

STRUCTURAL, MORPHOLOGICAL AND OPTICAL PROPERTIES OF TIN SULFIDE NANO PARTICLES

*Submitted in partial fulfilment of the requirements for the award
of Bachelor of Science in Chemistry*

By

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This is to certify that this Project Report is the bonafide work of **Mr.V.KARTHIKRAJA (40030015) & Mr.N.KESAVAN (40030018)** who carried out the project entitled **STRUCTURAL, MORPHOLOGICAL, AND OPTICAL PROPERTIES OF TIN SULFIDE NANO PARTICLES** under my / our supervision.

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DECLARATION

We **V.KARTHIKRAJA AND N.KESAVAN** student of **SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY** hereby declare that the Project Report entitled **“STRUCTURAL,MORPHOLOGICAL, AND OPTICAL PROPERTIES OF TIN SULFIDE NANO PARTICLES”** done by us under the guidance of **Dr.S.SUNITHA, M.Sc., M.Phil., Ph.D.** and **Dr.G.MURUGADOSS, M.Sc., Ph.D. Scientist-E** at **International Research Centre, Sathyabama Institute of Science and Technology, Chennai** is submitted in partial fulfillment of the requirements for the award of **Bachelor of Science degree in Chemistry.**

DATE: 08/03/2023

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V. KARTHIKRAJA

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STRUCTURAL, MORPHOLOGICAL AND OPTICAL PROPERTIES OF TIN SULFIDE NANO PARTICLES

VISION

**“TO EXPLORE NEW FRONTIERS IN CHEMICAL SCIENCE THROUGH
INNOVATION AND COLLABORATION”**

ABSTRACT

This project aims to study the optical and structural properties of Tin Sulfide nanoparticles. Tin Sulfide (SnS_2) nanoparticles have been gaining attention due to their potential applications in optoelectronic devices and solar cells. In this project, we focus on the optical and structural properties of SnS_2 nanoparticles, with a particular emphasis on their photoluminescence. We address the issues of surface passivation, size effects, and surface modification of SnS_2 nanoparticles. We also discuss the potential applications of SnS_2 nanoparticles and their use as a material for light harvesting and energy storage.

Key words :Tin Sulfide, optoelectronic devices, photocatalyst, photoluminescence.

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LIST OF ABBREVIATION

- 1) nm – Nanometer
- 2) NPs – Nanoparticles
- 3) XRD - X-Ray Diffraction
- 4) UV – Ultra Violet
- 5) SEM – Scanning Electronic Microscope
- 6) FT-IR – Fourier Transform Infrared
- 7) SnS - Tin Sulfide
- 8) 1D - One Dimensional
- 9) 2D – Two Dimensional
- 10) 3D – Three Dimensional
- 11) NaOH -Sodium Hydroxide
- 12) DI - De-Ionized Water
- 13) PVP - Polyvinyl pyrrolidon

CHAPTER-1

INTRODUCTION

Tin sulfide (SnS) is a semiconductor material with a unique combination of structural, morphological, and optical properties that make it a promising candidate for various technological applications, including solar cells, thermoelectric devices, and optoelectronic devices.

Structurally, SnS has a layered crystal structure, with each layer consisting of tin atoms sandwiched between two sulfur atoms. This structure gives rise to its anisotropic properties, with properties varying with the direction of the crystal axes.

Morphologically, SnS can exist in various forms, including thin films, nanoparticles, and nanowires, which exhibit different physical and chemical properties. The synthesis of SnS in different morphologies has been extensively studied, and various methods, such as chemical vapor deposition, hydrothermal synthesis, and solvothermal synthesis, have been developed to control its morphology.

The optical properties of SnS nanoparticles are another Important aspect that makes them attractive for various applications. SnS nanoparticles exhibit a direct bandgap in the range of 1.0–1.3 eV, depending on the size and morphology. This bandgap makes SnS nanoparticles suitable for various optoelectronic applications, such as solar cells, photodetectors, and light-emitting diodes. Additionally, the absorption coefficient of SnS nanoparticles is high, making them efficient absorbers of light.

Overall, the unique structural, morphological, and optical properties of SnS make it an exciting material for research and development in various technological fields, particularly in renewable energy and optoelectronics..

1.1 NANO PARTICLES

These days, we live in an escalated advancement of science known as nanotechnology. The normal size of particles framing a sample in the range of 1–1000 nm (1 nm = 10^{-9} m). There are so meanings of nanoparticles. One of them says that, whose at any rate one measurement is more than 100 nm, then such material can be called as “nanomaterial”.

Another definition “nanomaterial” as a material which after “miniaturization” to nanoscale uncovers changed physicochemical properties in contrast with the mass item (naturally visible). The nanoparticles displays a novel physical, chemical and biological properties at nanoscale contrasted with their specific particles at higher scales.

This happens because of a larger surface area to the volume, increase in reactivity or stability in a chemical reaction, improved mechanical strength, and so on. These properties of nanoparticles have prompted its utilization in different applications.

The nanoparticles differ from shapes, sizes and structure. It can vary from 1 nm to 100nm in size.

The surface can be uniform or unpredictable with surface varieties. Some nanoparticles are translucent or shapeless with single or multi gem solids either free or agglomerated. Various synthesis techniques are either being created or improved to upgrade the properties and lessen the production costs.

A few techniques are adjusted to accomplish measure explicit nanoparticles to increment their physical, chemical, optical and mechanical properties. A huge advancement in the instrumentation has prompted an improvement in nanoparticle characterization and application.

Based on the application like imaging, diagnosis or treatment various kinds of nanoparticles have been proposed, some of them are utilized for multiple purposes.

Nanoparticles come under two main categories: organic and inorganic nanoparticles. The primary group includes micelles, dendrimers, liposomes, hybrid and minimized polymeric nanoparticles. The second group includes fullerenes, quantum dots, silica and gold nanoparticles.

1.2 TYPES OF NANOPARTICLES

Nanoparticles can be classified into different types according to the size, dimension, Morphology, physical and chemical properties. Some of them are carbon-based Nanoparticles, ceramic nanoparticles, metal nanoparticles, semiconductor Nanoparticles, polymeric nanoparticles and lipid-based nanoparticles.

1.2.1 ZERO-DIMENSIONAL NANOPARTICLES

Zero-dimensional nanoparticles are the most common type of nanomaterials with dimensions within nanoscales (the dimension of nanoparticles is larger than 100nm). These nanoparticles are point like particles (i.e. as small as a point). The most common examples of these particles are quantum dots, hollow spheres, nano lenses.

1.2.2 ONE -DIMENSIONAL NANOPARTICLES

These types of nanoparticles are at least one dimension larger than nanoscales and other Dimensions are within nano range. The most common examples of one-dimensional nanoparticles Are nanofibers, nanotubes, and nanorods.

1.2.3 TWO-DIMENSIONAL NANOPARTICLES

These types of nanoparticles are two dimensions larger than nanoscale (100nm). The most Common examples of this class are nanofilms,

nanolayers, and nanocoating. This class of Nanomaterials have plate-like structures.

1.2.4 Three-Dimensional Nanoparticles

Three-dimensional nanoparticles have all three dimensions larger than 100nm but their components are Below 100nm in size. Nano range particles come together to form three dimension nanoparticles. These Particles are generally nonporous in nature and have many applications. The most common examples are nanocomposites, bundles of nanofibers, multi nanolayer-type Structures.

