively.

Careful analysis of the data shows the maximum intensity peaks obtained for our sample matches with those for the Zinc Oxide (hexagonal) standard sample value provided by the JCPDS file no: 00-900-4179. That is, from the peak values of XRD pattern confirm the presence of ZnO nanoparticles with hexagonal structure.

We can calculate grain size, using Debye- Scherrer equation [1]

1. $D = 0.9\lambda/\beta \cos\theta$

Where D denotes the particle size, λ is the wavelength of X -ray used (λ =1.5406A^0) and β is the full width at half maximum, θ is the glancing angle.

The average Grain size of ZnO and Fe doped ZnO is discovered to be 23.83 nm and 24.76 nm respectively.

The cell parameters are obtained by using the relation,

$$\frac{1}{d_{hkl}^{2}} = \frac{4}{3}\left(\frac{h^{2}+hk+k^{2}}{a^{2}}\right) + \frac{l^{2}}{c^{2}} \qquad (2.4)$$

For pure zinc oxide, we get the values as a =b =3.2533 A° and c =5.2073 A° and for Fe doped zinc oxide, a =b =3.2494 A° and c = 5.2038 A°.

4.2.2.W-H plot of ZnO and ZnO-Fe

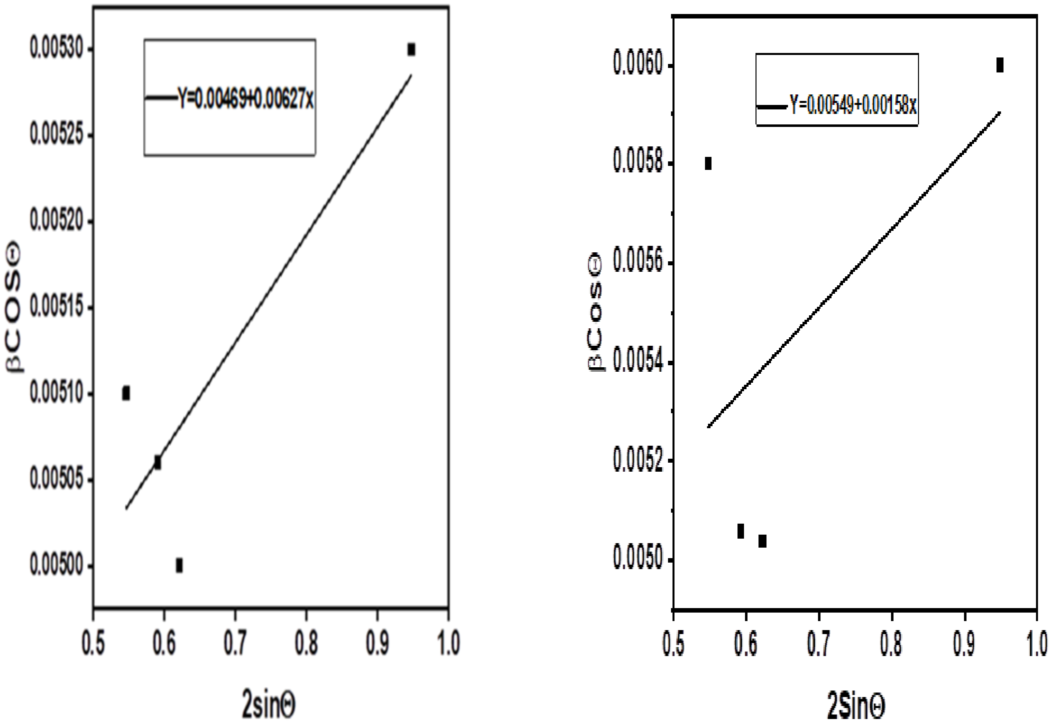

Fig.4.2.2 W-H plot of pure ZnO and Fe doped ZnO

Using W-H (Williamson – Hall) plot we can calculate the size and strain of the given crystals sample. Based on the linear fit to the data, the crystalline size was approximated from the

Y-intercept, and the strain, from the slope of the fit.

