Zinc Sulfide Nanoparticles - Synthesis and Characterization

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Abstract: SHMP stabilized ZnS nanoparticles were synthesized using an aqueous precipitation method at room temperature. The obtained particles were characterized using XRD, UV-Visible absorption spectrophotometer and EDAX. The particle size was estimated by XRD and optical absorption spectra, it resulted to be below 4 nm with a good agreement between both techniques. This particle size produce a quantum confinement observed in an increment on the band gap value.

Keyword: EDAX, Nanoparticles, quantum confinement, sodiumhexametaphosphate, XRD, ZnS,

I. INTRODUCTION

In recent years, there has been considerable interest in semiconductors of nanometer dimensions due to the quantum size effect that they exhibit. Nanocrystalline semiconductors have electronic properties intermediate between those of molecular entities and macro crystalline solids and are at present the subject of intense research. nanometric semiconductor particles exhibit novel properties due to the large number of surface atoms and the three dimension confinement of electrons[1]. The broad band gap II–VI semiconductors are usual to be the new materials for the Optoelectronic devices. ZnS is an important member of this family and it has been widely investigated [2] as it has many applications. ZnS has been used widely as an chief phosphor for photoluminescence (PL), electroluminescence (EL) and cathode luminescence (CL) devices due to its better chemical stability compared to other chalcogenides material such as ZnSe. ZnS is a commercially important II–VI semiconductor material having a wide optical band gap and it a very attractive material for optical application especially in nanocrystalline form. Zinc sulphide nanoparticles have many applications in solar cells [3], gas sensor [4] and antivirus agent in coating [5]. ZnS has been synthesized using several method as like homogeneous precipitation [6], microwave [7], thermal evaporation [8] and spray pyrolysis [9]. Zinc sulphide is a very important semiconductor with a wide band gap of 3.6 eV. ZnS have two crystal structures zinc blende and wurtzite ,both of which are direct band structure [10,11].

II. EXPERIMENTAL PROCEDURE

A 1.0M solution of zinc acetate and a 0.75M solution of sodium sulfide were prepared in distilled water at room temperature. A 20.4 gm of sodium hexametaphosphate was placed in 150 mL of distilled water and stirred until dissolved, in this 25 mL of the zinc acetate solution was then added and vigorous stirring continued. Finally, in one portion, 25 mL of the sodium sulfide solution was added. Immediately an off-white solution was observed. Stirring was continued for a total of 2 hour. The solution was then washed many times with distilled water, filtered and then dried. ZnS nanoparticles have been prepared of capping agent sodium hexametaphosphate as 102 g L^{-1}. The obtained samples were characterized by X-ray diffraction (XRD), UV-Visible absorption spectroscopy and EDAX.

III. RESULTS AND DISCUSSION

I have to characterize the physical properties of synthesized ZnS nanoparticles. X-ray diffraction (XRD) pattern was used to study the particle size of ZnS nanoparticles. Various spectroscopic techniques like; UV-Visible absorption spectroscopy were employed to study the optical properties of ZnS nanoparticles. EDAX was used to study the elemental analysis of the sample.

IV. X-RAY DIFFRACTION (XRD)

The XRD pattern of prepared sample were taken by Bruker D8 Advance X-ray diffractometer using the characteristic CuKα (1.5418 Å). The size of ZnS nanocrystals has been calculated using Debye-Scherrer formula using (111) reflection from the XRD pattern. Debye-Scherrer formula for crystallite size determination is given by [12] ($t = 0.9\lambda / B \cos \theta_B$) Where t is the crystallite size, λ is the wavelength of X-ray used ($\lambda=1.54A^0$) and B is the full width at half maximum (FWHM) after correcting the instrument peak broadening (B expressed in radians) and θ_B is the Bragg's angle. From this figure, it is clear that the sample prepared at room temperature have a high degree of crystallographic orientation. The higher intensity peak of sample is at $2\theta = 28.8^o$ is the characteristics of cubic (111) plane. In the sample three broad peaks observed in diffractogram at around 28.8^o, 48.4^o and 55.8^o reveals a cubic (Zinc blend) lattice structure of ZnS. These peaks could be easily assigned to the planes (111), (220) and

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(311) respectively of the cubic phase [13]. The peak broadening in all the XRD patterns of ZnS samples clearly indicates the formation of ZnS nanocrystals of small size. The calculated size is found to be 1.2 nm of sodium hexametaphosphate capped ZnS nanoparticles prepared at room temperature.