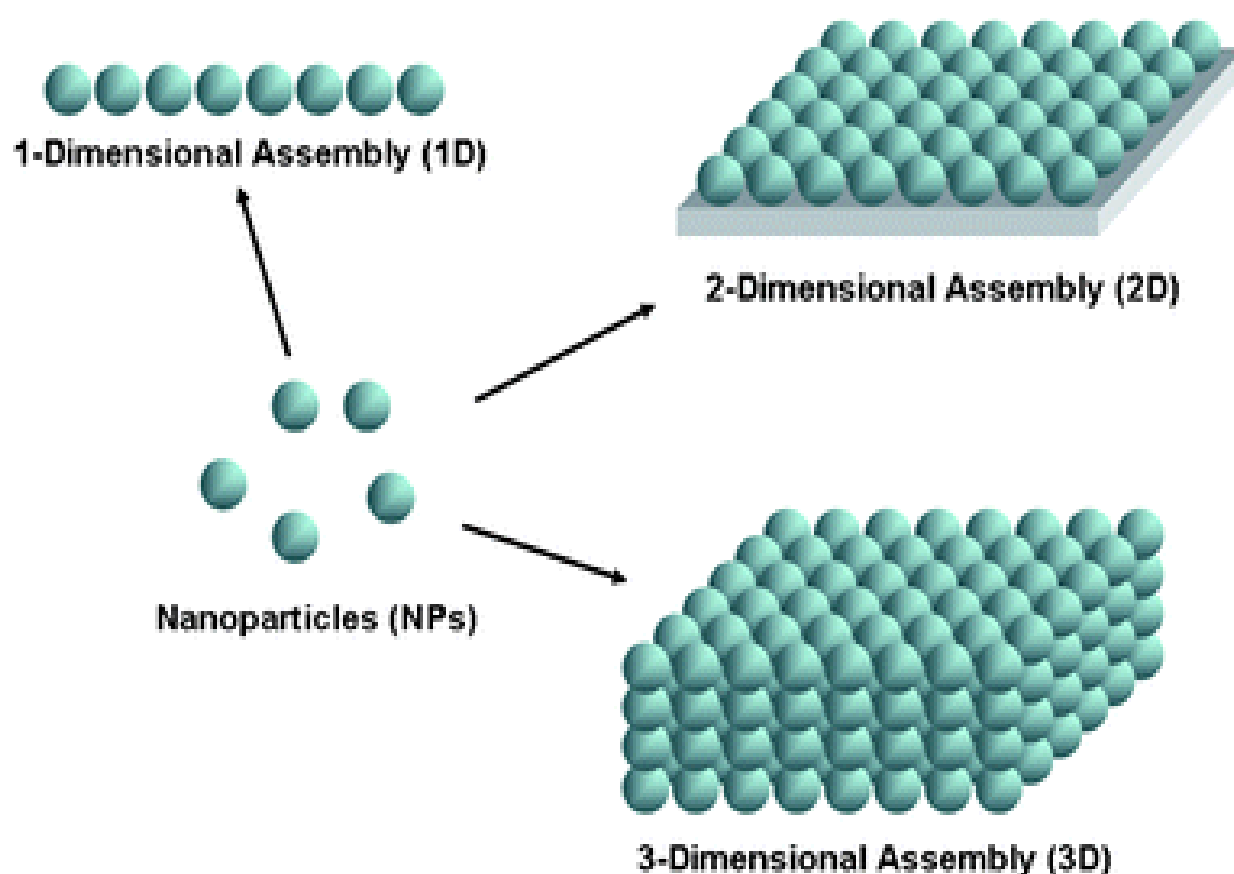


Figure 1.1 Types of nanoparticles in dimensional

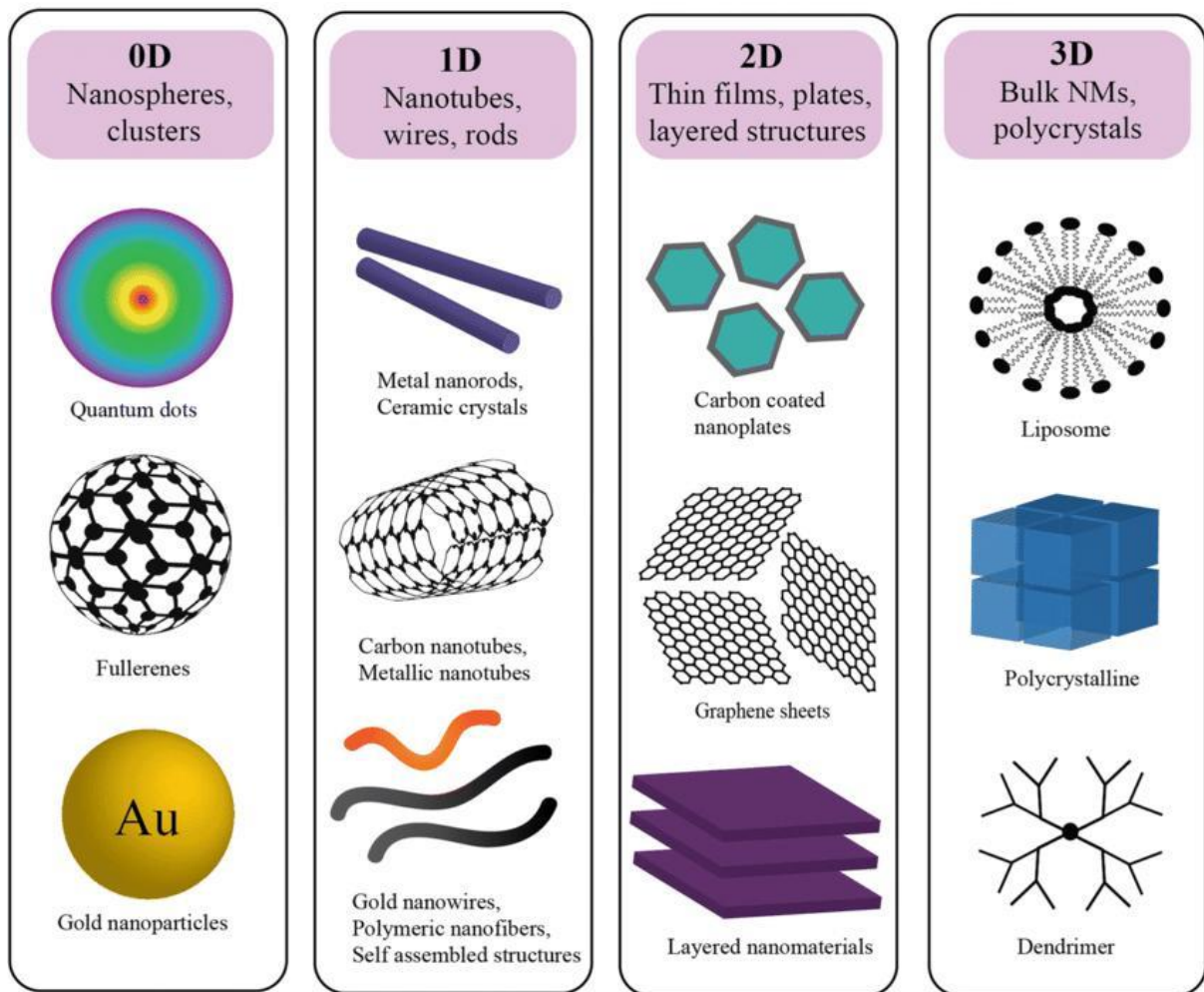


Figure 1.2 Types of Nanoparticle in dimensional and it's examples

1.3 CLASSIFICATION OF NANOPARTICLES

Nanoparticles can be classified into different types according to the size, morphology, physical and chemical properties. Some of them are carbon-based nanoparticles, ceramic nanoparticles, metal nanoparticles, semiconductor nanoparticles, polymeric nanoparticles and lipid-based nanoparticles.

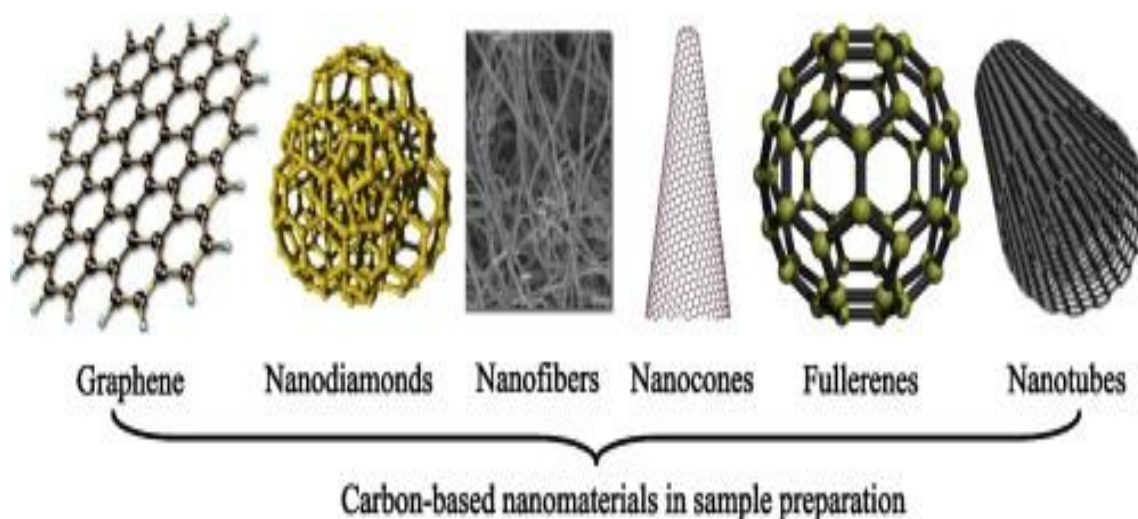
1.3.1 CARBON BASED NANOPARTICLES

Carbon-based nanoparticles are of two main materials: carbon nanotubes (CNTs) and fullerenes. CNTs are nothing but graphene sheets rolled into a tube. These materials are mainly used for the structural reinforcement as they are 100 times stronger than steel.

