

# Chapter One

## Introduction

### 1.1 Preface

The synthesis and properties of nanostructured chalcogenides, including semi conducting lead sulfide (PbS), cadmium sulfide (CdS), mercury sulfide (HgS), copper (II) sulfide (CuS), Cu<sub>2</sub>S, and sodium sulfide (Na<sub>2</sub>S) and silver sulfide (Ag<sub>2</sub>S), have been described in a number of review articles and books [1,5]. Silver sulfide nanoparticles (NPs) are currently being explored as infrared active nanomaterial that can provide environmentally stable alternatives to heavy metals such as lead. Moreover, silver sulfide can also be found as thin films. Recently, quantum dot NPs, such as PbS and PbSe, have particularly emerged as some of the most promising new materials for the development of advanced photoelectric devices, because of low-cost manufacturing, solution process ability, and their superior photoelectric properties including unique absorption and emission of light in the NIR regions [6]. The proposed synthesis and doping methods constitute a facile and efficient approach for fabricating graphene-NP-based hybrid two-dimensional (2D) photoelectric devices for various applications, including flexible devices and advanced phototransistors [5,6].

Thin films of silver sulfide have been prepared by different methods which include chemical bath deposition, [8, 10, and 13]. Spray pyrolysis, [14], thermal evaporation, sol-gel, successive ionic layer adsorption and reaction molecular beam epitaxy and electrochemical ion-exchange processes. Silver films have applications in microelectronics, as do parts in superconducting materials and as surface enhanced Raman active substrates. Physical vapour deposition methods such as vacuum evaporation, sputtering, electron beam evaporation pulsed laser deposition and molecular beam epitaxy have been used for the growth of silver

films [7]. But has superior step coverage and can be easily scaled up to deposit high purity films on larger areas [7].

Nowadays, the study of semiconductor nanoparticles has much consideration because of their novel electronic and optical properties. Nanoparticles (NPs) such as PbS, CdSe and CdS have thin band gap semiconductor quantum dots which have been used as photo catalysts in recently. Among all, silver sulfide nanoparticles are major materials as photo catalysts [8].

To minimize the amount of the solar absorber material, thin films of high absorptivity need to be considered. Silver sulfide (Ag<sub>2</sub>S) is an important metal chalcogenide semiconductor compound with the band gap of ~1.5 eV [6] and a high absorption coefficient of approximately 10<sup>4</sup> cm<sup>-1</sup> [7], able to ensure most of the solar light absorption in a thin layer. Ag<sub>2</sub>S belongs to I–VI compound semiconductor materials with monoclinic crystal structure. It possesses a unique combination of properties, such as high dark ionic or electronic conductivity, photoconductivity, as well as related photovoltaic and photochromic effects [6, 8 and 10]. Silver sulfide thin films have been produced as functional materials with applications in the contemporary advanced technologies ranging from photoconductive and photovoltaic cells, solar selective coatings, ion selective electrodes and membranes to IR detectors and laser recording media [9].

Thin film solar light absorbers are of particular interest for fabrication of large area arrays, solar selective coatings and solar cells. As such, tune able and controllable thickness thin film deposition is of crucial importance in attaining their desired absorbing. Previously, Ag<sub>2</sub>S thin films with different morphologies have been prepared via various methods, such as solid–vapor reactions, radio frequency sputtering, thermal evaporation, electro deposition, successive ionic layer absorption and reaction (SILAR) and chemical bath depositions (CBD) [6].

As in most of metal sulfide materials, a crucial property for solar energy harvesting is the resulting conductivity [1, 3 and 9].

The optical properties and antibacterial activity of silver Nanoparticles (Ag NPs) and their semiconductors from S and Se (Ag<sub>2</sub>S and Ag<sub>2</sub>Se nanoparticles). The nanoparticles were synthesized and stabilized in the matrix of the synthetic zeolite. Both compounds Ag<sub>2</sub>S and Ag<sub>2</sub>Se nanoparticles show a size of around 9 nm and quasi-spherical morphology. The interplanar distances calculated in the TEM images confirm the presence of metallic and semiconductor nanoparticles with hexagonal, monoclinic and orthorhombic structures for nanoparticles Ag, Ag<sub>2</sub>S and Ag<sub>2</sub>Se, respectively. The Surface Plasmon resonance for Ag<sub>2</sub>sNPs was obtained around 415 nm. Furthermore, in nanoparticles Ag<sub>2</sub>S and Ag<sub>2</sub>Se we identified absorptions bands in the visible region with maxima around 330 and 375 nm. The presence of SERS effect caused by metallic nanoparticles on the surface of the zeolite was observed. The antibacterial activity of the nanoparticles stabilized in the zeolite was analyzed against gram-negative bacteria and gram-positive bacteria [10].

## **1.2 Research Problem**

The organic/inorganic composite materials consisting of polymers doped with metal or semiconductor nanoparticles such as CuS, ZnS, PbS, have narrow band gap semiconductor and highly toxic and responsible for extensive environmental contamination and health problems in various parts of the world.

## **1.3 Aim of the Research**

The Ag<sub>2</sub>S NPs have been extensively studied as attractive NIR-absorbing NPs for photovoltaic, photoconductors, and IR detectors due to their high biocompatibility and unique absorption ability in the UV-NIR regions, which

can be used as multispectral photo detectors in UV, visible, and NIR spectrum because of their light absorption in a broad wavelength range.

## **1.4 Importance of Research**

The unique properties of Ag<sub>2</sub>S NPs led to a variety of application such as in solar cells, photo-detectors, light-emitting diodes and switches, photoconductors, IR detectors, magnetic field sensors, optical filters, super-ionic conductors, solar-selective coatings and room temperature oxygen sensors.

## **1.5 Thesis Lay out**

The thesis consists of the four chapters, chapter one is the introduction. Chapter two is Literature review. Chapter three is concerned method and materials. Chapter four is devoted results, analysis, discussion beside conclusion and recommendations.

# Chapter Two

## Literature Review

### 2.1 Introduction

In this section we review the studies carried out by scientists in this area in order to identify some of the results you get and review the results and a summary of their results

### 2.2 Synthesis of Silver Sulfide Nanowires in Ethylene Glycol through a Sacrificial Templating Route Shancheng Yan, Yuping and et al [18].

#### Abstract

In this study, Ag<sub>2</sub>S nanowires were prepared in ethylene glycol using single-crystal silver nanowires as a sacrificial templating and choosing suitable sulfur sources for the sulfuration reaction. X-ray powder diffraction and a transmission electron microscope equipped with an energy dispersive X-ray analysis were used to characterize the products. The results indicated Ag<sub>2</sub>S nanowires with diameters of about one hundred nanometers and lengths up to several micrometers could be obtained through this method. The selected area electron diffraction pattern and high-resolution transmission electron microscope imaging indicated that the Ag<sub>2</sub>S nanowires thus formed were crystalline.

#### Conclusions

To sum up, Ag<sub>2</sub>S nanowires were prepared in ethylene glycol through a sacrificial templating route. The Ag<sub>2</sub>S nanowires had diameters of a few hundred nanometers and lengths up to several micrometers. HRTEM image further indicated that the formed Ag<sub>2</sub>S nanowires were crystalline. In principle, it can be extended to the synthesis of other semiconductor sulfide nanostructures.

## **2.3 Structural, optical, photoluminescence and antibacterial properties of copper-doped silver sulfide nanoparticles, Ali Fakhri, and et al [8].**

### **Abstract**

The Ag<sub>2</sub>S and Cu doped Ag<sub>2</sub>S nanoparticles were prepared by simple chemical co-precipitation method and characterized by XRD, SEM, EDX, TEM, PL and UV–vis spectra. The photo catalytic activity of Ag<sub>2</sub>S and Cu doped Ag<sub>2</sub>S nanoparticles were investigated with Ofloxacin antibiotic, which is part of the fluoroquinolone family. The morphological study indicated that the products were spherical shape in with diameter size of 30 nm. The photo catalytic results demonstrated that the Cu doping increased the photo catalytic efficiency of Ag<sub>2</sub>S nanoparticles. The outcome of antibacterial experiment under visible light irradiation indicate that the Cu doped Ag<sub>2</sub>S nanoparticles represent increased antibacterial performance compared with un-doped Ag<sub>2</sub>S nanoparticles.

### **Conclusions**

The un-doped and doped Ag<sub>2</sub>S nanoparticles were successfully synthesized by a simple chemical co-precipitation method. The analysis of SEM and XRD showed that the particles of all samples were at the Nano-scale. UV–Vis spectra indicated that, as the doping Ag<sub>2</sub>S increased, the band gap shifted to higher wavelengths. The PL outcome demonstrated that the tuning range of emission wavelength is nearly between 456 and 477 nm in the present one type doped Ag<sub>2</sub>S nanoparticles. The doping Cu had an influence on the degradation of Ofloxacin antibiotic the Ag<sub>2</sub>S nanoparticles. The comparison of antibacterial activity of different synthesized Ag<sub>2</sub>S samples, clearly indicated that the Cu–Ag<sub>2</sub>S nanoparticles are the active antibacterial for inhibition properties over the growth of *S. aureus* and *E. facials*.