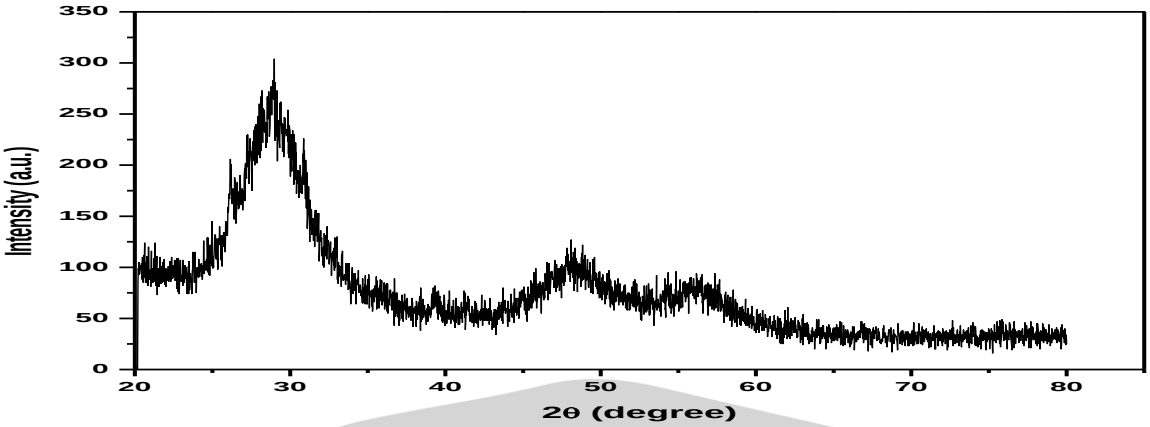

Figure 1: XRD patterns of SHMP capped ZnS nanoparticles.

V. OPTICAL ABSORPTION SPECTROSCOPY

Optical absorption spectrum of ZnS samples has measured at room temperature using a HITACHI-U 3400 UV-Vis Spectrophotometer. The blue shift of the absorption edge arises from quantum confinement effect in the as-prepared ZnS nanoparticles and it is 303 nm. Blue shifting of absorption peak (bulk 345 nm) is due to quantum confinement of the excitons present in the sample resulting in a more discrete energy of the spectrum of individual nanoparticles The relation between the absorption coefficient and the incident photon energy (hν) can be written by well known Tauc relation [14] $\alpha h\nu = A (h\nu - E_g)^n$ Where, A is a constant, hν is the photon energy, E_g is the optical band gap of the material and exponent n =1/2, for allowed direct transition) depends on the type of transition.

A plot of $(\alpha h\nu)^2$ versus photon energy (hν) as shown in figure 3, when extrapolated to zero absorption axis provide the value of optical energy gap. The average particle size present in the nanoparticles can also be determined by using the mathematical model of effective mass approximation [15]. For nanocrystalline ZnS this results in a relation between the particle radius (r, in nanometers) and the band gap (E_g, in electron volts) as follows:

$$r(E_g) = \frac{0.32 - 2.9\sqrt{E_g - 3.49}}{2(3.50 - E_g)}$$

It is clear from the figure 3, that on increasing band gap the particle size decreases. The particle size obtained from the shift in the band gap using above mathematical model of effective mass approximation (EMA) nearly matches and shows same trends that estimated from XRD patterns using Debye-Scherrer formula. Optical absorption spectrum of ZnS sample was measured at room temperature using a HITACHI-U 3400 UV-Vis Spectrophotometer. The as-prepared ZnS nano powder has been suspended in glycerol using magnetic stirrer at optical absorption spectra has been recorded at room temperature in the wavelength range 280-480 nm as shown in figure 2.

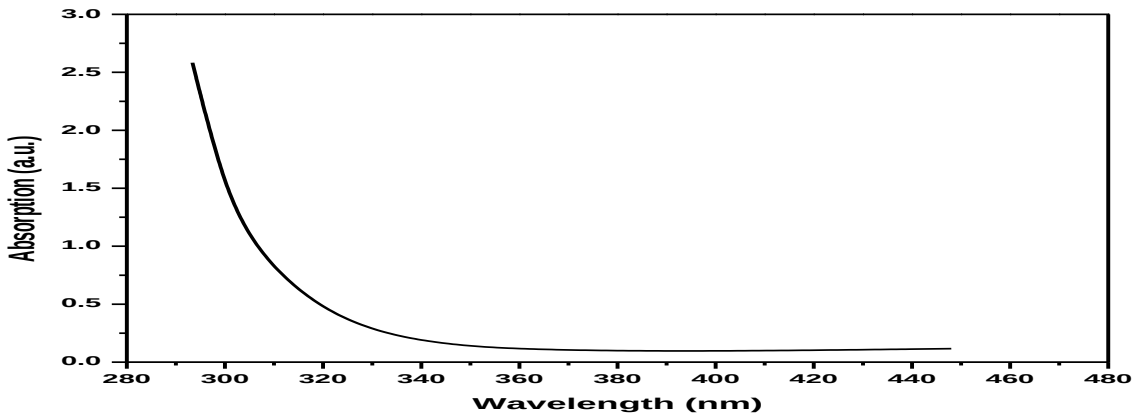

Figure 2: Absorption spectra of SHMP capped ZnS nanoparticles with concentration of SHMP as 102 g L^{-1}.

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A plot of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) as shown in figure 3, when extrapolated to zero absorption axis provide the value of optical energy gap. The obtained band gap value of the sample is 4.08 eV which is blue shifted as compared to the bulk band gap value and is evident of quantum confinement in the as-prepared samples of ZnS nanoparticles.