Williamson – Hall equation is [2]

$$\beta\cos\theta = 0.9/D + 2\xi\sin\theta$$

Crystalline size (D) of ZnO obtained is **29.55 nm** and ZnO - Fe is **25.24**

4.2.3.NR Plot of Pure ZnO

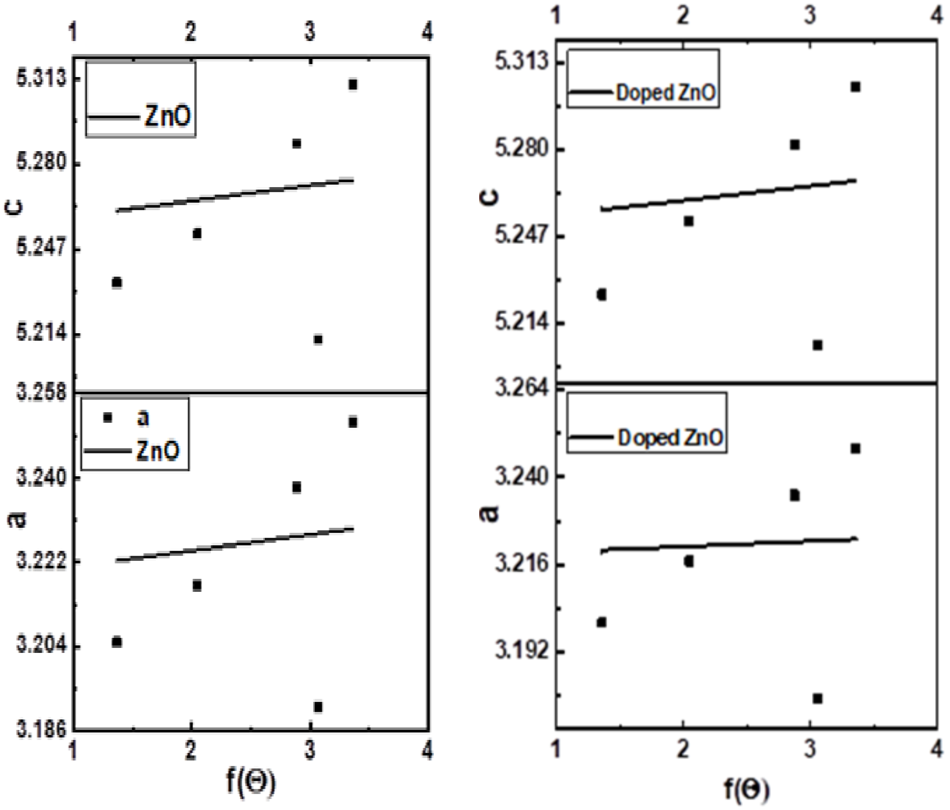

Fig.4.2.3 NR plot of pure ZnO and Fe Doped ZnO

Using NR (Nelson-Riley) plot we can determine the lattice parameter of the synthesised sample. From the fit to the data. From above plot lattice parameter of pure ZnO is, a=3.221, c=5.259 and ZnO-Fe is a=3.219, c=5.255.

Table1: Structural parameters of ZnO and ZnO - Fe

		PURE ZNO	FE DOPED ZNO
Average grain size	From Debye Scherrer	26.11	25.41
	WH PLOT	29.55	25.24
Lattice parameter	From equation	a=b=3.2533 c=5.207	a=b=3.2494 c=5.2038
	NR PLOT	a=b=3.221 c=5.259	a=b=3.219 c=5.255
Strain		0.00627	0.00158

