Synthesis and Electrochemical Study of Zinc Sulfide Nanoparticles

A DISSERTATION

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IN

PHYSICS

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2K22/MSCPHY/20

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I, Kanika Singh, 2K22/MSCPHY/20 student of M.Sc. Physics, hereby certify that the work which is presented in the Dissertation-II entitled in fulfilment of the requirement for the award of the **Master in Science in Physics** and submitted to the **Department of Applied Physics**, **Delhi Technological University**, Delhi in an authentic record of my own, carried out during a period from July 2023 to June 2024 under the supervision of **Prof. Vinod Singh.**

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Place: **Delhi** Date: 7th JUNE 2024 Prof. Vinod Singh SUPERVISOR

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Certified that Kanika Singh (2K22/MSCPHY/20) have carried out her search work presented in this thesis entitled "Synthesis and Electrochemical Study of Zinc Sulfide Nanoparticles" for the award of the Degree of Masters of Science in Physics from Department of Applied Physics, Delhi Technological University, Delhi under my supervision. The thesis embodies the result of the original work, and studies are carried out by the student. The contents of the thesis do not form the basis for the award of any other degree to the candidate or anybody else from this or any other University/ Institution.

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ABSTRACT

Zinc sulfide (ZnS) nanoparticles were synthesized using the coprecipitation method with thioacetamide and zinc acetate dihydrate as precursors. The nanoparticles were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), photoluminescence (PL), and UV-visible spectroscopy. XRD analysis confirmed the zinc blende structure with well-defined (111), (220), and (311) planes. SEM revealed an agglomerated, polygonal-shaped morphology. FTIR spectroscopy identified the functional groups present. The UV-visible absorption peak at 302 nm indicated a quantum size effect-induced blue shift. Deconvoluted PL spectra showed emission peaks at approximately 439 nm and 492 nm. Electrochemical measurements demonstrated the nanoparticles' electrocatalytic properties, with the lowest overpotential observed at a 50 mV/sec scan rate for the oxygen evolution reaction in an alkaline medium.

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Kanika Singh

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LIST OF ABBREVATIONS

1.	ZnS	Zinc Sulfide
2.	Zn (CH ₃ CO ₂) ₂ .2H ₂ O	Zinc Acetate
3.	(C_2H_5NS)	Thioacetamide
4.	КОН	Potassium Hydroxide
5.	ZnS @NF	ZnS painted Nickel foam

Chapter 1

INTRODUCTION

Hydrogen has attracted significant interest as a possible substitute for fossil fuels in addressing global energy demand and environmental degradation. Hydrogen has the ability to effectively store energy for utilization in electricity generation. Currently, most production relies heavily on fossil fuels and a high-energy consumption process. Since the discovery of TiO₂'s photocatalytic activity for hydrogen generation, photocatalysis has emerged as a highly desirable process for generating hydrogen using clean, ecologically friendly, and cost-effective procedures [1]. Over the last several decades, researchers have discovered various photocatalysts demonstrating exceptional watersplitting efficiency. The photocatalysts exhibit maximum activity only in response to UV light, constituting more than 4% of the overall solar radiation. To be useful in real-world scenarios, photocatalysts for hydrogen production must be capable of functioning using visible light. ZnS is intensively explored as a photocatalyst because of its capacity to form electron-hole pairs when exposed to light rapidly. It also has a very high level of activity in generating H₂ underg UV light. Zinc sulfide (ZnS) has a hexagonal crystal structure and a significantly negative potential for excited electrons. Zinc sulfide (ZnS) nanoparticles can be synthesized using a variety of techniques, each with advantages and disadvantages, including chemical precipitation, hydrothermal, sol-gel, microemulsion, solvothermal, and template-assisted synthesis, which are some of the most commonly used methods. Desirable nanoparticle characteristics, scalability, and cost-effectiveness determine the preferred process. Furthermore, because of its high band gap (~3.6 eV), ZnS has a hexagonal significantly negative potential for excited electrons and is sensitive to visible light. Efforts have been made to enhance the ability of ZnS to absorb light in the visible spectrum by introducing transition metal ions (such as Au, Ni, and Cu) into its structure. It is widely utilized in light-emitting diodes, piezoelectric devices, and photodetectors. Semiconductor nanostructures have gained global interest due to their unique optoelectronic capabilities, which make them appropriate for applications such as UV detectors, gas sensors, photovoltaic devices, and photocatalysis [2]. Metal sulfide semiconductors, such as ZnS, has attracted significant scientific attention for its numerous properties and potential applications. Due to their easily adjustable wide energy band gap, ZnS nanoparticles are used significantly for photocatalytic activities. However, ZnS is not directly involved in hydrogen creation. However, ZnS-based materials can contribute to hydrogen-generating processes indirectly, such as through photocatalysis for water splitting, co-catalytic systems, hydrogen sulfide elimination, and hydrogen storage materials. While ZnS is not an essential material for hydrogen production, its photocatalytic capabilities and compatibility with hydrogen-related processes make it worthwhile for some hydrogen production and purification aspects.