## **2.4 Optical properties of silver, silver sulfide and silver selenide nanoparticles and antibacterial applications Y., Delgado-Beleñoa, and et al [10].**

### **Abstract**

This work presents the optical properties and antibacterial activity of silver nanoparticles (Ag NPs) and their semiconductors from S and Se (Ag<sub>2</sub>S and Ag<sub>2</sub>Se nanoparticles). The nanoparticles were synthesized and stabilized in the matrix of the synthetic zeolite A4. Both compounds Ag<sub>2</sub>S and Ag<sub>2</sub>Se nanoparticles show a size of around 9 nm and quasi-spherical morphology. The interplanar distances calculated in the TEM images confirm the presence of metallic and semiconductor nanoparticles with hexagonal, monoclinic and orthorhombic structures for nanoparticles Ag, Ag<sub>2</sub>S and Ag<sub>2</sub>Se, respectively. The Surface Plasmon resonance for Ag NPs was obtained around 415 nm. Furthermore, in nanoparticles Ag<sub>2</sub>S and Ag<sub>2</sub>Se we identified absorptions bands in the visible region with maxima around 330 and 375 nm. The presence of SERS effect caused by metallic nanoparticles on the surface of the zeolite was observed. The antibacterial activity of the nanoparticles stabilized in the zeolite was analyzed against gram-negative bacteria and gram-positive bacteria.

### **Conclusions**

The zeolite A4 resulted to be a versatile matrix for the stabilization of Ag nanoparticles and semiconductors of Ag<sub>2</sub>S and Ag<sub>2</sub>Se. The absorption bands detected at 415, 330 and 375 nm indicated the stabilization of nanoparticles Ag, Ag<sub>2</sub>S and Ag<sub>2</sub>Se, respectively, in the zeolite A4. The synthesis method favors the obtaining of nanoparticles of a predominantly inferior size to 10 nm for the semiconductor nanoparticles and 4 nm for the silver nanoparticles according of the TEM analysis. The interplanar distances measured confirmed the presence of Ag, Ag<sub>2</sub>S and Ag<sub>2</sub>Se. For the concentrations of the precursor employed, the SERS effect was magnified by the samples containing only Ag. The results

suggest that the synthesized materials show a better inhibitory response against gram-positive bacteria. These results confirm that the nanoparticles Ag, Ag<sub>2</sub>S and Ag<sub>2</sub>Se may be potential candidates for a future implementation in biomedicine.

## **2.5 Ag<sub>2</sub>S deposited on oxidized polypropylene as composite material for solar light absorption, Valentina Krylova, and et al [9].**

### **Abstract:**

Thin film metal chalcogenides are superior solar light absorbers and can be combined into a functional material when deposited on polymeric substrates. Ag<sub>2</sub>S composite materials were synthesized on oxidized polypropylene using chemical bath deposition method and their properties were explored using XRD, XPS, AFM and UV–Vis. Polypropylene surfaces were modified using solution methods to introduce hydrophilicity via carboxylic group formation which resulted in Ag<sub>2</sub>S film deposition and adhesion. These films showed slightly sulfur enriched composition from XPS analysis with the sulfate-like species forming, presumably at the oxidized polymer surface sites. Ag<sub>2</sub>S particle growth mechanism included nucleation and rather large (few  $\mu\text{m}$ ) aggregate formation eventually covering the complete polymer surface, as inferred from AFM analysis. Absorption edge of the composite material shifted toward the higher wavelength in UV–Vis spectrum with the number of Ag<sub>2</sub>S exposure times showing a decreasing band gap and the possibility of obtaining tunable optical property Ag<sub>2</sub>S–polymer composites using CBD methods.

### **Conclusions**

Ag<sub>2</sub>S on oxidized polypropylene surface was synthesized using chemical bath deposition and the corresponding properties were explored using XRD, XPS, AFM and UV–Vis. We were able to successfully modify surface structure by inducing hydrophilic character via carboxylic group formation which resulted in



Ag<sub>2</sub>S film deposition and adhesion. Ag<sub>2</sub>S films were slightly sulfur rich with a specific sulfate-like species forming on the oxidized polypropylene sites. Absorption peak in UV–Vis shifted with the increasing exposures implying systematically decreasing absorption edge and possibility for a tunable optical property iPP-Ag<sub>2</sub>S composites. Atomic force microscopy revealed that increasing number of exposures to precursor solution provided a smoother and more homogeneously distributed Ag<sub>2</sub>S film on surface. Metal sulfide materials, including acanthite, possess high adsorption coefficients (10<sup>4</sup>cm<sup>-1</sup>) and will require small film thickness to absorb solar light. When incorporated into polymers using solution methods described here, tunable optical and structural property composites can be obtained.

## **2.6 Synthesis and characterization of silver sulfide nanoparticles for photocatalytic and antimicrobial applications, Pooja Kumari, and et al [19].**

### **Abstract**

The study describes a simple and novel method for the synthesis of silver sulfide nanoparticles (Ag<sub>2</sub>SNPs). The synthesized NPs were characterized by SEM (scanning electron microscope), XRD (X-ray diffraction), particle size analyzer, zeta seizer and EDX (Energy dispersive X-ray). The Ag<sub>2</sub>S NPs were spherical in shape with an effective diameter size of 30 nm. The synthesized particles possess photocatalytic activity under visible light and exhibited excellent antimicrobial effect. The photocatalytic property of Ag<sub>2</sub>S NPs was also evaluated by the degradation of methylene blue dye under visible light. NPs degraded 87% of methylene blue within 1 h at pH 8. The NPs of 0.1 lg/mL showed a growth inhibition of more than 75% against S.

### **Conclusion**

The present study revealed a simple and novel method for the synthesis of highly stable Ag<sub>2</sub>S NPs. Here the synthesized nanoparticles have spherical shape and exhibited photocatalytic property under visible light. The particles were highly

stable; hence we feel Ag<sub>2</sub>S NPs may be used for the degradation of dye bearing effluents these particles can play an important role in waste water treatment and other bioremediation processes. In addition the particles possessed excellent antimicrobial property.

## **2.7 Synthesis and Characterization of Silver Sulfide Nanoparticles Containing Sol-Gel Derived HPC-Silica Film for Ion-Selective Electrode Application, S. Shukla, and et al [14].**

### **Abstract**

Silver sulfide nanoparticles dispersed in sol-gel derived hydroxypropyl cellulose (HPC)-silica films have been successfully synthesized using H<sub>2</sub>S gas diffusion method. This is the first attempt to produce silver sulfide nanoparticles using this technique. Ag<sub>2</sub>S nanoparticles are generated through reaction of H<sub>2</sub>S gas with AgNO<sub>3</sub> precursor dissolved in the HPC-silica matrix. Transmission electron microscope (TEM) and atomic force microscope (AFM) analysis reveal nanoparticles size distribution from 2.5 nm to 56 nm for H<sub>2</sub>S gas exposed Sample. The surface chemistry of Ag<sub>2</sub>S nanoparticles and sol-gel derived HPC-silica matrix is confirmed by X-ray photoelectron spectroscopy (XPS). The negative shifts in the core-level XPS Ag (3d) binding energy of Ag<sub>2</sub>S Nanoparticles are attributed to Ag: S surface atomic ratio exhibited by these nanoparticles with varying processing conditions. Following processing and characterization, suitability of the present method to produce silver sulfide Ion-selective electrode is demonstrated by depositing Ag<sub>2</sub>S nanoparticles on a graphite rod. The high response function of the electrode is due to the presence of nanoparticles.

### **Conclusions**

Sol-gel derived HPC-silica films containing Ag<sub>2</sub>S nanoparticles with average particle size of 26 nm (H<sub>2</sub>S/not heated sample) and 200–300 nm (H<sub>2</sub>S/heated sample) have been successfully synthesized. The formation of Ag<sub>2</sub>S

nanoparticles and the presence of sol-gel derived HPC-silica matrix are confirmed using TEM, AFM and XPS analysis. The core-level Ag 3d5/2 B.E. is found to be dependent on relative Ag: S surface atomic ratio exhibited by Ag<sub>2</sub>S nanoparticles. The present technique is effectively utilized in preparing ion-selective electrodes with high response function (approaching the theoretical value) by depositing Ag<sub>2</sub>S nanoparticles on the graphite rods to detect Ag<sup>+</sup> ions in the sol.

## **2.8 Prospects and challenges of silver sulfide thin films: A review**

**P. A. Nwofe [17].**

### **Abstract**

Silver sulfide (Ag<sub>2</sub>S) has been a subject of investigation over the years for possible applications in various optoelectronics, electronics and industrial applications. Materials cost, abundance of the constituents elements, and its efficiency in use are fundamental properties in selecting materials for use as absorber or window layers in solar cell devices, or other optoelectronic applications. This paper reviews recent published work on the subject of Ag<sub>2</sub>S based solar cells and, including Ag<sub>2</sub>S crystalline structure, defect formation and metal composition. This review paper focuses Nano applications. The reasons for the sudden interest in the utilization of Ag<sub>2</sub>S by the scientific community for Ag<sub>2</sub>S solar cells and other applications are discussed; coupled with the crystallographic properties and optoelectronic properties on Ag<sub>2</sub>S synthesis processes and Devi.

### **Conclusion**

The major aim of this review is to create a platform to identify the fundamental steps that will enhance increased efficiency in Ag<sub>2</sub>S based solar cells and in other optoelectronic, electronics, and in solar thermal applications; through identification of the major setbacks limiting the efficiency of Ag<sub>2</sub>S based-devices as reported in the literature. The low solar conversion efficiency

based on the current literature report is attributed to; the wide difference between the theoretical value and the experimental value of the energy band gap, and the low value of the absorber layer thickness and small grain sizes. More so, the energy band gap is reported to be indirect [54, 124, 159- 160, 162, 170] while some research groups also show direct energy band gap as summarized in Table 1. In quantum dot solar cells, the efficiency of quantum dot silver sulphide based-solar cells are mostly limited by charge recombination amongst other factors. However successful use of Ag<sub>2</sub>S nanoparticles in different applications is common in the literature, and this strongly suggest that though silver sulphide is not possibly a good absorber layer [10].