4.3. PHOTOLUMINESCENCE (PL)

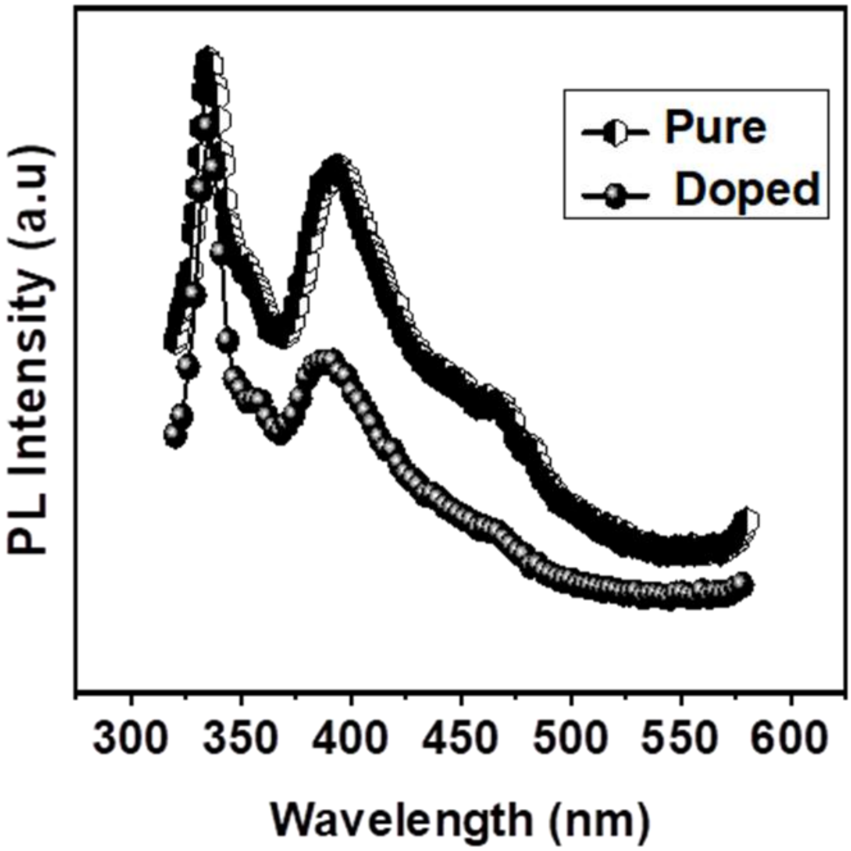

Fig.4.3. Photoluminescence spectrum of ZnO and Fe doped ZnO

The optical characteristics of zinc oxide nanoparticles were characterized by the PL for an excitation wavelength of 325nm and it is depicted in fig 3.3. The result shows the PL spectra of zinc oxide nanoparticles shows two major emission peaks, UV emission peak at 325nm and a broad UV emission peak at 390nm. It found that all the PL spectra were composed of two emission band which consist of Near band edge emission (NBE) peak lying at UV transition region and a strong visible light emission peak related to the deep level defect (DLD) transition. Based on the PL result it can be found that the UV emission peak in doped samples were suppressed severally because of Fe doping. Fe doping causes decrease in the UV emission band because of the increase in the density of defects /

oxygen vacancies (intrinsic defects). That is photogenerated electrons preferentially occupy the Fe ion induced trap centres results in the quenching of luminescence [3,4].

When Fe ions are doped into zinc oxide it can actually affect the electronic and optical properties of the sample. The presence of Fe introduces additional energy levels and defects in the zinc oxide crystal structure. These types of defects can act as non- radiative recombination centers, meaning they trap and dissipate some of the energy instead of emitting it as light. The blue to red with green emission that was created in the Fe-doped ZnO nanoparticles can be explained by the Fe-ion's energy level transitions quenching the violet and UV emission. Hence the Fe doped Zinc oxide behave as a quencher [5].

4.4 SEM ANALYSIS

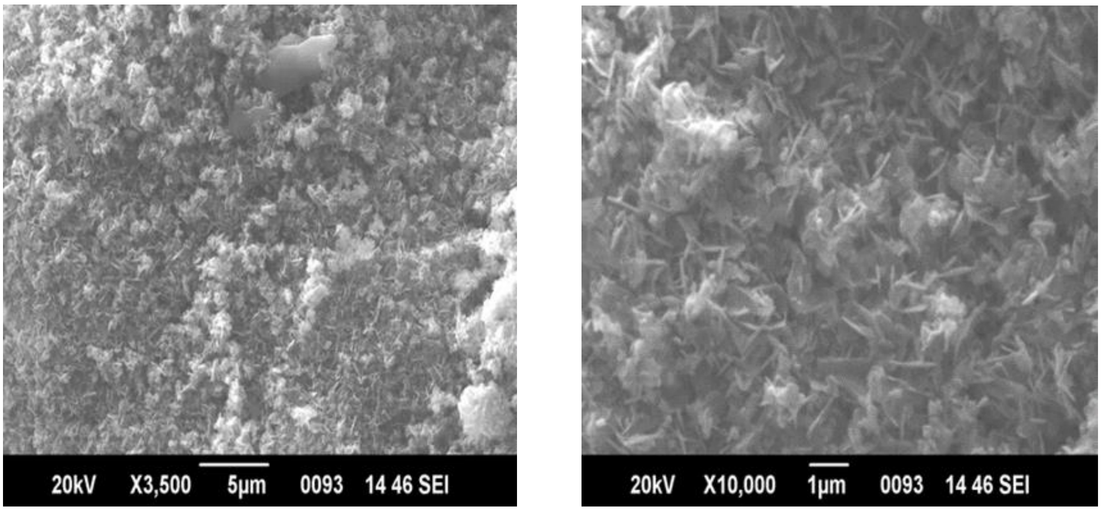

Fig.4.4.SEM image of ZnO

The ZnO nanoparticles were analysed by SEM to observe their morphology and structure. SEM image of pure ZnO samples are shown in figure 5.SEM images were seen in different magnification ranges from 1µm to 10µm. Images at lower magnification shows that the particles are agglomerated and complete separation is not occur. Images at higher magnification can reveal finer details of individual particles. In 1µm and 2µm magnification particles shows flower like (complex, petals – like) surface morphology can be seen. In the image of 1µm magnification nanometre ranges shows “size of rod like structure”.