In this work, we synthesis of ZnS nanoparticles by using co-precipitation and hand grinding methods. To analyse the structural and morphological characteristics, X-ray diffraction (XRD), Fourier transform spectroscopy (FTIR), and scanning electron microscopy (SEM) techniques were used. To examine the optical and electrochemical properties, uv-visible spectroscopy (UV), photoluminescence (PL), cyclic voltammetry (CV), and electrochemical impedance spectroscopy (EIS) were performed.

Hydrogen has various applications, including use in industrial processes, as rocket fuel, and in fuel cells for electricity generation and powering vehicles. Many operators of natural gas power plants are exploring the potential of hydrogen as an alternative or supplement to natural gas. In hydrogen fuel cells, electricity is produced through the reaction of hydrogen and oxygen atoms. In an electrochemical cell, hydrogen reacts with oxygen to produce electricity, water, and a bit of heat, similar to how a battery operates. Hydrogen fuel cells are being employed to power electrical equipment on spaceships and to provide electricity on Earth. Small fuel cells have been developed to power electrical devices like laptop computers and mobile phones. Several automobile manufacturers have created fuel cells to power vehicles. Fuel cells have the ability to generate electricity for emergency power in buildings as well in remote regions that are not connected to electric grids.

All fuel has a unique flammable range, which determines the minimum and maximum air concentrations required for burning. Combustion requires an igniting source and a flammable fuel. Hydrogen has a high flammability range (4% -75% in air) compared to other fuels, as illustrated below. Hydrogen poses a safety risk due to its high flammability and low ignition energy. At ideal combustion conditions (a 29% hydrogen-to-air volume ratio), hydrogen combustion requires significantly less energy than other fuels, requiring only a small spark to ignite. At low quantities of hydrogen in the air, the energy required for burning is similar that of other fuels. To avoid fires or explosions, prevent hydrogen

from accidentally interacting with an oxidizer. Proper system design, installation, and maintenance can prevent hydrogen leaks in equipment. If a leak occurs, ventilation can help to dilute the hydrogen and keep the concentration below the lower flammable limit.

1.1 Photoelectrochemical cell

Photoelectrochemical cells differ from standard photovoltaics in several ways. PECs typically consist of a p-type photocathode and an n-type photoanode. The photoelectric effect generates electric potential, which is then transferred directly to chemical energy, removing the need for batteries [3]. Typically, water splitting is the principal process employed to store chemical energy. Its two half-reactions are water oxidation and water reduction. The US Department of Energy views PEC research as a long-term research goal rather than an immediate breakthrough for the hydrogen economy. Recently, PEC research has moved its focus to CO2 reduction in conjunction with water oxidation. Not unexpectedly, such a system would resemble natural photosynthesis, the primary source of energy acquisition for most life on Earth. Nature's photosynthesis has a low energy efficiency (<6%), therefore, simply mimicking is insufficient. To compete with other renewable energy sources, our artificial photosynthesis must outperform natural photosynthesis by a large margin. In any case, water oxidation is a critical reaction for the future of PEC research and development [4].

1.2 Zinc Sulfide as a photocatalyst

Zinc sulfide (ZnS) is used in water splitting activities due to its distinct photocatalytic properties. ZnS has a broad band gap (~3.6 eV), making it good for UV absorption. ZnS's huge band gap allows it to generate enough energy to fuel the water splitting reaction when exposed to UV light. ZnS is chemically stable in aquatic environments, which is crucial for maintaining the photocatalyst's efficacy and longevity during water splitting. ZnS has good photocatalytic activity for hydrogen and oxygen evolution activities. This is due to its high charge separation efficiency and strong redox characteristics, which are essential to fueling the water splitting reaction. Zinc and sulphur are plentiful and cheap, making ZnS an affordable material for large-scale applications [5]. ZnS can be produced in a variety of nanostructures (nanoparticles, nanorods, etc.), which increases its surface area and reactivity. Furthermore, doping ZnS with other elements (such as transition metals or nonmetals) may improve both visible light absorption and overall photocatalytic efficacy. ZnS can be combined with other materials to produce composites with superior

photocatalytic efficacy due to synergistic effects. When ZnS is combined with other semiconductors, the absorption range extends to the visible light spectrum, which improves charge separation efficiency [6].

CHAPTER 2

METHODOLOGY

2.1 Material used

Zinc acetate (Zn (CH₃CO₂)₂.2H₂O) and thioacetamide (C₂H₅NS) were used as source precursors with their purity of 98%. No further purification is required of the precursors due to its high level of purity.