Fullerenes contain nanomaterial that are made of globular empty enclosure like allotropic structures of carbon because of their electrical conductivity, high strength, structure, electron affinity, and flexibility. These materials possess pentagonal and hexagonal carbon units, while every carbon is sp^2 hybridized. CNTs are tubular, elongated, 1–2 nm in measurement. These can be anticipated as metallic or semiconducting dependent on their measurement helicity. These resemble graphite sheet rolling upon itself.

The sheets can be single, double or numerous walls and in this way, they are named as single-walled (SWNTs), double walled (DWNTs) or multi-walled carbon nanotubes (MWNTs). Individually, they are synthesized by carbon precursors, by electric arc on metal nanoparticles. They are synthesised by Chemical Vapour Deposition (CVD).

Because of their unique physical, chemical and mechanical properties, these materials are utilized in various applications like filler and as in organic and inorganic catalyst.



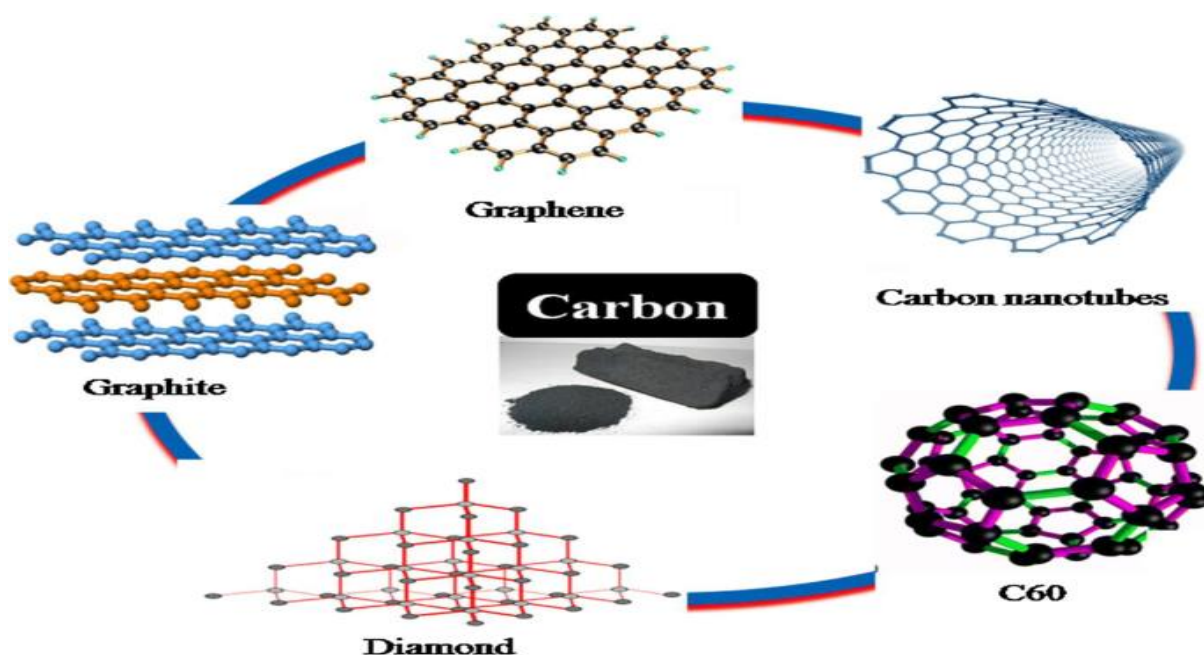


Figure 1.3.1: carbon based nanoparticles

1.3.2 CERAMIC NANOPARTICLES

Ceramic nanoparticles are made up of oxides, carbides, carbonates, and phosphates and are Inorganic solids. These nanoparticles are chemically inert and heat resistant. They can be Used for photocatalysis, dye photodegradation medication administration, and imaging. Ceramic nanoparticles can be used as a good drug delivery agent by manipulating several of Their properties, such as size, surface area, porosity, surface to treat a variety of ailments, Including bacterial infections, glaucoma, and cancer.

1.3.3 METAL NANOPARTICLES

Metal precursors are used to create metal nanoparticles. Chemical, electrochemical, or photochemical method can be used to create these nanoparticles. Metal nanoparticles are created chemically by reducing metal-ion precursors in solution with chemical reducing agents. These have a high surface energy and the ability to adsorb small molecules. These nanoparticles have application in research, biomolecule detection and imaging, and environmental and bioanalytical applications.

For example, gold nanoparticles are used to coat the sample prior to SEM analysis. This is typically done to improve the electronic stream, allowing us to obtain high-quality SEM images.

1.3.4 SEMICONDUCTOR NANOPARTICLES

Semiconductor-based nanoparticles are a class of nanomaterials that are composed of Semiconductor materials, such as silicon, cadmium selenide, and zinc oxide. These Nanomaterials have unique electronic and optical properties that make them useful in various Applications, such as electronics, energy production, and sensing. For example, cadmium Selenide nanoparticles are used in solar cells and LEDs due to their unique optical properties, while silicon nanoparticles are used in electronic devices. Zinc oxide nanoparticles are used in UV sensing and photo catalysis.

1.4 TIN SULFIDE NANOPARTICLES

Tin sulfide is a compound of tin and sulfur with the chemical formula SnS . It is a semiconductor material with a band gap of 1.3 eV. This exhibits high optical transparency, excellent electrical properties and good thermal stability. SnS nanoparticles (NPs) have been widely used in various applications such as solar cells, photodetectors, and photocatalysts and optoelectronic devices. SnS nanoparticles have been studied extensively in recent years, with a particular emphasis on their photoluminescence, due to its unique properties such as high absorption coefficient, low cost, and earth-abundant elements. When tin sulfide is synthesized in the form of nanoparticles, it exhibits even more enhanced properties due to the large surface area and quantum confinement effect.

SnS crystallises in the form of an orthorhombic structure, where each Sn(II) atom is coordinated to six S atoms – with three short Sn–S bonds within the surface and three longer Sn-S bonds connecting outer surface of the same layer.

(A)

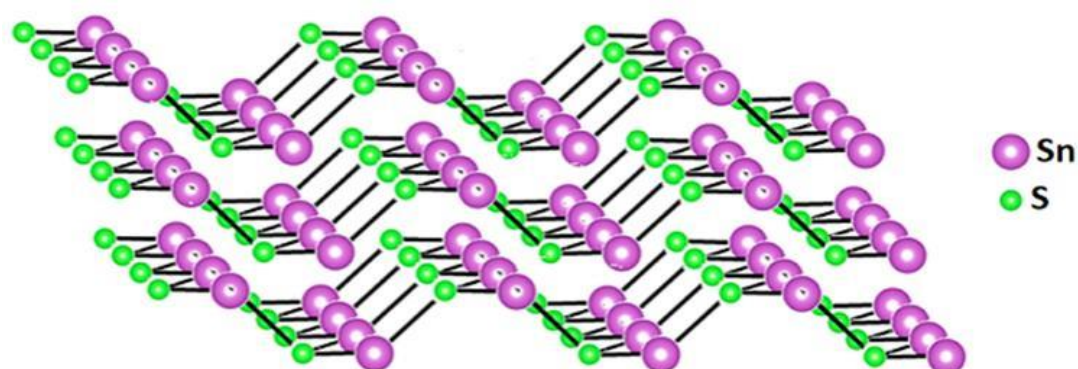


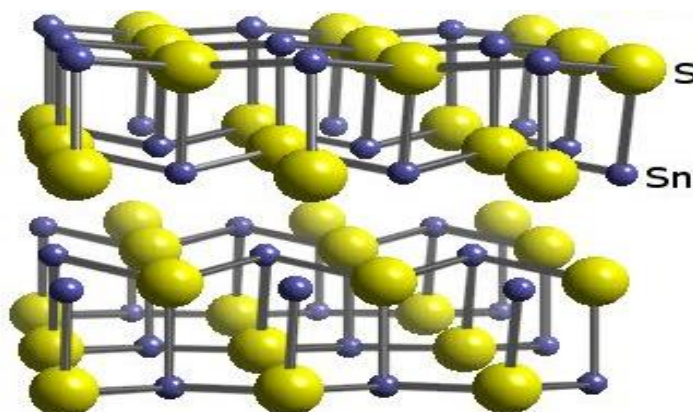
Figure 1.4: (A) Rough diagram of Tin (II) Sulfide

1.4.1 APPLICATION OF TIN SULFIDE

- **Solar Cells:** Tin sulfide nanoparticles have been explored as a promising material for photovoltaic devices. Their tunable bandgap, good electrical conductivity, and high absorption coefficient make them suitable for capturing solar energy efficiently. They can be used as an absorber layer in thin-film solar cells, potentially offering a cost-effective and sustainable alternative to traditional photovoltaic materials.
- **Optoelectronic Devices:** The optical properties of tin sulfide nanoparticles, such as their size-dependent bandgap and quantum confinement effects, make them suitable for optoelectronic applications. They can be utilized in light-emitting diodes (LEDs), photodetectors, and optical sensors. By controlling the size and composition of the nanoparticles, their emission wavelength and sensitivity can be tailored for specific device requirements.
- **Lithium-Ion Batteries:** Tin sulfide nanoparticles have shown promise as anode materials in lithium-ion batteries. They exhibit a high theoretical specific capacity, which can contribute to increasing the energy storage capacity of batteries. However, tin sulfide suffers from volume expansion during lithiation, leading to poor cycling stability. Researchers are actively working on addressing this issue to enhance the performance and lifespan of tin sulfide-based battery electrodes.