## **2.9 Optical Properties of Chemical Bath Deposited Ag<sub>2</sub>S Thin Films, Ezenwa, et al [11].**

### **Abstract**

Thin films of Ag<sub>2</sub>S were deposited at room temperature on glass substrates immersed in a bath mixture containing aqueous solutions of silver nitrate (AgNO<sub>3</sub>), thiourea, Ethylenediaminetetra-acetate disodium Salt (EDTA) and ammonia solution. AgNO<sub>3</sub>, for silver ion source, thiourea for sulfide ion source, EDTA as a complexing agent and ammonia for pH adjustment.. The films were studied for its optical properties using a Jan way 6405 UV/VIS spectrophotometer and the results showed highest absorbance in the UV region (<400nm). Poor averaged reflectance, very low transmittance in the UV region which increases in the visible region/near infrared region, with an average of about 85% at >700nm. The optical band gap energy was found to be 1.8eV. Also the films exhibited averaged refractive index range of 1.9eV to 2.5eV. +**Keywords:** optical properties, photovoltaic, Ag<sub>2</sub>S thin films, chemical bath deposition technique, complexing agent

### **Conclusion**

Silver sulfide thin films have been successfully fabricated using chemical bath deposition technique. Solutions of silver nitrate ( $\text{AgNO}_3$ ), thiourea, Ethylenediaminetetra-acetate disodium Salt (EDTA) and ammonia solution formed the reaction bath. Good quality thin films of silver sulfide were deposited. It showed a uniform distribution of particles as shown in photomicrograph. The grains are small. The films were found to have high absorbance in the ultra violet region and depreciate as the wavelength increased. They have generally high transmittance in the visible / near infra-red region. It has high refractive index. The energy gap for the fabricated  $\text{Ag}_2\text{S}$  thin film was found to be 3.00 eV.

## **2.10 Investigation of New Optical Properties of Cd and Co-doped $\text{Ag}_2\text{S}$ Colloidal Solution, [12].**

### **Abstract**

In this work we report new optical properties of the Cd and Co-doped  $\text{Ag}_2\text{S}$  colloidal solution. Cd and Co-doped  $\text{Ag}_2\text{S}$  nanoparticles were prepared using a wet chemical method. The influence of doping on the optical properties of Cd and Co-doped nanoparticles was investigated. The TEM images showed the shape of samples is spherical of average particle size of about 6-18 nm for all pure and doped  $\text{Ag}_2\text{S}$  nanoparticles. The absorption spectra of the doped samples are red shifted as compared with of the pure  $\text{Ag}_2\text{S}$  samples. The PI intensity of the Cd-doped  $\text{Ag}_2\text{S}$  nanoparticles decreased as Cd concentration was increased. However, the PI intensity of the Co-doped  $\text{Ag}_2\text{S}$  reduced as the concentration of Co is enhanced. The particle sizes as calculated from the absorption spectra were in agreement with the results obtained from TEM.

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## **Conclusions**

Cd and Co-doped Ag<sub>2</sub>S have been successfully synthesized by a wet chemical method. The TEM results show that the products were a spherical shape with a size of about 6-15 nm for all Cd and Co-doped Ag<sub>2</sub>S nanoparticles. A red shift Phenomenon was found to increase directly with the concentration of Cd and Co doped on to the Ag<sub>2</sub>S; this effect has been observed in the UV-vis spectra and PL spectra of Cd-doped Ag<sub>2</sub>S and Co-doped Ag<sub>2</sub>S samples. With an increasing concentration of Cd incorporated in the nanoparticles, the Cd emission intensity decreases while the intensity of red emission of Co increases. A novel PL phenomenon can be observed from the Ag<sub>2</sub>S nanoparticles doped with Cd<sup>2+</sup> and Co<sup>2+</sup> ions. This result shows the important roles of dopants in changing the emission color from Ag<sub>2</sub>S nanoparticles. Therefore, the size calculated using the Burs equation is closer to values obtained from TEM when the size of particles increases due to the quantum size confinement.

## **2.11 All Procedures for the Synthesis of Silver Nan sheets, Zarin, et al [13].**

### **Abstract**

Two dimensional silver(I) coordination polymer, [Ag( $\mu$ -5-T4S)]<sub>n</sub> (1), (T4S- = toluene-4-sulfonate), has been synthesized and characterized by Inductively Coupled Plasma (ICP) and elemental analyses, IR spectroscopy and powder X ray diffraction. This compound was calcined at 450, 500 and 700 °C in a furnace and static atmosphere of air. The resulting compound from 1 at 450 °C is a spongy nanostructure which could not be characterized due to its partial calcination process .At 500 and 700 °C, the compound is a mixture of silver and silver sulfate compacted Nano sheets and microstructures. By increasing the temperature from 500 to 700 °C, the tendency for the formation of agglomerated mixture of silver and silver sulfate was increased. In addition, a number of one-

step techniques including electrochemical deposition method, electro less deposition method, direct deposition method, redox reaction, mirror reaction, coating technique, self-seeding or self-assembling process and wet chemical method have been reviewed for the synthesis of two dimensional Nano silver.

## **Conclusions**

The present investigation is focused on the synthesis of two dimensional silver(I) coordination polymer,  $[\text{Ag}(\mu_5\text{-T4S})]_n$  (1), which can be used as new precursor for preparation of silver and silver sulfate nanostructures by calcination process. Calcination process of 1 was performed at 450, 500 and 700 °C in a furnace and static atmosphere of air. XRD pattern of the residue obtained from calcination of 1 at 450 °C is different from those obtained from calcination of it at 500 and 700 °C. With attention to the TG analysis, we find out that calcination of 1 is not complete at 450 °C and we could not identify the product formed by calcination of it at this temperature. At 500 °C, the final residue product is formed. Thus, the resulting compounds from calcination of 1 at 500 and 700 °C are compacted mixtures of silver and silver sulfate Nano sheets and microstructures, respectively. By increasing the temperature from 500 to 700 °C, the tendency of silver and silver sulfate Nano sheets to agglomeration was increased. Thus, the appropriate temperature for preparation silver and silver sulfate Nano sheets from 1 is 500 °C. Furthermore different methods of synthesizing silver Nano sheets were reviewed in this paper. In addition to calcination process reported by us, here other procedures such as electrochemical deposition methods, electro less deposition method, direct deposition method, redox reaction, mirror reaction, coating technique, self-seeding or self-assembling process and wet chemical method employed so far for the synthesis of silver Nano sheets were also reviewed.

**2.12 Introduction of Ag and Ag<sub>2</sub>S Nanoparticles into Nylon 6 Film and Fiber, et al [16].**

## **Abstract**

Nylon 6 doped with Ag nanoparticles (nylon 6/Ag) was prepared by immersion of nylon 6 in aqueous AgNO<sub>3</sub> solution followed by reductive treatment by heating under H<sub>2</sub> gas or immersing in an aqueous NaBH<sub>4</sub> solution. As a result of either reductions, Ag nanoparticles below 10 nm in diameter were successfully dispersed within the nylon 6 matrix. The Ag content and the dispersion state of the Ag nanoparticles can be varied by the concentration of the AgNO<sub>3</sub> solutions and the reduction methods, respectively. From the results of the absorbed Ag<sup>+</sup> content in nylon 6 *via* immersion in AgNO<sub>3</sub> solution, nylon 6 was found to have some affinity for silver ion. Nylon 6 doped with Ag<sub>2</sub>S nanoparticles (nylon 6/Ag<sub>2</sub>S) was also prepared by immersion in AgNO<sub>3</sub> aqueous solution and the following immersion of Na<sub>2</sub>S aqueous solution. Ag<sub>2</sub>S particles having several nanometers in diameter were dispersed in nylon 6. The nylon 6/Ag<sub>2</sub>S film showed strong photoluminescence in the near infrared region.

## **Conclusions**

Simple methodologies were developed for the preparation of nylon 6 composite films containing Ag and Ag<sub>2</sub>S nanoparticles. Nylon 6/Ag composite films were prepared by reduction of Ag<sup>+</sup> ions to Ag metal using H<sub>2</sub> or NaBH<sub>4</sub> as reducing agents. The Ag nanoparticles in the composite were formed with larger particles distributed near the film surface, while relatively smaller particles were located in the inner parts the film. For nylon 6/Ag<sub>2</sub>S composites, β-Ag<sub>2</sub>S nanoparticles were formed by the fulguration of Ag<sup>+</sup> ions with Na<sub>2</sub>S. The particles were dispersed densely in the vicinity of the film surface, with the number of the particles decreasing gradually away from the film surface. Nylon 6/Ag<sub>2</sub>S film exhibited a strong photoluminescence peak at around 1310 nm. Acknowledgments. This work was supported by Grant-in-Aid for 21st Century COE Program by the Ministry of Education, Culture, Sports, Science and Technology