2.2 Synthesis processes

2.2.1 Hand Grinding

Hand grinding synthesis using a mortar and pestle is a traditional and straightforward method used in chemistry and materials science for the preparation of fine powders and the mixing of solid reagents. This method is particularly useful for the synthesis of solid-state compounds, catalysts, and nanomaterials. The hand grinding synthesis method relies on mechanical forces to achieve particle size reduction and intimate mixing of solid reactants. The key principles include

- Mechanical Force: The application of force through grinding crushes the particles into finer sizes.
- Homogenization: Continuous grinding and mixing ensure that the reactants are evenly distributed, leading to uniform reactions.
- Surface area enhancement: Reducing particle size increases the surface area, enhancing the reaction rates due to better contact between reactants.

The method is easy to perform and requires minimal equipment; hence, it is inexpensive, with the mortar and pestle being reusable as well as suitable for small-scale synthesis, particularly in research and educational settings. It is ideal for reactions that need to avoid solvents, reducing the risk of contamination and environmental impact.

2.2.2 Co-precipitation

Coprecipitation refers to the simultaneous precipitation of multiple compounds from a solution. It is the most convenient and cost-effective method for producing nanoparticles. In this process, metal hydroxide precipitates from a salt precursor in the presence of a base in a solvent. Fig 2.1 depicts a flowchart for the coprecipitation process.

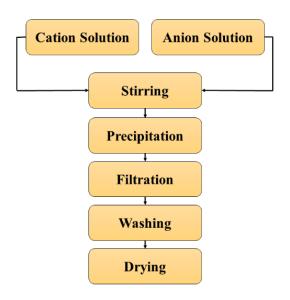


Fig 2.1 Typical coprecipitation method for the synthesis of micro- and nanoparticles.

The cation and anion solutions are combined and stirred constantly to precipitate hydroxide, which is subsequently dried to create oxide. Coprecipitation involves several simultaneous processes, including initial nucleation, growth, coarsening, agglomeration, and ripening. The regulated release of anions and cations modulates the nucleation and particle development kinetics, which enables the creation of monodispersed nanoparticles.

2.3 Sample preparations

2.3.1 ZnS structural information

ZnS is a distinctive composite that has two types of crystalline structures [7]. There are mainly two polymorphs, including zincblende (also known as sphalerite) and wurtzite, as shown in (Fig 2.2) respectively [8].

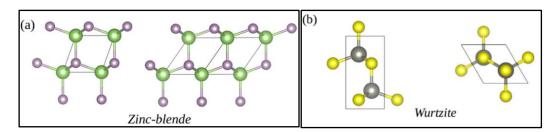


Fig 2.2 The two polymorphs of ZnS (a) zincblende (b) wurtzite

2.3.2 Hand grinded ZnS

The hand grind method procedure was used to synthesiz ZnS particles from zinc acetate Zn $(CH_3CO_2)_2.2H_2O$ and thioacetamide (C_2H_5NS) as source material, as shown in Fig 2.3. Both compounds were poured in the mortar and the mixture was continuously grinded clockwise and anticlockwise with the help of pestle to reduce the size of the materials. The grinding was performed continuously for 4 hours. After this process the mixture was kept in high furnace to get the desired output in the form of ZnS nanoparticles [9].



Fig 2.3 Hand grind synthesis of ZnS nanoparticles

2.3.3 Co-precipitated ZnS

The schematic diagram of the synthesis process is shown in Fig 2.4. The co-precipitation procedure was used to synthesize nano-sized ZnS particles from zinc acetate Zn $(CH_3CO_2)_2.2H_2O$ and thioacetamide (C_2H_5NS) as source materials. All compounds were used without additional purification, with 50 mL of deionized water was a solvent. The ZnS nanoparticles were produced in the following order: 0.05 m Zn $(CH_3CO_2)_2.2H_2O$

was dissolved in 50 ml of deionized water and swirled constantly for 2 hours, while 0.4 M C_2H_5NS was added to 50 ml of deionized water and mixed continuously for 2 hours in separate beakers [10]. The C_2H_5NS solution was then put into Zn (CH₃CO₂)₂.2H₂O and stirred continuously for 4 hours. After the reaction is finished, a white precipitate is formed. The mixture was centrifuged at 4000 rpm for 30 minutes, rinsed twice with water to remove the thioacetamide, washed three times with isopropyl alcohol, and dried overnight at 50 °C [11].

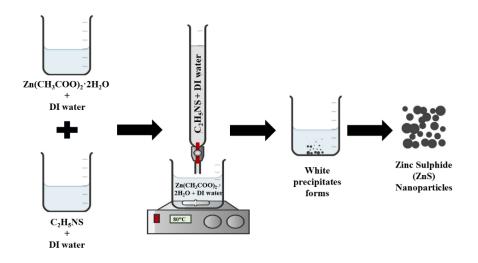


Fig 2.4 Schematic diagram for the synthesis of ZnS nanoparticles

CHAPTER 3

CHARACTERIZATION TECHNIQUES

The crystallographic study was performed on a Bruker D8 advanced X-ray diffractometer using Cu-K α source with a wavelength of λ = 1.54 Å. The XRD spectrum was recorded for the 2 θ range 20° - 80°. The photomicrographs were taken with the help of scanning electron microscopy (JEOL JSM 6610LV, powered by tungsten electron source). To investigate the attached functional groups, Fourier transform infrared spectroscopy (FTIR) was performed on the ZnS fine powder using the PerkinElmer spectrum-II instrument. The absorption spectra of ZnS nanoparticles were taken Uv-visible spectroscopy (V 750). The emission spectra of photoluminescence (PL) were measured using a JASCO FP-8300 PC-Spectro fluorophotometer equipped with a xenon arc lamp, with excitation at a wavelength of 488 nm.