- **Gas Sensing:** Tin sulfide nanoparticles have been investigated for gas sensing applications, particularly for detecting toxic gases and environmental pollutants. The high surface area and reactivity of the nanoparticles make them sensitive to gas molecules, leading to enhanced gas sensing performance. By functionalizing the nanoparticle surface or incorporating them into composite materials, the selectivity and sensitivity of gas sensors can be improved.
- **Photocatalysis:** Tin sulfide nanoparticles have shown potential as photocatalysts for various environmental and energy applications. They can harness solar energy to drive catalytic reactions, such as water splitting, pollutant degradation, and hydrogen production. The unique electronic and optical properties of the nanoparticles enable efficient charge separation and utilization, enhancing the overall photocatalytic activity.
- **Biomedical Applications:** Tin sulfide nanoparticles have also been explored in biomedical applications. Their biocompatibility and potential for controlled drug delivery make them promising candidates for targeted therapy. Additionally, their optical properties can be utilized for bioimaging and diagnostic purposes, such as fluorescence imaging and biosensing.
- It's worth noting that the field of tin sulfide nanoparticles is still actively researched, and ongoing advancements may lead to the exploration of new applications and further optimization of existing ones.

Figure 1.4: (B) 3D diagram of Tin (II) Sulfide



CHAPTER -2

2.1 LITERATURE SURVEY

Nanoparticles are defined as particles with size in the range of 1 to 100 nm at least in one of the three dimensions. Because of this very small size scale, they possess an immense surface area per unit volume, a high proportion of atoms in the surface and near surface layers, and the ability to exhibit quantum effects. Nanoparticles exist with great chemical diversity in the form of metals, metal oxides, semiconductors, polymers, carbon materials, organics or biological. They also exhibit great morphological diversity with shapes such as spheres, cylinders, disks, platelets, hollow spheres and tubes, etc. Nanoparticles can be generated via a number of synthetic routes based on gas, liquid or solid phase approaches. The synthesized nanoparticles have to be surface modified in most cases, in order to passivate and stabilize them since their nanoscale renders them chemically very reactive and/or physically aggregative. The nanoparticles are also surface functionalized in order to meet the needs of specific applications (Nagarajan et al., 2008)

(Oskam et al., 2006) The synthesis of metal oxide nanoparticles is described in terms of precursor formation, nucleation, growth, and aging processes. The main parameters governing these processes are the solution properties, including the solvent viscosity, dielectric constant and the presence of adsorbing anions, the solubility of the Tin sulfide, and the Tin sulfide surface energy.

Over the last three decades nanoparticles have received an increasing amount of research interest. This is due to unique size dependent properties of nanoparticles, which are often thought as a separate and intermediate state of matter lying between individual atoms and bulk material (Schnid, 2004).

CHAPTER-3

AIM AND SCOPE

3.1 AIM

To study the structural, morphological, and optical properties of tin sulfide nanoparticles.

3.2 SCOPE

Characterization of the synthesized SnS nanoparticles is crucial for understanding their Properties and potential applications.

Determination of structural properties of Tin (II) Sulfide nanoparticles.

Determination of optical properties of Tin (II) Sulfide nanoparticles.

Morphology of Tin (II) Sulfide

Techniques such as FTIR, UV-visible, X-ray diffraction, scanning electron microscopy, and Spectroscopy can be used to study the size, shape, crystallinity, and surface properties of the nanoparticles.

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIAL

Tin chloride, Distilled water, Ethanol, Acetone, Sodium sulfide, Polyvinylpyrrolidone (PVP).

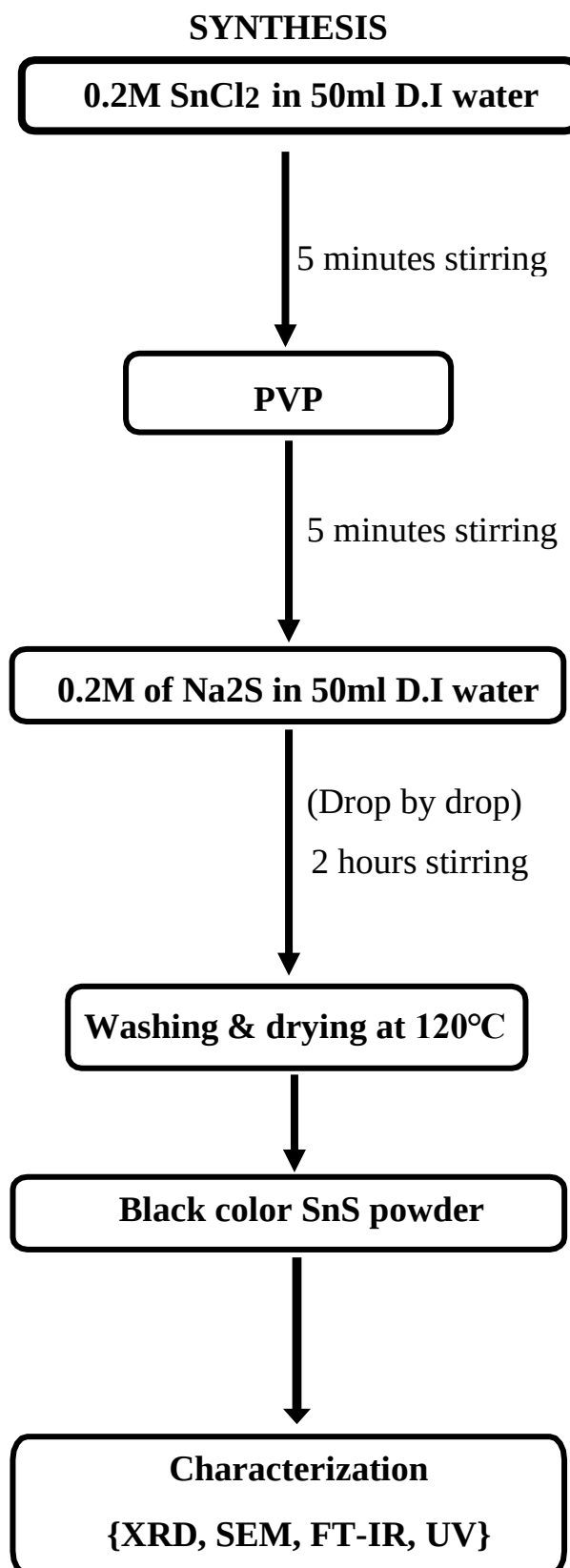
4.2 EXPERIMENTAL METHOD

Co-precipitation is a standard synthesis method for preparation of Tin sulfide nanoparticles. In This method a salt precursor, commonly nitrate, chloride, or oxychloride, is dissolved in water and then the corresponding Sulfide is precipitated by addition of a base such as sodium sulfide. Co-precipitation is a cost-effective and fast process and is easily implemented on a larger Scale for industrial application. It is an efficient method for synthesizing nanomaterials without requiring hazardous organic solvents. The Co-precipitation method was performed by using Tin salt like Tin (II) Chloride of 0.2M dissolved in double distilled water with continuous stirring at constant Temperature of 120°C. While stirring, 0.2M Na₂S solution was added till the pH of the solution Become 12. The stirring was continued for 2 hour at a constant temperature of 120°C. Black Precipitate formed was then filtered and washed with ethanol or acetone



Figure 4.2: CO PRECIPITATION METHOD

4.2.1 SYNTHESIS OF NANOPARTICLES



4.3 CHARACTERIZATION TECHNIQUES

Crystal structure, morphology, compositional information, and photocatalytic application of the synthesized nanoparticles were characterized using the following instruments. These include X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), Fourier Transform Infra-red spectroscopy (FTIR), and UV-Vis spectroscopy.