# CHAPTER Three

## Material and Methods

### 3.1 Introduction to Nanoparticles

Nanotechnology is the science that deals with matter at the scale of 1 billionth of a meter ( $10^{-9} \text{ m} = 1 \text{ nm}$ ), and is also the study of manipulating matter at the atomic and molecular scale. A nanoparticle is the most fundamental component in the fabrication of a nanostructure, and is far smaller than the world of everyday objects that are described by Newton's laws of motion, but bigger than an atom or a simple molecule that are governed by quantum mechanics. The United States instituted the National Nanotechnology Initiative (NNI) back in 2000, which was soon followed (2001) by a plethora of projects in nanotechnology in nearly most of the U.S. Departments and Agencies [1]. About 20 Research Centers were subsequently funded by the National Science Foundation (NSF), an agency responsible solely to the President of the United States and whose mandate is to fund the best of fundamental science and technology projects. NSF was the lead U.S. agency to carry forward the NNI. The word "nanotechnology" soon caught the attention of various media (TV networks, the internet, etc.) and the imagination and fascination of the community at large. In general, the size of a nanoparticle spans the range between 1 and 100 nm. Metallic nanoparticles have different physical and chemical properties from bulk metals (e.g., lower melting points, higher specific surface areas, specific optical properties, mechanical strengths, and specific magnetizations), properties that might prove attractive in various industrial applications. However, how a nanoparticle is viewed and is defined depends very much on the specific application. In this regard, summarizes the definition of nanoparticles and nanomaterial by various organizations. Of particular importance, the optical property is one of the fundamental attractions and a

characteristic of a nanoparticle. For example, a 20-nm gold nanoparticle has a characteristic wine red color. A silver nanoparticle is yellowish gray. Platinum and palladium nanoparticles are black. Not surprisingly, the optical characteristics of nanoparticles have been used from time immemorial in sculptures

### 3.2 Material

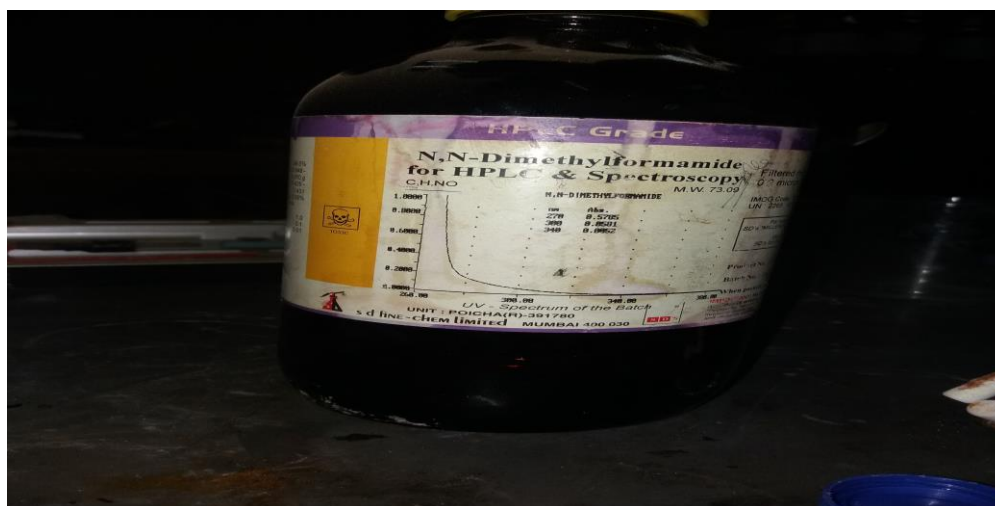
A. Chemicals Materials used in this study were silver nitrate hex hydrate (99.0%  $\text{AgNO}_3 \cdot 6\text{H}_2\text{O}$ ), sodium sulfide Nona hydrate ( $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ ), 95.0%), N,N-Dimethylformamide molecular weight 250 - 300 (from Merck, Germany), and ethanol. (3, 4-ethylene dioxythiophene), (styrenesulfonate) (PEDOT: PSS), Poly (3-hexylthiophene), (regioregular, electronic grade) and [6, 6]-phenyl-C61-butyric acid methyl ester were purchased from Ossila. Sodium hydroxide (NaOH) was used as the reducing agents.



**Figure (3.1) silver nitrate**



**Figure (3.2) sodium sulfide**



**Figure (3.3) N.N-Dimethylformamide**

### **3.3 Methods**

#### **3.3.1 Wet Chemical Route Synthesis of Silver Nanowires**

Metallic nanoparticles exhibit unusual optical, thermal, chemical and physical properties that are due to a combination of the large proportion of high-energy surface atoms compared to the bulk solid and to the nanometer-scale mean free path of an electron in a metal ( $\sim 10$ - $100$  nm for many metals at room temperature).<sup>1-4</sup> Silver is an especially attractive metal to examine on the Nano scale, due to its extremely high electrical conductivity in the bulk,<sup>5</sup> and its

unusual optical properties that depend on nanoparticle size and shape.<sup>6-10</sup> Previous strategies to control the growth of anisotropic nanoparticles have involved either hard templates<sup>11-14</sup> or softer directing agents, including surfactants or polymers.<sup>15-21</sup> Although good control over nanoparticle dimensions can be realized in these syntheses, removal of the template or directing agent from the nanoparticle surface can require harsh conditions or multiple washings to remove unwanted materials. For sensing applications such as surface enhanced Raman scattering that require analytic adsorption to the metallic nanoparticle surface, the presence of residue from the synthesis on the nanoparticle surface may be a significant interferent.<sup>22</sup> Linking metallic Nano scale objects together in a rational way requires knowledge of the exposed surface of the nanoparticle;<sup>4, 23</sup> again, surface-bound residue from the synthesis increases the difficulty of doing chemistry at the surface. In this paper, we report a synthetic method to make silver nanowires in the absence of a surfactant or soft template, in an attempt to prepare metallic nanostructures with as “clean” surfaces as possible, in water. A typical synthesis involves the combination of two silver solutions, A and B, prepared in glassware washed thoroughly with soap (Alconox), deionized water, and aqua Regina. Solution A consisted of 100 mL of deionized H<sub>2</sub>O, 1.5-2 of 1 M NaOH (Fisher), and 40 of 0.1 M AgNO<sub>3</sub> (Sigma).

This solution, in an Erlenmeyer flask, was brought to a boil with rapid stirring. Quickly added to A was 5 mL of 0.01 M trisodium citrate (Fisher), and then the solution was allowed to boil for an additional 10 min. Concomitantly, solution B was made by mixing 150 mL of deionized H<sub>2</sub>O and 1.5-2 of 1 M Na OH with 20 of 0.1 M AgNO<sub>3</sub> and bringing this to a boil. Solution B was added to A, the neck of the flask was covered with an inverted 50 mL beaker, and the mixture was allowed to boil for 60 min while stirring rapidly. When the mixture evaporated to 75 mL, additional quantities (75 mL) of boiling deionized water were added (this

usually occurred at  $\sim 30$  min). The final volume was typically 75-100 mL, and the final pH was 7.0-7.1.



**Figure (3.4.a) shows the silver nitrate on magnetic stiller**



**Figure (3.4.b) shows the silver nitrate on magnetic stiller**



**Figure (3.5) shows the sodium sulfide on magnetic stiller**



**Figure (3.6.a) shows the process of mixing sodium sulfide and silver nitrate**



**Figure (3.6.b) shows the process of mixing sodium sulfide and silver nitrate**

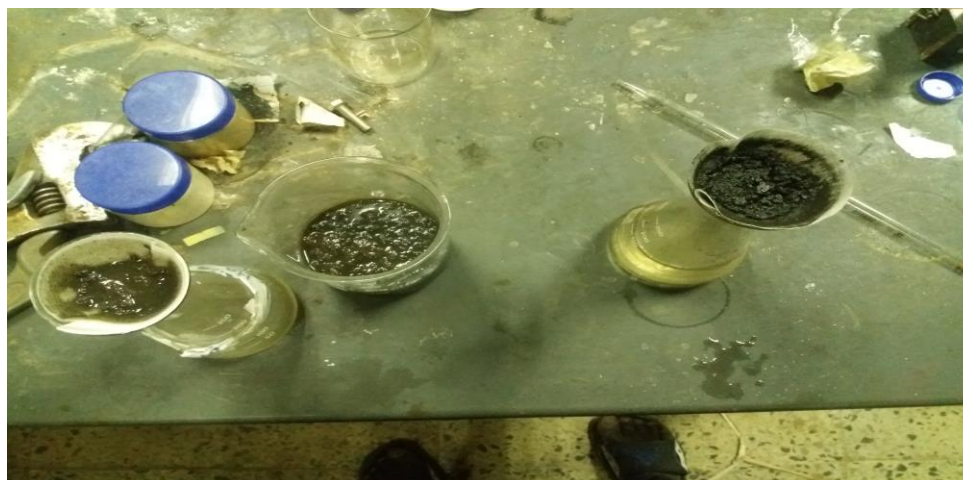
### **3.4 Applications**

The unique properties of  $\text{Ag}_2\text{S}$  NPs led to a variety of application such as in solar cells, photo-detectors, light-emitting diodes and switches, photoconductors, IR detectors, magnetic field sensors, optical filters, super-ionic conductors, solar-selective coatings and room temperature oxygen sensors. Ultraviolet (UV), visible, and near infrared (NIR) imaging with nanoparticles (NPs) has been extensively investigated owing to their utility in many applications, such as

electroluminescent devices, optical modulators, photo detectors, and quantum dot NPs, such as Na<sub>2</sub>S and Na<sub>2</sub>S<sub>2</sub>Se<sub>3</sub>, have particularly emerged as some of the most promising new materials for the development of advanced photoelectric devices.