4.5. UV-Vis Absorption Spectroscopy

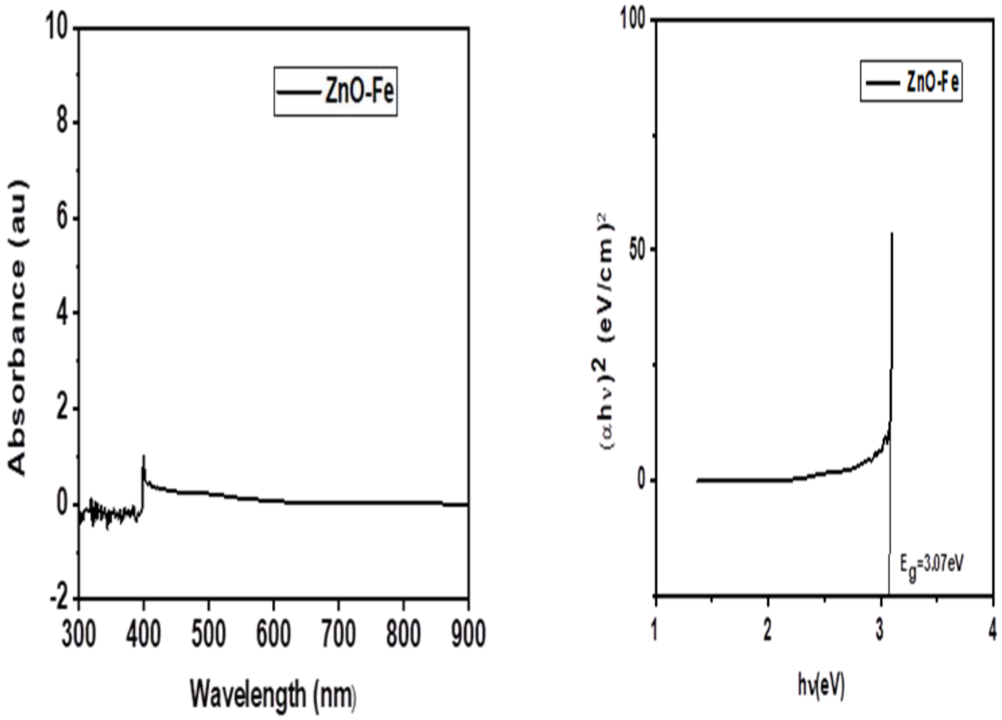

Fig.4.5.UV-Vis Absorbance spectra of ZnO-Fe and Tauc plot diagram of ZnO-Fe Nanoparticles

From the UV-Vis absorption spectra the sample absorb the radiation in the UV range about 398nm. This is the characteristic absorption peak of ZnO-Fe.

The band gap Energy of ZnO nanoparticles is calculated by Tauc plot diagram, by extrapolating the linear region of the curve between (h ν) and (α h ν)^2 shown in above figure. Where α is the absorption coefficient and ν is the frequency. The Energy band gap of synthesised Fe doped zinc oxide nanoparticles was 3.07eV.

4.6 CONCLUSION

This report presents a systematic study of ZnO and ZnO-Fe nanoparticles synthesised using the chemical precipitation method and their characterizations using XRD, UV-Vis spectroscopy, Photoluminescence and Sem.

The XRD analysis revealed the hexagonal structure of ZnO and ZnO-Fe nanoparticles with specific peaks corresponding to various lattice planes. The average crystalline size of nanoparticles was determined to be 26.11 and 25.41 respectively using Debye Scherrer equation. The crystalline size can also measure using W-H plot[table1].

The lattice parameters are also calculated from the XRD pattern. It is observed that for Zno a=b=3.2533 & c=5.2073 and for ZnO-Fe a=b=3.23.2494 & c= 5.2038. It can also determine using N-R plot[table1].

Results shows the PL spectra of ZnO nanoparticles shows two major emission peaks, UV emission peak at 325nm and broad UV emission peak at 390nm. Due to Fe doping, UV emission peak suppressed because of intrinsic defect.

SEM results shows, in 1µm and 2µm magnification particles shows flower -like surface morphology. In the image of 1µm magnification nanometre ranges shows 'size of rod like structure'.

UV absorption studies of the sample ZnO-Fe shows, band gap energy is 3.07 eV.

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