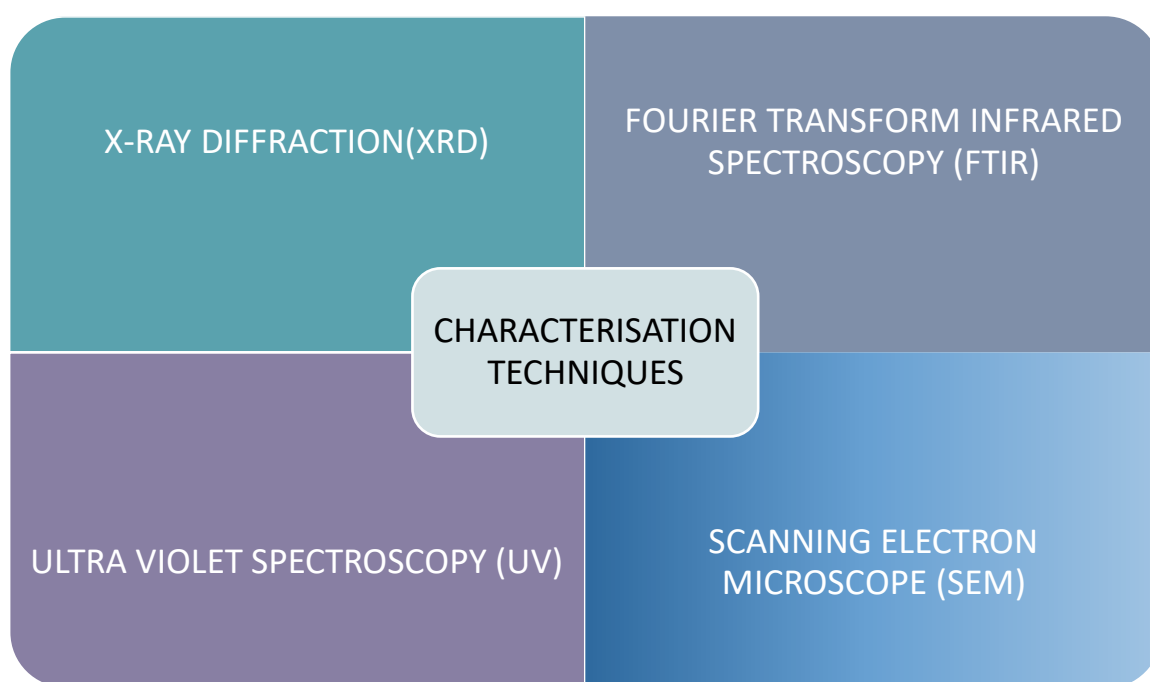


Figure 4.3: characterisation techniques of nanocomposite

4.3.1 X-RAY DIFFRACTION (XRD)

XRD is one the characterisation technique that reveals the structural properties of nanoparticles. It provides information on the phase, chemical composition, and symmetry of unit cells, crystallinity, lattice constant and properties of the nanoparticles. This technique is employed for the identification of both single and multiphase nanoparticles.

XRD is considered as one of the oldest non-destructive methods for the analysis of nanoparticles. The XRD pattern is influenced by the size and shape of the unit cell of the crystal. Consequently, properties of a material are dependent on their structure. Of about 90% solid materials are crystalline in nature and each crystalline has a significant X-ray diffraction pattern which can be used as fingerprint to detect the material.

Diffraction pattern is obtained only when the X-rays are interacted with the crystal. Information like symmetry of the unit cell, stress, strain, size of crystallite and growth orientation etc. are obtained from the diffraction patterns.

BRAGG'S LAW

When a material is irradiated with a monochromatic light of wavelength around the inter atomic distance of the material, light gets dispersed by the atoms and interfere to give rise to diffracted light with a higher intensity. The arrangement of atoms in the material is related to the intensity of the scattered light. If wavelength of light is λ and the incident angle is θ and the constructive interference is obtained at an angle 2θ from the incident beam of light if Bragg's equation is satisfied. Bragg's equation is given by,

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

Where,

n is the diffraction order,

d is the distance between the diffracting planes and

θ is the Bragg's angle.

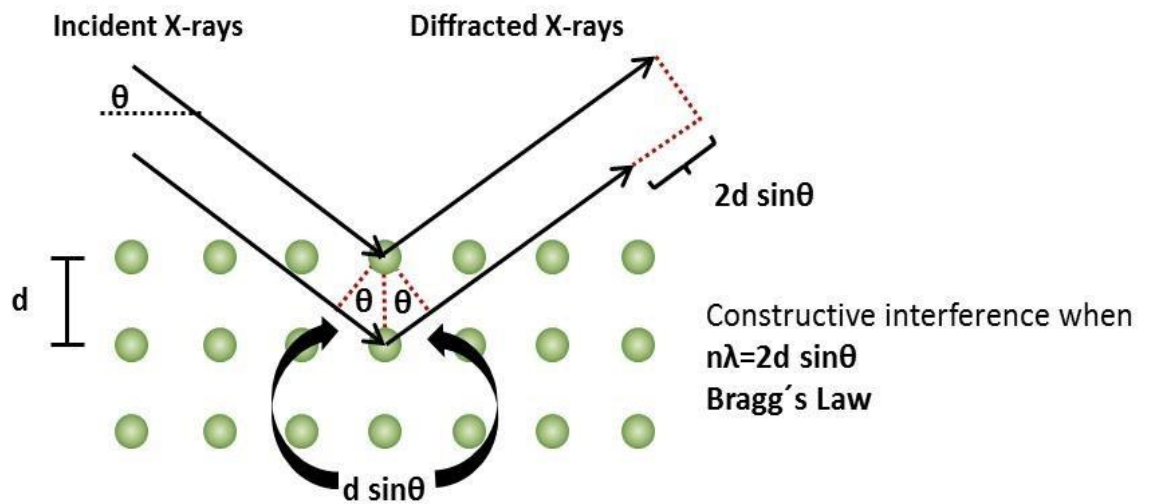


Figure 4.3.1: schematic representation of Bragg's law

4.4.2 SCANNING ELECTRON MICROSCOPE (SEM)

High resolution scanning electron microscope (SEM) is a very useful for the characterization of size and shape of the nanoparticle. The sample preparation and acquisition of images are relatively easy and convenient. Since the SEM image is a two-dimensional (2D) representation of three-dimensional (3D) structures from a certain viewing angle, it does provide a certain amount of 3D detail that can be used to recreate the shape of a basic structure with sub-nm precision using model-based measurement.

Secondary electrons (SEs) and/or backscatter electrons (BSE) produced when the sample is bombarded by the primary electron (PE) beam provide the signal that causes a SEM image to be created. A SEM's overall accelerating voltage is usually 30 kV. The SE has a limited escape depth in the region of a few nm owing to their low energy below 50 eV, while the BSE has a slightly higher escape depth in the range of a few nm.

The demand for SEM in laboratories to characterise nanoparticulate materials is increasing because of the application of nanoparticles in the commercial products.