3.1 X-ray Diffraction (XRD)

X-ray diffraction (XRD) is a technique used to study the atomic structure of crystalline materials by observing the patterns produced when X-rays are scattered off a crystal lattice. The discovery of X-ray diffraction is attributed to the work of several key scientists in the early 20th century. The phenomenon was first observed in 1912 by Max von Laue, a German physicist. Von Laue theorized that if a crystal were irradiated with X-rays, the crystal lattice would act as a diffraction grating for the X-rays. He proposed that the periodic arrangement of atoms in a crystal would cause the incident X-rays to diffract in specific directions, creating a pattern that could be analyzed to determine the structure of the crystal. Von Laue conducted an experiment where they directed X-rays at a copper sulfate crystal and captured the resulting diffraction pattern on a photographic plate. This experiment confirmed von Laue's hypothesis and demonstrated that crystals have a regular, repeating atomic structure. For this groundbreaking work, X-ray diffraction has since become a crucial tool in many scientific fields, including chemistry, physics, and materials science, allowing researchers to uncover the detailed atomic structure of a vast array of crystalline substances.

3.1.1 Bragg's Law

Based on the phenomena of diffraction through a crystal, Bragg gave a simple formula using interplanar spacing 'd' of crystal planes and angle ' θ ' of incident of X-rays. This can be written as

$$2d \sin\theta = n\lambda \tag{3.1}$$

where, n is an integer, n=1, 2, 3.....

Bragg's law schematic diagram is shown in Fig 3.1, which is true for all $\lambda \leq 2d$ and λ is the wavelength of X-rays.

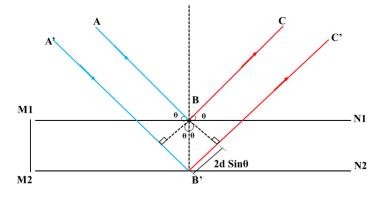


Fig 3.1 Bragg's Law

The generated XRD pattern has three pieces of information about the sample; a space group determined by the location of the peaks, information about the basis given by the intensity of the peaks; and the determination of crystallite size and strain with the help of the width of the peaks.

3.1.2 Methods of estimating the microstructural parameters from XRD profile analysis

To investigate the microstructural parameters (crystallite size and microstrain) of the synthesised material, they were calculated using the Scherrer formula and the Williamsson halls (W-H) method.

3.1.3 Scherrer's Formula

The Scherrer formula is

$$D_{hkl} = \frac{K\lambda}{\beta \cos\theta}$$
(3.2)

Where, D_{hkl} is the crystalline size of the nanoparticles, **K** is the shape, λ is the X-ray wavelength, β is the FWHM (full-width at half maximum), and θ is the Braggs angle.

3.1.4 Williamson-Hall Method

The Williamson-Hall (W-H) (Fig 3.2) method is a simplified and indirect method that can clearly differentiate between the size induced and the microstrain induced peak broadening by considering the FWHM as a function of peak position.

$$(\beta \cos\theta)^{n} = (0.9 \lambda / D_{WH})^{n} + (4 \varepsilon_{WH} \sin\theta)^{n}$$
(3.3)

where, β = FWHM,

 λ = wavelength of X-rays,

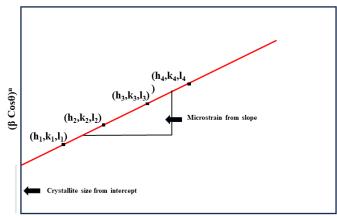
D_{WH} = crystallite size from the W-H method,

 ε_{WH} = microstrain from the W-H method,

 $\mathbf{n} = 1$ for the Lorentzian (Cauchy) peak profile,

 $\mathbf{n} = 2$ for the Gaussian peak profile

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(4 Sinθ)ⁿ

Fig 3.2 W-H Plot

3.2 Scanning Electron Microscope

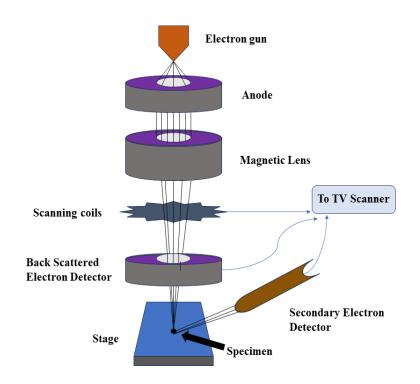


Fig 3.3 Block Diagram of SEM

Scanning electron microscopy (Fig 3.3) was used to examine ZnS nanoparticles. SEM creates high resolution images of the sample by scanning the surface using electron beams and making high-esolution enlarged images. The electron beam is fired by an electron gun and then passes through the column of the microscope where it is focused as it moves down through the lenses and apertures. No atoms can interact with this electron beam in the microscope column due to the vacuum. SEM ensures images of high quality, which provide detailed information on the topography, morphology, and composition of the sample.