### 3.5 synthesis of silver sulfide nanoparticles

The synthesis of silver sulfide nanoparticles was synthesized through the wet chemical route with the following fundamental steps. Initially, 1.44g (0.1M), 4.93g (0.3M), 4.97g (0.5M), 5g (0.7M) and 4.99g (0.9M), were dissolved in 82mL of silver nitrate hex hydrate (AgNO<sub>3</sub>·6H<sub>2</sub>O), 1.44g (0.1M), 4.93g (0.3M), 4.97g(0.5M), 5g (0.7M) and 4.99g (0.9M), of sodium sulfide, {(Na<sub>2</sub>S·6H<sub>2</sub>O)} and 2.5g of N.ND were dissolved in 180mL of deionized Water solvent. The solutions were mixed together starting with a silver nitrate solution followed by a Drop wise addition of sodium sulfide and N.ND solutions, respectively. The solution was stirred continuously under magnetic stirrer for 1hours. The precipitate was then centrifuged two times at 4500 rpm for 5 min Followed by washing successively with deionized water and ethanol, respectively. This precipitate was Dried in a vacuum oven circa for 1-2 hours



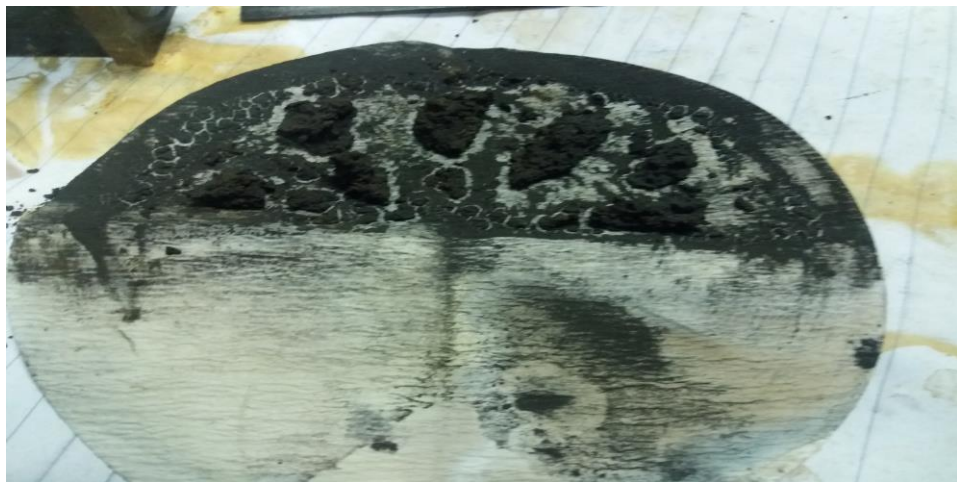
**Figure (3.7) shows the process steps of evaporation the sample**



**Figure (3.8) shows the process of evaporation the sample**



**Figure (3.9) shows the process of evaporation the sample**



**Figure (3.10) shows the sample after drying**





**Figure (3.11) shows evaporation furnace**



**Figure (3.12) shows the sample inside the furnace**



**Figure (3.13) shows the samples inside the test tubes**

# Chapter Four

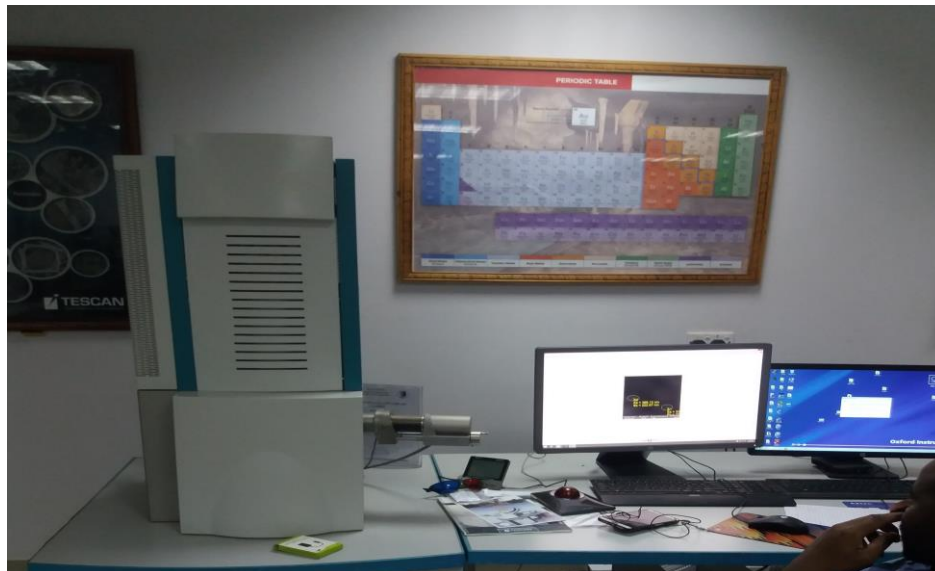
## Results and Discussion

### 4.1 Introduction

In this section we talk about the devices used in this topic and the results obtained, discussed and analyzed and extracted the Nano scale properties of this material that was prepared

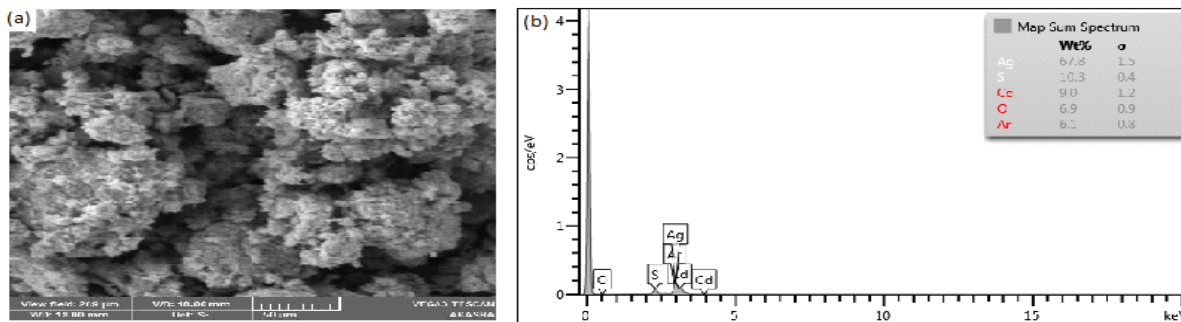
### 4.2 Scanning electron microscopy SEM

The surface morphology and microstructure of silver sulfide nanoparticles Ag<sub>2</sub>S with different concentration were investigated by scanning electron microscopy SEM. The SEM image with 1% by wt. of silver sulfide provide in fig 1 (a) a scanning electron microscopic SEM is a tool for seeing otherwise invisible worlds of micro space (1micron=10m) and Nano space (one Nano space =10m). By using a focused beam of electrons, the SEM reveals levels of detail and complexity inaccessible by light microscopy and Energy Dispersive X-ray EDX data provided in Fig. 1(b) suggests that the peaks corresponding to

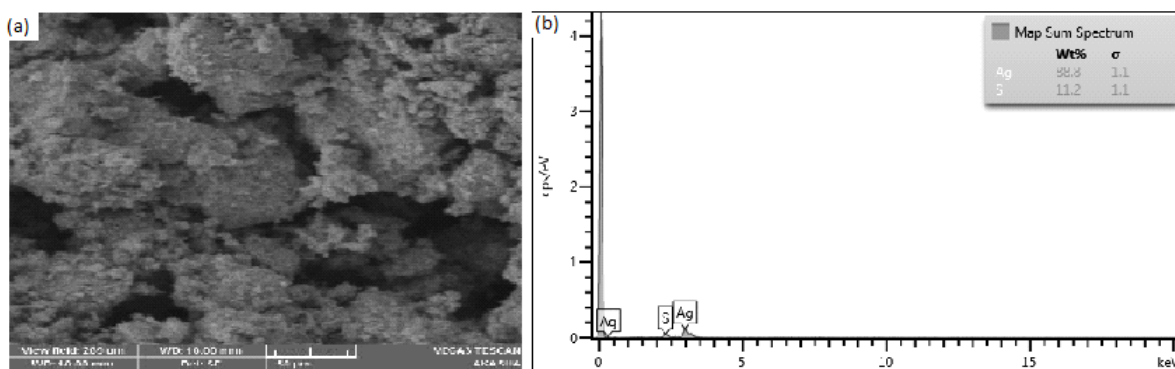


Ag, S

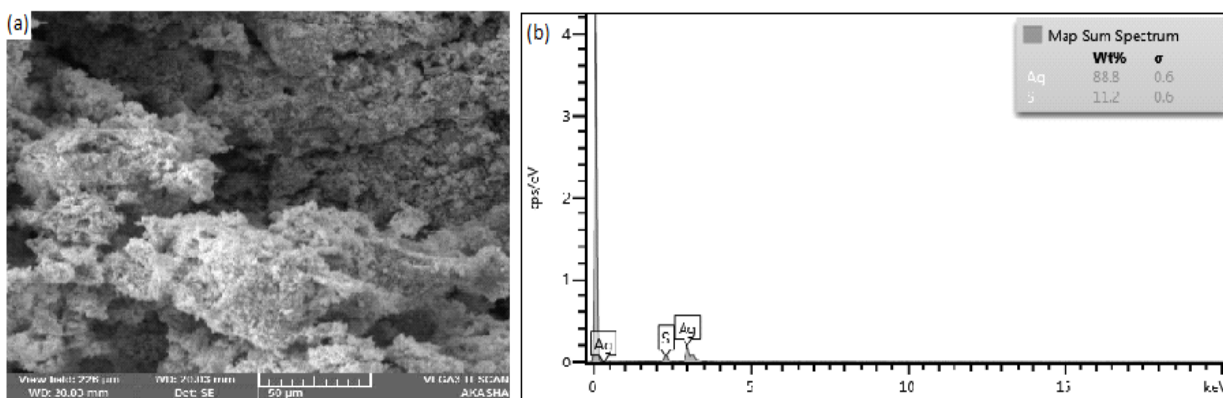
**Figure (4.1) Scanning electron microscopy SEM**