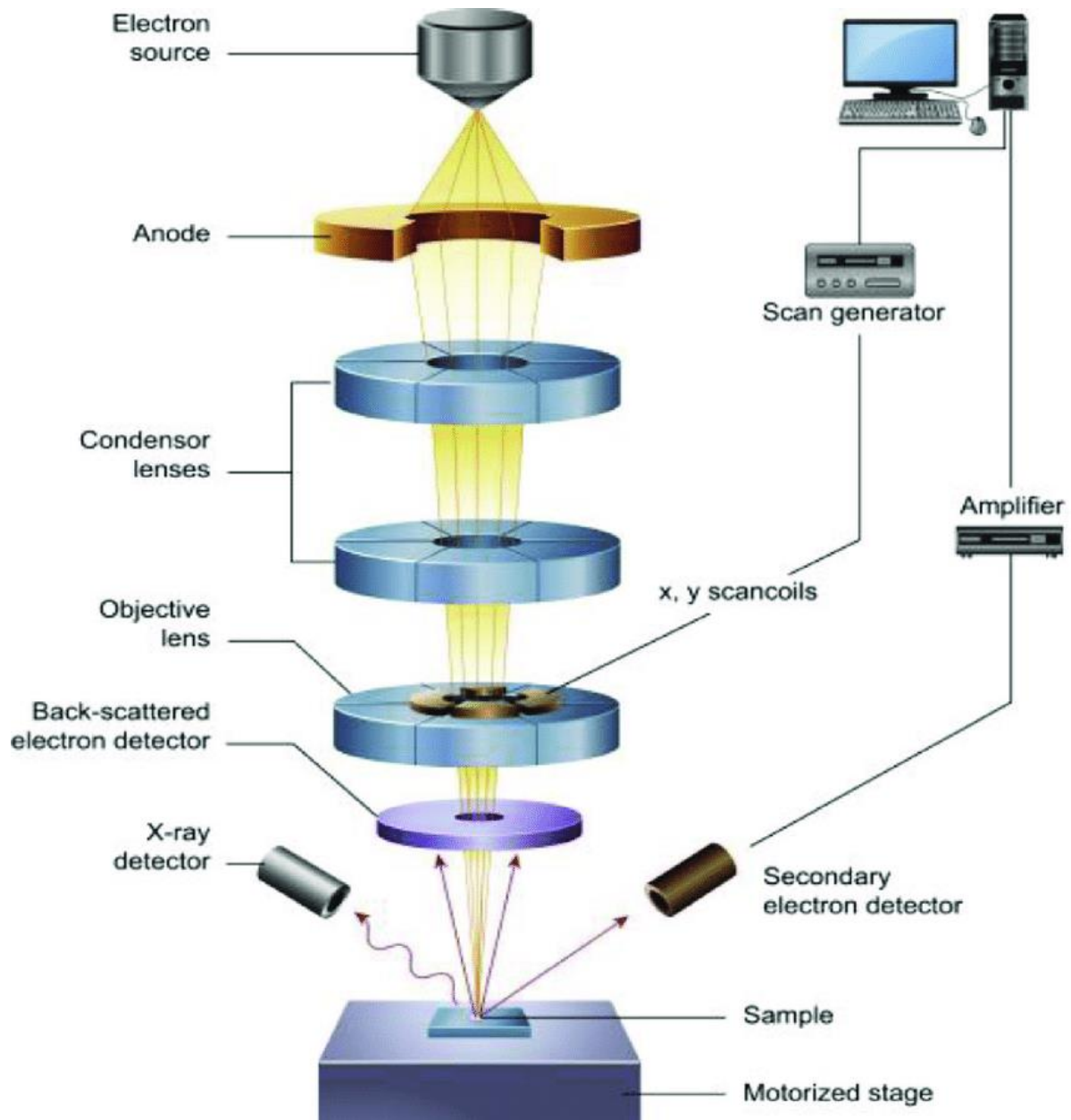


Figure 4.3.2: Schematic representation of SEM

4.3.3 FOURIER-TRANSFORM INFRARED SPECTROSCOPY

Fourier transform infrared (FTIR) spectroscopy is a very efficient non-destructive characterization technique for surface characterization of nanoparticles' surfaces, providing a specific setup is connected to the spectrometer.

it is used to determine the functional groups. The chemical structure of the nanoparticles' surface and the surface reactive sites responsible for surface reactivity can be determined under particular conditions.

The structure and bands of the synthesised nanoparticles, FTIR may be used to test a variety of components, including bulk or thin films, liquids, solids, pastes, powders, fibres, and other materials. FTIR analysis may be used for quantitative (amount) analysis as well as qualitative (identification) analysis of materials where appropriate criteria are used. FTIR can test samples up to 11 mm in diameter and weigh in bulk or the top 1 mm of the sample.

The difficulty in FTIR characterization is mainly because of high degree of overlapping of the IR absorption bands which makes difficult for the attribution of certain functional groups, despite the fact that up to date computer-searchable databases are available.

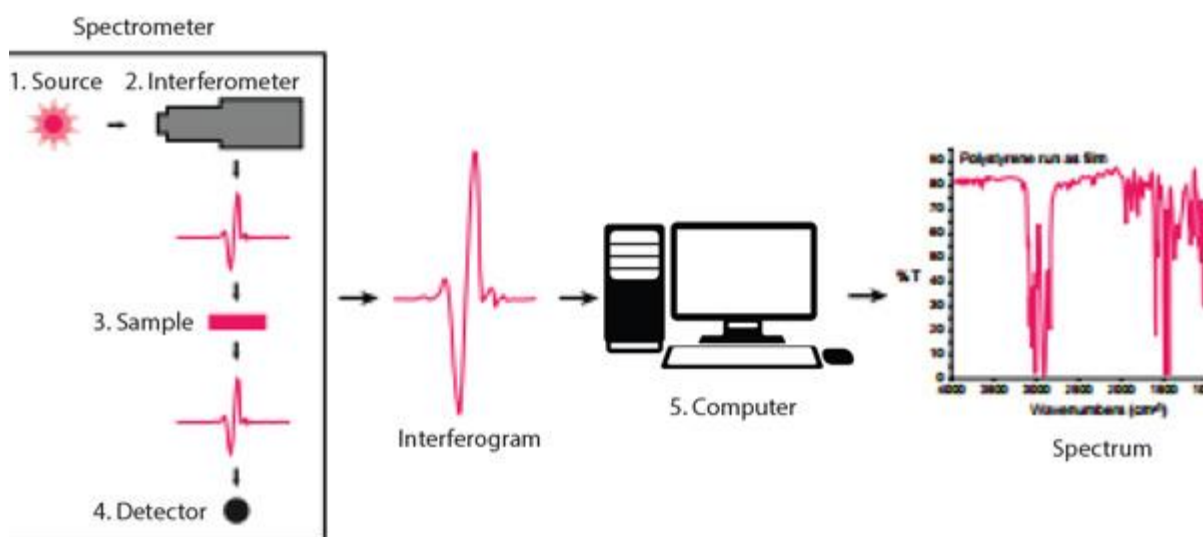


Figure 4.4.3: Schematic representation of FTIR

4.3.4 UV-VISIBLE SPECTROSCOPY

UV-Vis spectroscopy is an analytical technique that measures the amount of discrete wavelengths of UV or visible light that are absorbed by or transmitted through a sample in comparison to a reference or blank sample.

Absorption spectroscopy is complementary to fluorescence spectroscopy, in that fluorescence deals with transitions of electrons from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state.

Molecules containing bonding and non-bonding electrons (n-electrons) can absorb energy in the form of ultraviolet or visible light to excite these electrons to higher anti-bonding molecular orbitals.

The more easily excited the electrons (i.e. lower energy gap between the HOMO and the LUMO), the longer the wavelength of light it can absorb. There are four possible types of transitions ($\pi\text{--}\pi^*$, $n\text{--}\pi^*$, $\sigma\text{--}\sigma^*$, and $n\text{--}\sigma^*$), and they can be ordered as follows: $\sigma\text{--}\sigma^* > n\text{--}\sigma^* > \pi\text{--}\pi^* > n\text{--}\pi^*$

UV/Vis spectroscopy is routinely used in analytical chemistry for the quantitative determination of different analytes or sample, such as transition metal ions, highly conjugated organic compounds, and biological macromolecules. Spectroscopic analysis is commonly carried out in solutions but solids and gases may also be studied.

- All diluted inorganic solutions are uv active

- Solutions of transition metal ions can be colored (i.e., absorb visible light) because d electrons within the metal atoms can be excited from one electronic state to another.
- The colour of metal ion solutions is strongly affected by the presence of other species, such as certain anions or ligands. For instance, the colour of a dilute solution of copper sulfate is a very light blue; adding ammonia intensifies the colour and changes the wavelength of maximum absorption (λ_{max}).
- While charge transfer complexes also give rise to colours, the colours are often too intense to be used for quantitative measurement.

The Beer–Lambert law states that the absorbance of a solution is directly proportional to the concentration of the absorbing species in the solution and the path length. Thus, for a fixed path length, UV/Vis spectroscopy can be used to determine the concentration of the absorber in a solution. It is necessary to know how quickly the absorbance changes with concentration. This can be taken from references (tables of molar extinction coefficients), or more accurately, determined from a calibration curve.

A UV/Vis spectrophotometer may be used as a detector for HPLC. The presence of an analyte gives a response assumed to be proportional to the concentration. For accurate results, the instrument's response to the analyte in the unknown should be compared with the response to a standard; this is very similar to the use of calibration curves. The response (e.g., peak height) for a particular concentration is known as the response factor.

The wavelengths of absorption peaks can be correlated with the types of bonds in a given molecule and are valuable in determining the functional groups within a molecule. The Woodward–Fieser rules, for instance, are a set of empirical observations used to predict λ_{max} , the wavelength of the most intense UV/Vis

absorption, for conjugated organic compounds such as dienes and ketones. The spectrum alone is not, however, a specific test for any given sample.

The method is most often used in a quantitative way to determine concentrations of an absorbing species in solution, using the Beer–Lambert law:

where A = absorbance

I = intensity of the incident is the transmitted intensity,

L = the path length

c = concentration of absorbing species

ϵ = molar absorptivity coefficient.