3.3 UV-Visible Absorption Spectra

UV-visible absorption spectroscopy (Fig 3.4) is a method used to measure the absorption of ultraviolet (UV) and visible light by a substance. This method provides valuable information about the electronic structure of molecules, which can be used to identify substances and study their properties. The basic principle of Uv-visible absorption is that when a molecule absorbs UV light or visible light, then electrons are promoted from a lower energy state (ground state) to a higher energy state (excited state). The energy difference between these states corresponds to the energy of the absorbed light. The energy associated with the wavelength can be described by the formula $E = \frac{hc}{\lambda}$, where *E* represents energy, *h* is Planck's constant, *c* is the speed of light, and λ denotes the wavelength. The absorption spectrum is a plot of absorbance (or sometimes transmittance) against wavelength. Peaks in the spectrum correspond to wavelengths where light is absorbed by the sample. The position and intensity of these peaks provide information about the electronic structure and environment of the absorbing species. Peak Position: The wavelength of maximum absorbance (λ_{max}) indicates the energy difference between the ground and excited states. Peak Intensity: The intensity of the absorbance peak is related to the concentration of the absorbing species and the molar absorptivity (ε), according to Beer's Law, i.e, $A = \epsilon c l$, where *A* is the absorbance, *c*c is the concentration, and *l* is the path length of the sample cell.

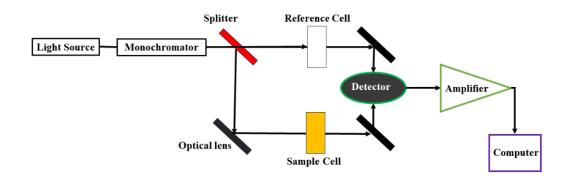


Fig 3.4 Block Diagram of UV-Visible Spectroscopy

3.4 Fourier transform infrared (FTIR)

The term "fourier transform infrared" (FTIR) represents the commonly used technique in infrared spectroscopy as shown in the block diagram in Fig 3.5. Infrared spectroscopy methods rely on the principle that certain infrared radiation is absorbed as it passes through a material. By analyzing the radiation that enters the sample, valuable information about the sample can be obtained. These spectroscopic measurements can be utilized to identify and distinguish between different molecules, as each molecule emits a unique spectrum based on its specific structure. Covalent bonds present within molecules possess the ability to selectively absorb light at particular wavelengths, resulting in changes in the vibrational energy of the bond. The type of vibration, such as stretching or bending, induced by the interaction with infrared radiation depends on the

nature of the atoms involved in the bond. Due to these variations, different molecules exhibit distinct transmittance patterns, as specific bonds and functional groups absorb particular frequencies. The representation of the spectrum involves plotting the wavenumber (cm-1), which indicates the energy of molecular bond vibrations, on the X-axis, while the transmittance is plotted on the Y-axis. Analyzing the spectrum entails identifying the groups and bonds associated with each peak.

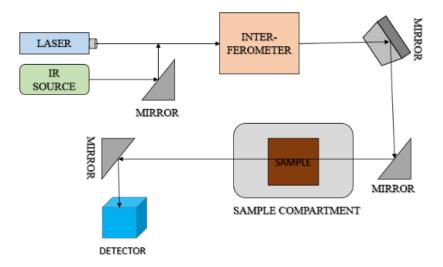


Fig 3.5 Block Diagram of FTIR

3.5 Photoluminescence (PL) Spectroscopy

The block diagram of photoluminescence spectroscopy is depicted in Fig 3.6. Photoluminescence (PL) is the emission of light from a material after it absorbs photons. The PL spectrum provides valuable information about the electronic and structural properties of the material. Here's an explanation of the theory behind photoluminescence: The basic principles of photoluminescence are photon absorption. When a material is exposed to light, photons with energy higher than the material's band gap can be absorbed. The excited electrons from the valence band to the conduction band, therefore, creating electron-hole pairs. Relaxation Processes, once excited, these electron-hole pairs can undergo various relaxation processes. Some of the energy may be lost as heat through non-radiative processes, where the excitons interact with phonons (vibrations in the crystal lattice). Radiative Recombination: eventually, the electron may recombine with the hole, releasing the excess energy in the form of a photon. This process is radiative recombination, and the energy of the emitted photon corresponds to the energy difference between the electron's excited state and its ground state. PL spectrum is a plot of the intensity of emitted light as a function of wavelength (or energy). The features of the spectrum can reveal important characteristics about the material as:

Peak Position: The position of the main peak in the PL spectrum corresponds to the band gap energy of the material. For semiconductors, this peak typically appears near the band gap energy.