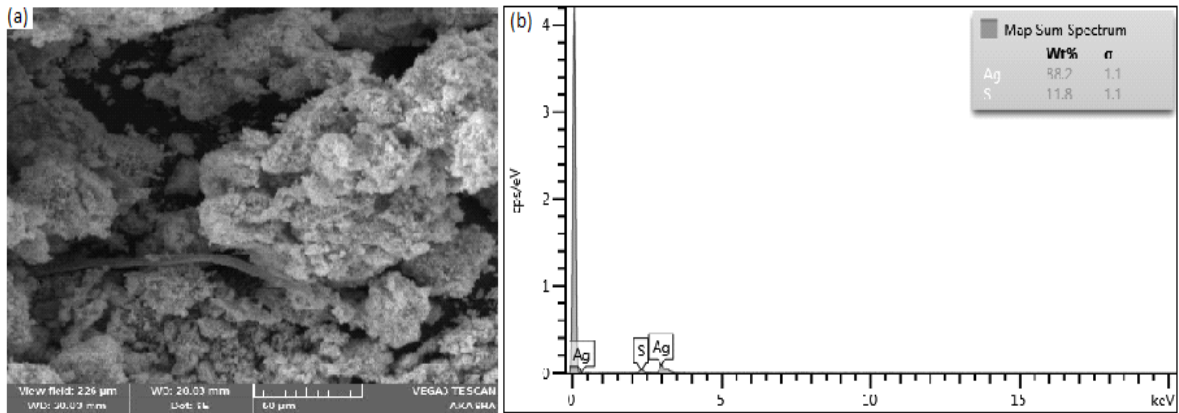
**Figure (4.2) (a) Scanning electron microscopy SEM of silver sulfide with concentration 1% by wt. (b) EDX**



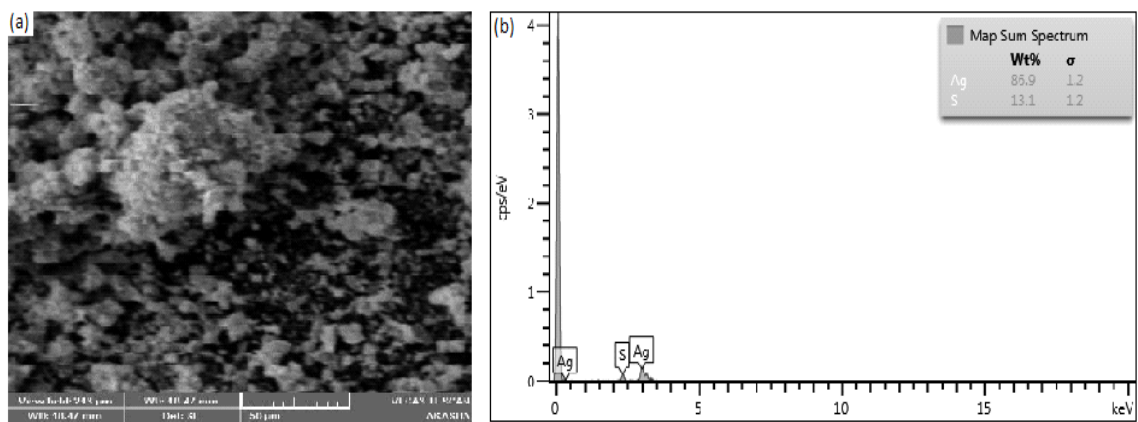
**Figure (4.3) (a) Scanning electron microscopy SEM of silver sulfide with concentration 3% by wt (b) EDX**



**Figure (4.4) (a) Scanning electron microscopy SEM of silver sulfide with concentration 5% by wt (b) EDX**



**Figure (4.5) (a) Scanning electron microscopy SEM of silver sulfide with concentration 7% by wt (b) EDX**



**Figure (4.6) (a) Scanning electron microscopy SEM of silver sulfide with concentration 9% by wt. (b) EDX**

Using the Scanning electron microscopy SEM, consistency appeared in the monocrystalline structure in all the samples according to the concentration of each sample with a slight difference in the sample wt. 1% and appeared when other elements analyzed by X-ray energy dispersal with an increase in the percentage of sulfide in the sample concentration wt. 9%

### **4.3 Transmission electron microscopy TEM**

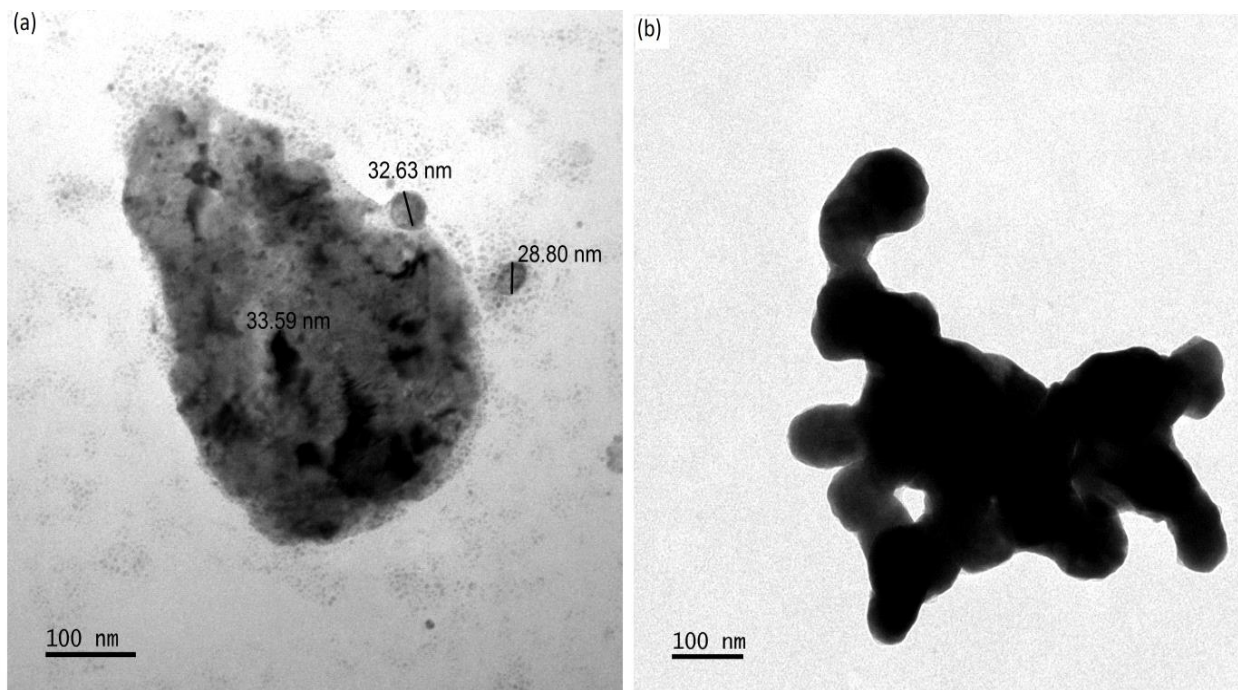
Is a microscopy technique in which a beam of electrons is transmitted through an ultra-thin specimen, interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen;

the image is magnified and focused onto an imaging device, such as a fluorescent screen, on a layer of photographic film, or to be detected by a sensor such as a CCD camera. TEMs are capable of imaging at a significantly higher resolution than light microscopes, owing to the small de Broglie wavelength of electrons. This enables the instrument's user to examine fine detail even as small as a single column of atoms, which is thousands of times smaller than the smallest resolvable object in a light microscope.

TEM forms a major analysis method in a range of scientific fields, in both physical and biological sciences. TEMs find application in cancer research, virology, materials science as well as pollution, nanotechnology, and semiconductor research.



**Figure (4.7) Transmission electron microscopy**



**Figure (4.8.)Transmission electron microscopy TEM of silver sulfide**

#### **4.4 UV – visible absorption**

UV–Vis measurements The UV–Vis spectra (200 to 1100 nm) of the iPP-Ag<sub>2</sub>S com-posites were recorded using a Spectronic Genesys 8 UV/Visible spectrophotometer with compensation for absorption while (190–320 nm) without compensation.