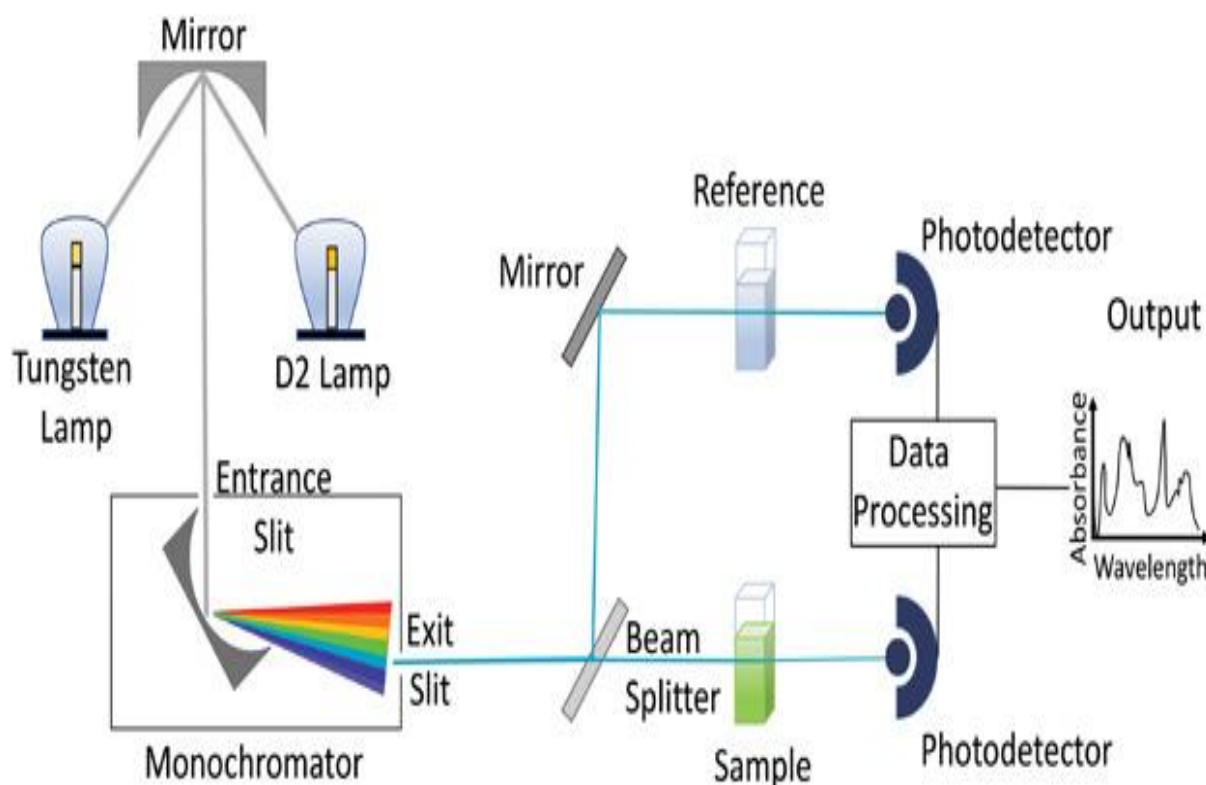


Figure 4.3.4: schematic representation of UV-visible spectroscopy

CHAPTER - 5

RESULTS AND DISCUSSION

Crystal structure, morphology, structural composition and functional group analysis of the given nanocomposite have been confirmed by characteristic techniques like XRD, SEM, UV-Vis spectroscopy, FTIR.

5.1 X-RAY DIFFRACTION (XRD)

Figure 5.1 shows X-ray diffraction study of Tin (II) sulfide nanoparticles synthesized by co-precipitation method. From the XRD pattern it is clear that Tin (II) sulfide nanoparticles synthesized were purely crystalline in nature. Average particle size of Tin sulfide nanoparticles was found to be 23.0 to 30.0 nm

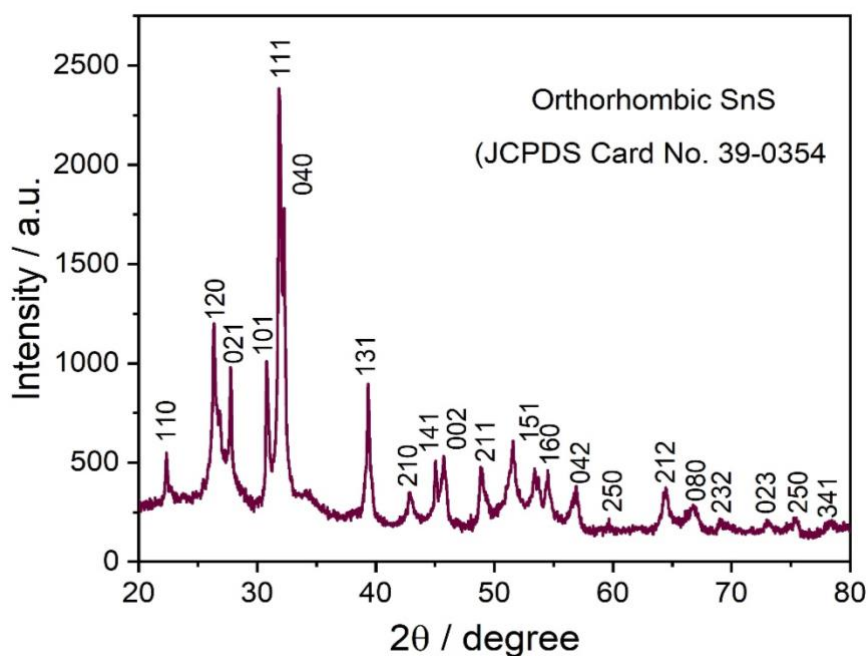


Figure 5.1: X-ray diffraction pattern of Tin (II) sulfide

5.2 FTIR SPECTROSCOPY

Figure 5.2 shows FT-IR spectra of Tin (II) sulfide nanoparticles synthesized by co-precipitation method. FTIR spectroscopy was carried out in order to ascertain the purity and nature of Tin (II) sulfide nanoparticles as synthesized by co-precipitation method

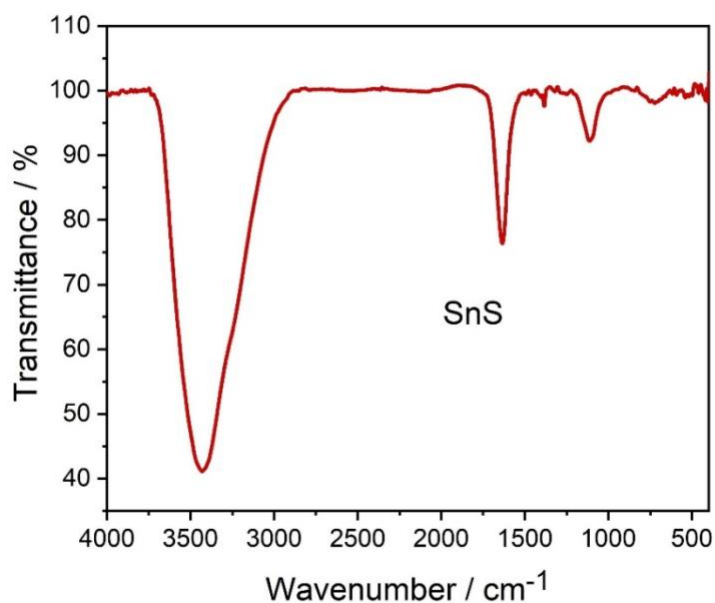


Figure 5.2: FT-IR spectra of Tin (II) sulfide nanoparticles

S.NO	FUNCTIONAL GROUP	WAVENUMBER (cm ⁻¹)
1	O-H	3450.74
2	C=O	1634.38
3	C-O	1113.69

From the above result we conclude that the synthesized nanomaterial is Tin sulfide. Absorption peak Observed at 3450.74 cm⁻¹ may be due to O-H stretching vibrations. The absorption Peaks at 1634.38 cm⁻¹, 1113.69 cm⁻¹ may be due to C=O Stretching, C-O stretching respectively.

5.3 UV- VISIBLE SPECTROSCOPY

Figure 5.3 shows UV-Visible spectra of Tin (II) sulfide nanoparticles synthesized by co-precipitation method as a function of wavelength. The UV-visible absorption shows sharp absorption at 442.3 nm due to Tin (II) sulfide nanoparticles.