Peak Width: The width of the peak provides information about the quality of the material. A narrow peak suggests high crystalline quality and low defect density, while a broad peak may indicate the presence of impurities, defects, or inhomogeneities.

Intensity: The intensity of the PL signal is related to the efficiency of the radiative recombination process. Higher intensity suggests a higher probability of radiative recombination, which can be influenced by factors such as temperature, material purity, and crystal quality.

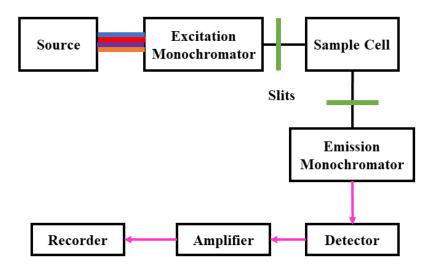


Fig 3.6 Block diagram of Photoluminescence

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 X-Ray Diffraction

XRD was performed to determine the structural properties, as shown in Fig 4.1. The prominent peaks for the cubic crystal lattice have been observed at 2θ values of 29.1° , 48.7° , and 57.8° , corresponding to the (111), (220), and (311) planes, respectively. The crystallite size was determined using the Debye-Scherrer formula and the Williamson-Hall technique. The Debye Scherrer formula is provided as mentioned in Section 3.2:

$$D = \frac{\kappa \lambda}{\beta \cos \theta}$$
(3.2)

In this equation, λ represents the wavelength of light, q is the Bragg's angle, whereas, K is the form factor, and b is the full breadth at half maxima. The crystallite size, determined using the Debye Scherrer formula, was 2.70 nm [12]. The W-H method was used to separate strain-induced broadening using equation (3.3):

$$(\beta \cos\theta)^{n} = (0.9 \,\lambda / D_{WH})^{n} + (4 \,\varepsilon_{WH} \,x \sin\theta)^{n}$$
(3.3)

where β = FWHM, λ = X-ray wavelength, **D**_{WH} = crystallite size from theW-H method, ε_{WH} = micro strain from W-H method, **n** = 1 for the Lorentzian (Cauchy) peak profile, and **n** = 2 for Gaussian peak profile [7]. The crystallite and slope were measured at 1.53 nm and -0.02475, respectively. The slope's negative value indicates anisotropy in the sample [13].

Method's	Crystallite size (nm)
Scherrer's formula	2.70
W-H plot	1.53
Slope	-0.02475

Table 4.1 Table shows the crystallite size of ZnS nanoparticles using different method

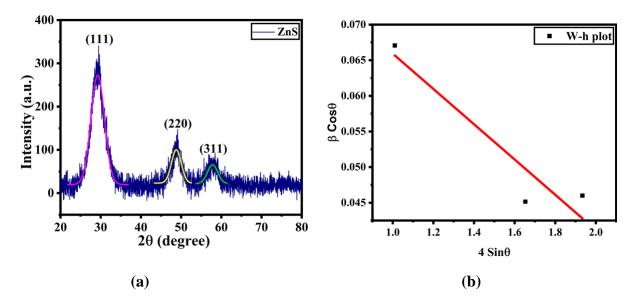


Fig 4.1 (a) Show the X-ray diffraction profile of the ZnS sample showing the corresponding miller indices of diffraction planes with the diffraction angle (**b**) Shows the W-H plot for ZnS nanoparticles.

4.2 Scanning electron microscope

The photomicrographs of the ZnS nanoparticles produced by SEM show the morphology. The polygonal-shaped agglomerated ZnS nanoparticles were clearly observed in Fig 4.2. The particle size of ZnS nanoparticles was calculated at 87 nm, as shown in Fig 4.3.

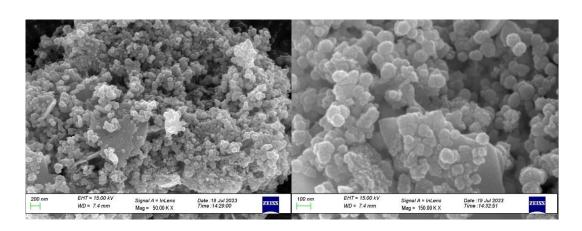


Fig 4.2 SEM images of ZnS nanoparticles

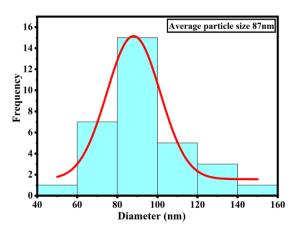


Fig 4.3 Distribution curve of ZnS nanoparticles

4.3 Fourier transformed infrared spectroscopy

Fig 4.4 displays the FTIR spectra of a pure ZnS sample that was recorded in the range of 500–4000 cm⁻¹ in order to distinguish the functional groups that were present in the prepared sample. The broad and strong peak at 3328 cm⁻¹ in the higher energy region may be attributed to the O-H stretching of absorbed water on the ZnS floor [14]. The Amide II band (N-H deformation) is attributed to the max peak at 1557 cm⁻¹. C-N bond stretching is ascribed to the peak at 1015 cm⁻¹ (Amide III band). The CO stretching is responsible for the peak at 465 cm⁻¹. The Zn-S bond vibrations have been linked to the peak at 465 and 1015 cm⁻¹.