**Figure (4.9) UV- visible absorption**

##### **4.4.1 Optical Absorption**

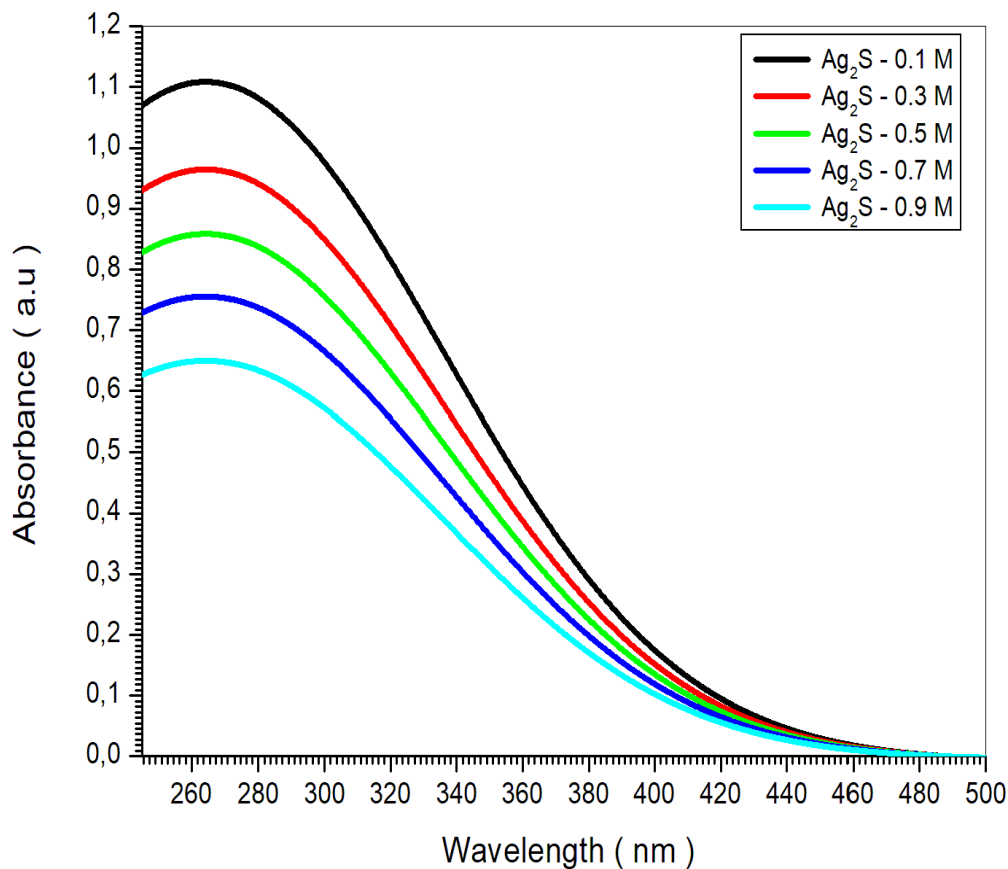
Photons incident on the surface of a semiconductor will be either reflected from the top surface, will be absorbed in the material or, failing either of the above two processes, will be transmitted through the material. For photovoltaic devices, reflection and transmission are typically considered loss mechanisms as photons which are not absorbed do not generate power. If the photon is absorbed it has the possibility of exciting an electron from the valence band to the conduction band. A key factor in determining if a photon is absorbed or transmitted is the energy of the photon. Therefore, only if the photon has enough energy will the electron be excited into the conduction band from the valence band. Photons falling onto a semiconductor material can be divided into three groups based on their energy compared to that of the semiconductor band gap:

$E_{ph} < E_G$  Photons with energy  $E_{ph}$  less than the band gap energy  $E_G$  interact only weakly with the semiconductor, passing through it as if it were transparent.

$E_{ph} = E_G$  have just enough energy to create an electron hole pair and are efficiently absorbed.

$E_{ph} > E_G$  Photons with energy much greater than the band gap are strongly absorbed. However, for photovoltaic applications, the photon energy greater than the band gap is wasted as electrons quickly thermalize back down to the conduction band edges.

Normalized optical absorption properties of the  $Ag_2S$  nanoparticles with different concentration were measured from powder suspension in deionized water is provided in Fig 10



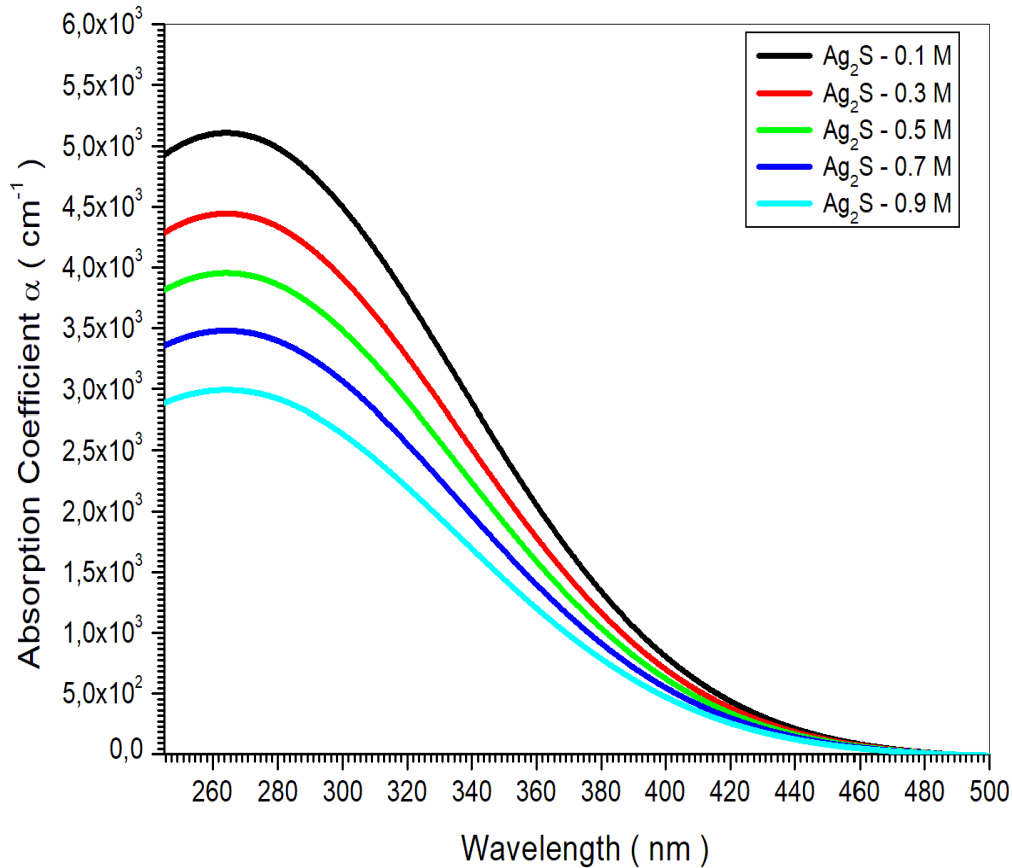
**Figure (4.10) Optical absorption spectra of the silver sulfide Ag<sub>2</sub>S nanoparticles with different concentration.**

#### 4.4.2 Absorption Coefficient

The absorption coefficient determines how far into a material light of a particular wavelength can penetrate before it is absorbed. In a material with a low absorption coefficient, light is only poorly absorbed, and if the material is thin enough, it will appear transparent to that wavelength. The absorption coefficient depends on the material and also on the wavelength of light which is being absorbed. Semiconductor materials have a sharp edge in their absorption coefficient, since light which has energy below the bandgap does not have sufficient energy to excite an electron into the conduction band from the valence



band. Consequently, this light is not absorbed. The absorption coefficient silver sulfide  $\text{Ag}_2\text{S}$  with different concentration is shown below.

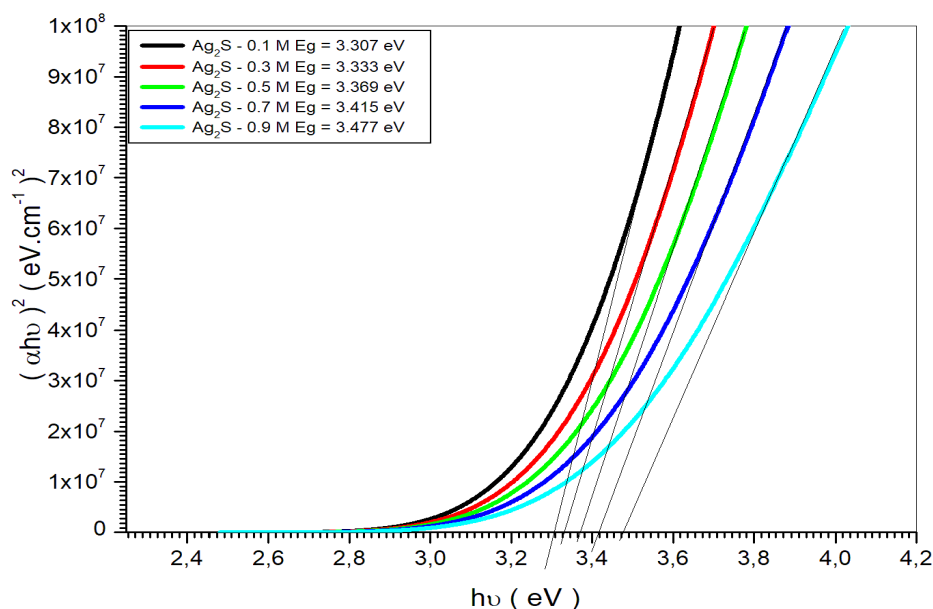


**Figure (4.11) Optical absorption coefficient spectra of the silver sulfide  $\text{Ag}_2\text{S}$  nanoparticles with different concentration**

#### 4.4.3 Optical Energy Gap

The band gap of a semiconductor is the minimum energy required to excite an electron that is stuck in its bound state into a free state where it can participate in conduction. The band structure of a semiconductor gives the energy of the electrons on the y-axis and is called a "band diagram". The lower energy level of a semiconductor is called the "valence band" (EV) and the energy level at which