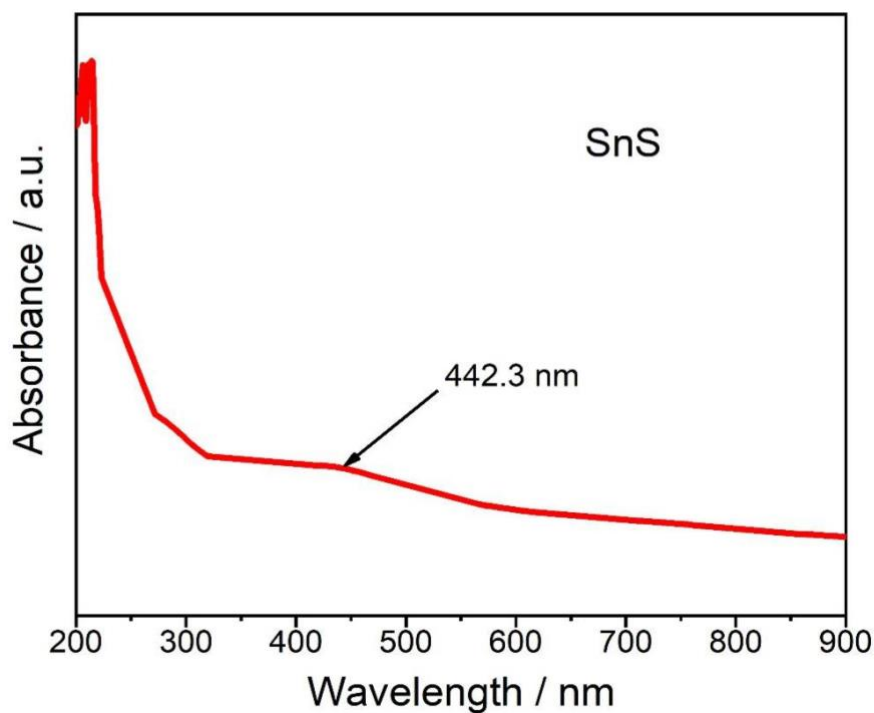


Figure 5.3 UV-Visible spectra of Tin (II) sulfide nanoparticles

5.4 SCANNING ELECTRON MICROSCOPY (SEM)

The surface morphological studies of nanoparticles have been performed by Scanning Electron Microscope (SEM). The SEM images of SnS nanoparticles are portrayed in Fig 5.4. It was well documented that the surface morphology has significant impact on the performance of nanostructure materials. The uniform distributions of grains are observed in the micrographs. The particles are nearly spherical in shape has uniform size and found to have an average size of about 50 nm. Surface morphology and nanostructure of the solids were obtained with the aid of a field emission scanning electron microscope.

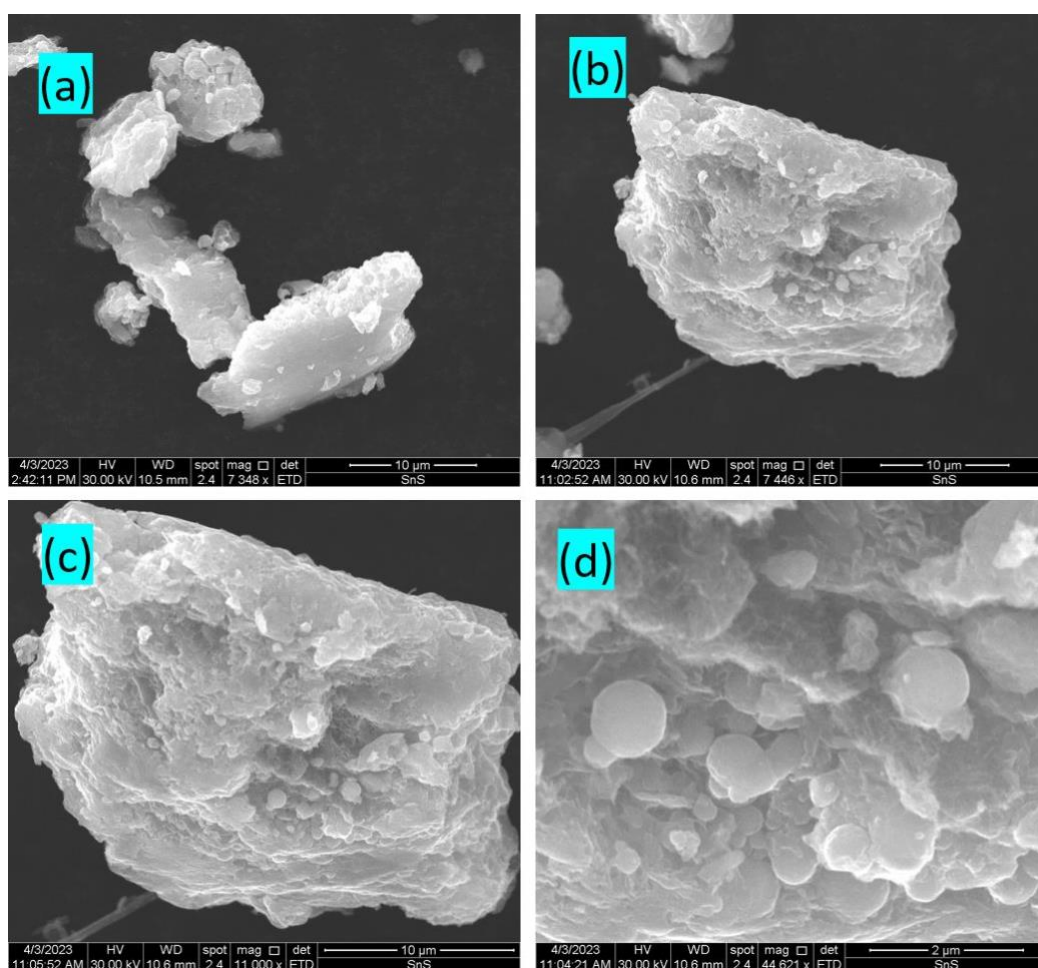


Figure 5.4: SEM images of SnS nanoparticles

CHAPTER- 6

SUMMARY AND CONCLUSION

- SnS nanoparticles have been successfully synthesized by Co-Precipitation method.
- The nanoparticles are characterized using advanced characterization techniques such as XRD, SEM, UV and FTIR techniques.
- The synthesized SnS nanoparticles have found in orthorhombic structure with high crystal quality.
- The FT-IR spectral analysis reveals the characteristics peaks along with some addition surface functional groups due to surfactant was used during the sample preparation.
- The UV-visible absorption shows sharp absorption peak at 442.3 nm in the blue region due to metal nanoparticles.
- Highly homogeneous with spherical shape of SnS nanoparticles was confirmed by the scanning electron microscopy.
- The homogeneous growth of the nanoparticles production is more suitable for scale-up the materials production in industrial application using the facile chemical method.
- There are large numbers of potential applications can be used the chemically synthesized SnS metal nanoparticles due to its high crystal quality, visible light absorption and homogeneous growth.

REFERENCES

- Band-structure, optical properties, and defect physics of the photovoltaic semiconductor SnS, J. Vidal et al., Appl. Phys. Lett. 100, 032104 (2012); DIO: 10.1063/1.3675880.
- Valley physics in tin (II) sulfide, A. S. Rodin et al., Phys. Rev. B, 93, 045431 (2016); DOI: 10.1103/PhysRevB.93.045431.
- Growth of Large-Size SnS Thin Crystals Driven by Oriented Attachment and Applications to Gas Sensors and Photodetectors, J. Wang et al., ACS Appl. Mater. Interfaces, 8, 9545–9551 (2016); DOI: 10.1021/acsami.6b01485
- Two-Dimensional SnS: A Phosphorene Analogue with Strong In-Plane Electronic Anisotropy, Z. Tian et al., ACS Nano, 11, 2219–2226 (2017); DOI: 10.1021/acsnano.6b08704.
- Few-Layer Tin Sulfide: A New Black-Phosphorus-Analogue 2D Material with a Sizeable Band Gap, Odd–Even Quantum Confinement Effect, and High Carrier Mobility, C. Xin et al., J. Phys. Chem. C, 120, 22663–22669 (2016); DOI: 10.1021/acs.jpcc.6b06673.
- Nanostructured SnS with inherent anisotropic optical properties for high photoactivity, M. Patel et al., Nanoscale, 8, 2293 (2016); DOI: 10.1039/c5nr06731f.
- Burda, Clemens, et al. “Chemistry and properties of nanocrystals of different Shapes.” Chemical reviews 105.4 (2005): 1025-1102

- Catalano, Paolo N., et al. “A survey on analytical methods for the characterization of green synthesized nanomaterials.” *Current Pharmaceutical Biotechnology* 22.6 (2021): 823-847
- Makhlouf, Abdel Salam Hamdy, and Ahmed Barhoum, eds. *Fundamentals of nanoparticles: classifications, synthesis methods, properties and characterization*. William Andrew, 2018.
- N. Sui, Y. Duan, X. Jiao, and D. Chen, “Large-scale preparation and catalytic properties of one-dimensional α/β -Sns nanostructures,” *Journal of Physical chemistry C*, vol. 113, no. 20, pp. 8560–8565, 2009
- Nagarajan, Ramanathan, and T. Alan Hatton, eds. *Nanoparticles: synthesis, stabilization, passivation, and functionalization*. American Chemical Society, 2008
- Bayda, Samer, Muhammad Adeel, Tiziano Tuccinardi, Marco Cordani, and Flavio Rizzolio. “The history of nanoscience and nanotechnology: From chemical– physical applications to nanomedicine.” *Molecules* 25, no. 1 (2020): 112.
- Bayda, Samer, Muhammad Adeel, Tiziano Tuccinardi, Marco Cordani, and Flavio Rizzolio. “The history of nanoscience and nanotechnology: From chemical– physical applications to nanomedicine.” *Molecules* 25, no. 1 (2020): 112.