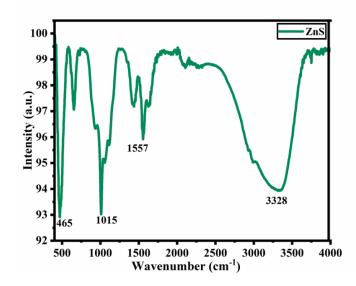


Fig 4.4 FTIR spectrum of ZnS nanoparticles at room temperature

4.4 UV-Visible Spectroscopy

UV-vis spectroscopy is used to investigate the effect of certain deficiencies on the electrical and the optical properties of ZnS nanoparticles. The absorbance curve in Fig 4.5 shows that the generated sample consumes sunlight, hence showing that the ZnS NPs have visible-light-induced photocatalytic activities due to vacancies in their particular band structures [14]. Zn-deficient samples have significant absorption intensity. The band gap (Eg) values for direct band gap materials in the structure can be calculated using Tauc relations (Fig 4.5) using Tauc's equation:

$$(\alpha hv)^{1/n} = A (hv - E_g)$$
 (4.4)

In this equation, ' α ' represents the absorption coefficient, '**h**' is the Planck's constant, '**v**' represents the photon frequency, '**A**' is the proportionality constant, and E_g is the band gap energy. [11 For direct allowed transition, n = ½. To determine viable transitions, construct the graph of $(\alpha hv)^2$ against hv and estimate the band gap by extending the straight region of the curve on the hv axis. The $(\alpha hv)^2$ Vs hv graph in Fig 4.5 shows that the ZnS sample exhibits a straight band gap of 3.64 eV, indicating a blue shift. According to previous studies, ZnS has a bulk band gap of 3.72 eV [15].

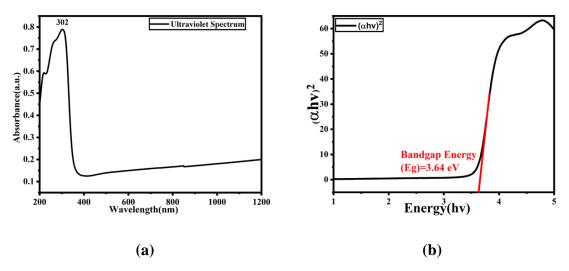


Fig 4.5 (a) UV-Vis absorption spectra of ZnS nanoparticles (b) Tauc plot of ZnS nanoparticles

4.5 Photoluminescence (PL) Spectrum

Fig 4.6 represents the photoluminescence (PL) spectra of ZnS nanoparticles in various S/Zn proportions provided at an excitation wavelength of 488 nm. Zinc vacancies (V_{Zn}), sulfur vacancies (Vs), , interstitial sulfur atoms (Is), and interstitial zinc atoms (Izn) are all common types of point defects encountered in pure ZnS. As stated by earlier reports, the point defects that take effect as luminous sites in the course of photoluminescence processes. The photoluminescence spectra show that the emissions peaks are at 439, 468, and 492 nm. The transition from interstitial Zn produced emission wavelengths of 439 and 468 nm, respectively. Transitions at 439 nm result from IZn in the samples. The strength of the broad blue fluorescence (492 nm) of the PL spectra of ZnS nanoparticles from the produced ZnS NPs varied with percentage [16]. The intense emission at 492 nm was induced by the reconnection of holes in the surface state (SS) with electron which get trapped at sulfur vacancy (Vs) sites. The example exhibits a solid blue discharge with a peak at 492 nm. ZnS emits from surface or lattice defects with modest concentrations of sulfide nanoparticles. However, the generated ZnS contains many sulfur vacancies (Vs), which act as doubly ionized donor sites. The comparable variation in intensity of all the emission bands in undoped ZnS nanoparticles as stoichiometry changes suggests a link with Vs centers. These defect-induced emission peaks, as well as changes in PL intensity, are caused by crystal defects.