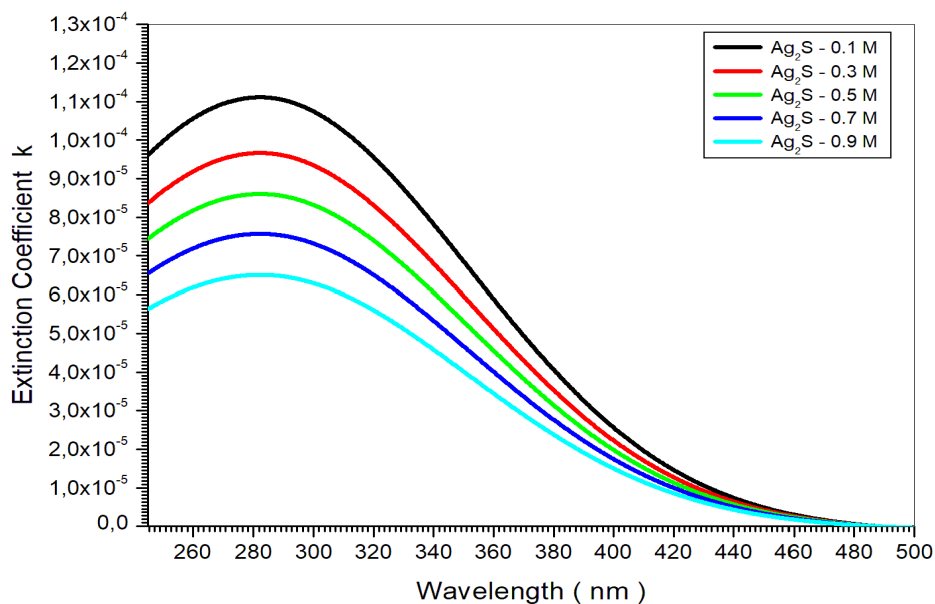
an electron can be considered free is called the "conduction band" (EC). The band gap (EG) is the gap in energy between the bound state and the Free State, between the valence band and conduction band. Therefore, the band gap is the minimum change in energy required to excite the electron so that it can participate in conduction. Figure 8 illustrates the energy band gap of silver sulfide was calculated using the Truces equation in the high absorption region of the semiconductor. Where is a constant,  $E_g$  is the optical energy gap,  $h$  is Planks constant, is the absorption coefficient. Based on the plot of. it is possible to directly determine the energy band gap a material From the intercept of the straight line at  $= 0$ , the energy band gap of  $Ag_2S$  with different concentration such as (1%, 3%, 5%, 7%, and 9%) are determined to be (3.31, 3.33, 3.37, 3.42, and 3.47 eV) respectively, which is similar to the value reported in the literature.



**Figure (4.12) the energy band gap of silver sulfide  $Ag_2S$  nanoparticles with different concentration.**

#### 4.4.4 Extinction coefficient (k)

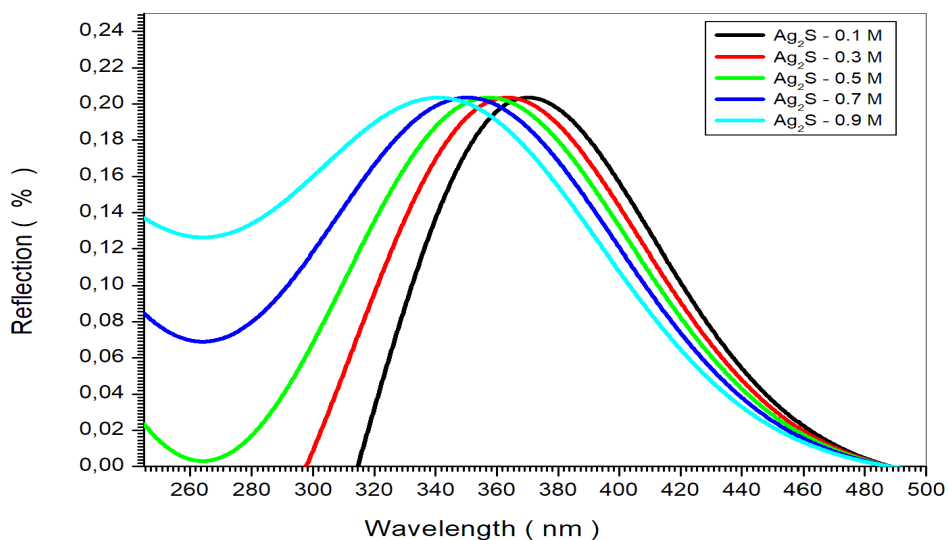
A guide to understanding extinction coefficients, with emphasis on spectrophotometric determination of protein concentration



**Figure (4.13) the extinction coefficient of silver sulfide  $\text{Ag}_2\text{S}$  nanoparticles with different concentrations.**

#### 4.4.5 Reflection

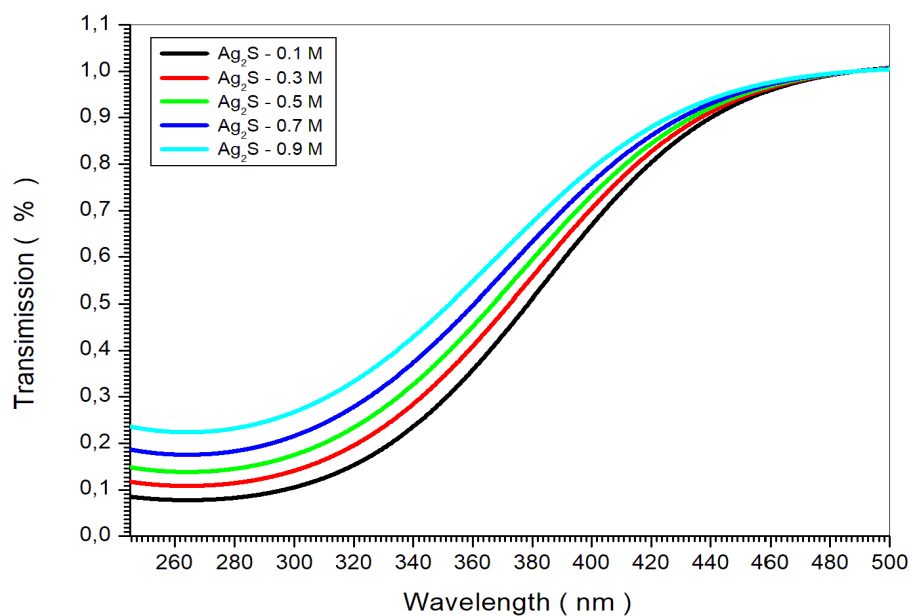
Reflection from rough material surfaces is generally considered to consist of a proportional addition of diffuse reflection and specular reflection.



**Figure (4.14) the reflection of silver sulfide Ag<sub>2</sub>S nanoparticles with different concentrations.**

#### 4.4.6 Transmission

The optical transmittance spectra of the silver sulfide Ag<sub>2</sub>S nanoparticles with different concentrations is shown in Fig 10 which show little more than 90% transmittance throughout the visible range of wavelengths ( $\lambda = 380-770$  nm)



**Figure (4.15) the transmission of silver sulfide Ag<sub>2</sub>S nanoparticles with different concentrations.**

**Table (4.1) the table demonstrates comparison between energy band gap and extinction coefficient and absorption coefficient.**

<b>Slide No.</b>	<b>Energy band gap (eV)</b>	<b>Extinction coefficient K</b>	<b>Absorption coefficient <math>\alpha(cm^{-1})</math></b>
<b>Ag<sub>2</sub>S- 1%M</b>	<b>3.31</b>	<b><math>9.6 \times 10^{-5}</math></b>	<b><math>4.9 \times 10^3</math></b>
<b>Ag<sub>2</sub>S- 3%M</b>	<b>3.33</b>	<b><math>8.4 \times 10^{-5}</math></b>	<b><math>4.3 \times 10^3</math></b>
<b>Ag<sub>2</sub>S- 5%M</b>	<b>3.37</b>	<b><math>7.5 \times 10^{-5}</math></b>	<b><math>3.8 \times 10^3</math></b>
<b>Ag<sub>2</sub>S- 7%M</b>	<b>3.42</b>	<b><math>6.5 \times 10^{-5}</math></b>	<b><math>3.4 \times 10^3</math></b>
<b>Ag<sub>2</sub>S- 9%M</b>	<b>3.48</b>	<b><math>5.6 \times 10^{-5}</math></b>	<b><math>2.9 \times 10^3</math></b>

## **4.5 Conclusions**

In summary, the present study revealed a simple and novel method for the synthesis of highly stable Ag<sub>2</sub>S NPs. Here the synthesized nanoparticles have spherical shape and exhibited photo catalytic property under visible light. Synthesized metallic silver sulfide Ag<sub>2</sub>S nanoparticles by a wet chemistry process. The analysis of SEM, EDX and TEM showed that the particles of all samples were at the Nano - scale. UV–Vis spectra indicated that, as the doping Ag<sub>2</sub>S increased, the band gap shifted to higher wavelengths. the tuning range of emission wavelength is nearly between 380 nm and 450 nm in the present one type doped Ag<sub>2</sub>S nanoparticles The results are clearly showed that the presence of the metallic nanoparticles in the active layer improvement the optical and electrical properties (3.31ev, 3.33ev, 3.37ev ,3.42ev and 3.44ev)

## **4-6 Recommendation**

1. The state interest in scientific research and researchers.
2. Encouraging the scientific researcher to discover and innovate.
3. The state should pay attention to scientific research because it has the benefits of the nation's patronage.
4. The state universities to prepare the scientific environment to be suitable for scientific research.
5. The state should like to provide practical equipment that enables the researcher to complete his scientific research.
6. Laboratories must be equipped with the necessary safety equipment when preparing chemical compounds.
7. Attention should be paid studying the nanoparticles of mineral sulfide because their importance

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optical properties, photovoltaic, Ag<sub>2</sub>S thin films, chemical bath deposition technique, complexing agent ,

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