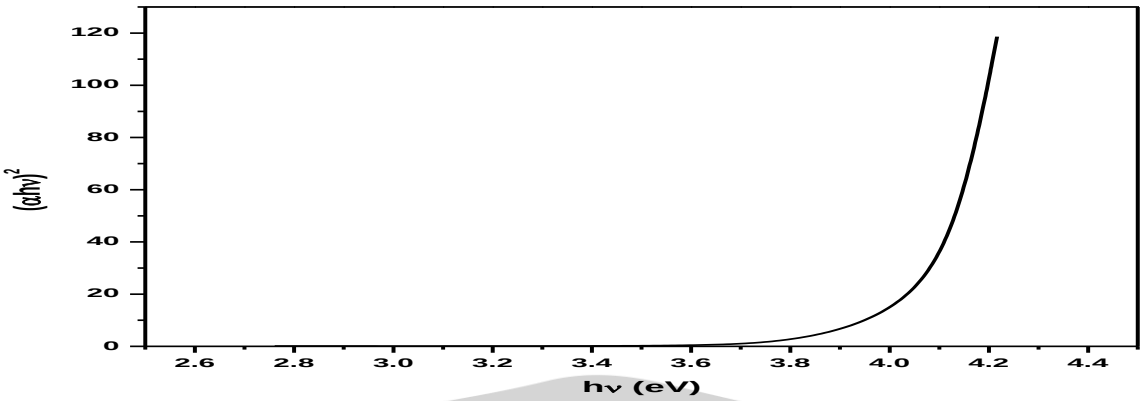

Figure 3: Determination of energy band gap of SHMP capped ZnS nanoparticles with concentration of SHMP as 102 g L^{-1}.

The average particle size present can also be determined by using the mathematical model of effective mass approximation [8] and it is 3.28 nm.

VI. ENERGY DISPERSIVE X-RAY ANALYSIS

EDX is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample.

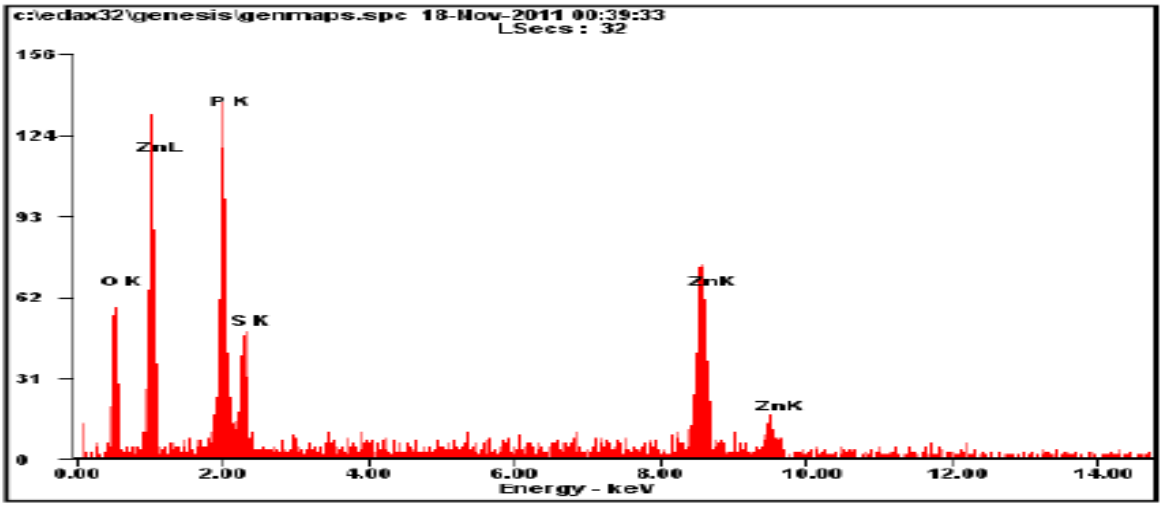

Figure 4: EDAX spectrum of SHMP capped ZnS nanoparticles with concentration of SHMP as 102 g L^{-1}.

The elemental composition of the nanoparticles formed were confirmed by energy dispersive x-ray (EDX) analysis as shown in figure 4.

VII. CONCLUSION

In this research zinc sulfide nanoparticles powder was prepared using zinc acetate. I have successfully synthesized the ZnS nanoparticles by a simple aqueous chemical precipitation method using sodium hexametaphosphate as a capping agent. The ZnS nanoparticles were characterized by X-ray diffraction (XRD), ultraviolet-visible (UV-VIS) and energy dispersive X-ray analysis (EDX). The average crystallite size of zinc sulfide was calculated from the Debye- Scherrer formula ~3.5 nm which is good agreement of UV-Visible spectra. UV spectra revealed that the absorption band was blue shifted from the bulk. EDX is elemental analysis which is used to confirm the presence of zinc sulfide.

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