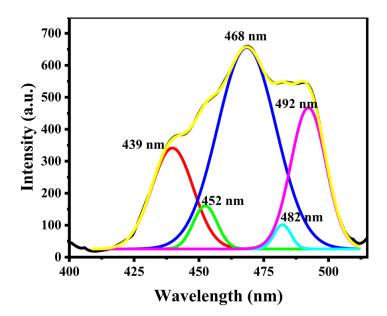


Fig 4.6 Photoluminescence spectra of ZnS nanoparticles

4.6 Electrochemical study

The cyclic-voltammetry (CV) analysis of the fabricated ZnS nanoparticles is achieved in a 0.5 M KOH solution with pH 14 at a scanning rate of 10 mV/sec in the potential range of -0.2 - 0.8 V. The cyclic voltammetry urves may depict the oxidation and reduction reactions on the electrodes' surfaces. Fig 4.7 (a) indicates that there were low current increases on the ZnS nanoparticles. ZnS painted nickel foam (ZnS@NF) electrodes at a low voltage. However, a strong rise in current in the electrode made up of ZnS@NF nanoparticles was observed at a slightly higher potential, 0.9 V, due to the electrocatalytic oxidation. This demonstrates the oxidization and reduction of ZnS nanoparticles as electrocatalysts [17]. The histogram of overpotential with different scan rates is shown in Fig 4.7 (b). The lowest overpotential was observed at 50 mV/sec due to the higher charge transfer rate. The specific capacitance of the ZnS nanoparticles was calculated by the plot between scan rate and oxidation current densities, which was 9.8535x10⁻⁴ A/V/sec, as shown in Fig 4.7 (c). Electrochemical impedance spectroscopy (EIS) was performed in 0.5M KOH without DC biasing, as shown in Fig 4.7 (d) [18]. The Nyquist plot was fitted using the rindless circuit, which informs the electrolyte and wire resistance value, which was R1 3.777 ohms; charge transfer resistance (Rct), which was a combination of R2 (66.88 ohms) and Q1; and the electrode–electrode resistance (R3), which was obtained 36 KOhms, which is higher due to the NF as substrate.

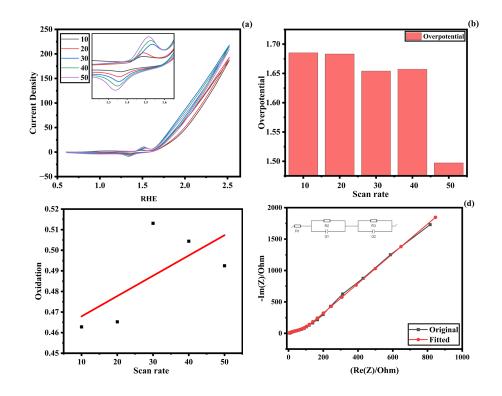


Fig 4.7 (a) CV of ZnS nanoparticles **(b)** histogram: scan rate v/s overpotential **(c)** Double layer capacitance of ZnS nanoparticles **(d)** Nyquist plots of ZnS nanoparticles

CHAPTER-5

CONCLUSION

This study confirmed the successful synthesis of ZnS nanoparticles. The crystallite size was calculated using the Scherrer formula and the W-H method, and Cs ~ was 2.7 nm and $C_{W-H} 1.53$ nm, respectively. The particle size was calculated from SEM images, which was 87 nm, and the direct band gap of ZnS nanoparticles was also determined at 3.64 eV. Electrochemical properties were studied by using CV and EIS. Here, ZnS nanoparticles were found to have a low overpotential of 1.4973 at a high scan rate (50 mV/sec). ZnS nanoparticles can be used for photoelectrochemical water splitting as an electrode material and many other applications.

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Synthesis and electrochemical studies of ZnS nanoparticles

Kanika Singh, Jasveer Singh, Vinod Singh#

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Abstract:

Zinc sulfide (ZnS) nanoparticles were synthesized using the coprecipitation method with thioacetamide and zinc acetate dihydrate as precursors. The nanoparticles were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), photoluminescence (PL), and UV-visible spectroscopy. XRD analysis confirmed the zinc blende structure with well-defined (111), (022), and (311) planes. SEM revealed an agglomerated polygonal-shaped morphology. FTIR spectroscopy identified the functional groups present. The UV-visible absorption peak at 302 nm indicated a quantum size effect-induced blue shift. Deconvoluted PL spectra showed emission peaks at approximately 439 nm and 492 nm. Electrochemical measurements demonstrated the nanoparticles' electrocatalytic properties, with the lowest overpotential observed at a 50 mV/sec scan rate for the oxygen evolution reaction in an alkaline medium.

Introduction:

Hydrogen has attracted significant interest as a possible substitute for fossil fuels in addressing the global energy demand and environmental degradation. Most production relies heavily on fossil fuels and a high-energy consumption process. Since the discovery of TiO₂'s photocatalytic activity for hydrogen generation, photocatalysis has emerged as a highly desirable process for generating hydrogen using clean, ecologically friendly, and cost-effective procedures [1]. Over the last several decades, researchers have discovered various photocatalysts demonstrating exceptional water-splitting efficiency. The photocatalysts exhibit maximum activity only in response to UV light, constituting more than 4% of the overall solar radiation. To be useful in real-world scenarios, photocatalysts for hydrogen production must be capable of functioning using visible light. ZnS is intensively explored as a photocatalysts

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