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# Study of Zinc Sulfide Nanoparticles into Sodium Hexametaphosphate Matrix

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## ABSTRACT

Zinc sulfide nanoparticles are synthesized into sodium hexametaphosphate matrix by chemical precipitation method at room temperature, in this method ZnS nanoparticles were prepared by the reaction of zinc acetate and sodium sulfide into sodium hexametaphosphate . The effects of the process parameters on the properties of ZnS particles were investigated by X-ray diffraction (XRD), UV-Visible spectrophotometer and scanning electron microscopey (SEM). The fact that X-ray have a wavelength of the order of angstroms, which is suitable for inter-atomic distances in solids, makes this technique an excellent instrument to investigate the crystalline structure of the materials. X-ray diffraction (XRD) pattern shows that the particle size decreases (FWHM increases) with increasing the concentration of sodium hexametaphosphate matrix. The optical absorption spectroscopic technique is used to study the electronic band structure of semiconductors and metals for determination of their optical properties. The optical absorption is a result of interaction between the material and light. When a frequency of light is on resonance with the energy difference between states the transition allowed or partly allowed by selection rules, and photon is absorbed by the material which is reflected as a decrease of transmission or an increase in absorbance of the light passing through the sample. By measuring the transmission or absorbance of sample as a function of the wavelength of the light, one can obtains a spectrum characteristic of the material. HITACHI-U 3400 UV-Vis Spectrophotometer determines the band gap of nanoparticles. Scanning electron microscopy (SEM) produces images of the sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, produces various signals that contain information about the sample surface. The average size of ZnS nanoparticles was calculated less than 5 nm by XRD, UV-Visible spectrophotometer and SEM.

**KEYWORDS;** Nanoparticles, colloidal solution, ZnS, sodium hexametaphosphate,

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## INTRODUCTION

Nanotechnology is the latest technology developing rapidly since 1980s. Nanocrystals attracted considerable attention in recent years because of their individual properties, such as quantum size effects[1–2] and luminescence phenomenon[3–5]. ZnS nanocrystals have extensive application in lasers, sensors, infrared windows, displays, and various other fields[6–9]. ZnS nanocrystals have two kinds of structures: zinc blend and wurtzite structure. ZnS is useful in glowing materials commonly have zinc blend structure[10]. In the present research work I have used chemical method to synthesize the ZnS nanostructured material into sodium hexametaphosphate matrix.

## EXPERIMENTAL PROCEDURE

A 1.0M solution of zinc acetate and a 0.85M solution of sodium sulfide were prepared in distilled water. A 16.4 gm of sodium hexametaphosphate was placed in 160 mL of distilled water and stirred until dissolved, 20 mL of the zinc acetate solution was then added and vigorous stirring continued. Finally, in one portion, 20 mL of the sodium sulphide solution was added. Immediately an off-white solution was observed. Stirring was continued for a total of 1 hour. The solution was then washed two times with distilled water, filtered and then dried. ZnS nanoparticles has been prepared of capping agent SHMP as 82 g $L^{-1}$. The obtained samples were characterized by X-ray diffraction (XRD), UV-Visible absorption spectroscopy and Scanning electron microscopy (SEM).

## RESULTS AND DISCUSSION

We have used a number of experimental techniques to characterize the physical properties of as-synthesized ZnS nanoparticles. X-ray diffraction (XRD) patterns were used to study the phase, lattice constants and particle size of ZnS nanoparticles. Various spectroscopic techniques like; UV-Visible absorption spectroscopy were employed to study the optical properties of ZnS nanoparticles. Scanning electron microscopy (SEM) was used to study the morphology of the sample.

### *X-ray diffraction (XRD)*

The XRD patterns of prepared samples were taken by Bruker D8 Advance X-ray diffractometer using the characteristic CuKα (1.5418 Å), lines of metal anticathode. The peak broadening in all the XRD patterns of ZnS samples clearly indicates the formation of ZnS nanocrystals of small size. The peak broadening at lower angle is more meaningful for the calculation of particle size; therefore 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[11] ($t = 0.9\lambda / B \cos \theta_B$) 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 $\theta_B$ is the Bragg's angle. Figure shows the XRD patterns of ZnS nanoparticles ZnS. From this figure, it is clear that all sample prepared at room temperature have a high degree of crystallographic orientation. The higher intensity peak of sample is at $2\theta = 28.7^o$ is the characteristics of cubic (111) plane. In the sample three broad peaks observed in diffractogram at around $28.7^o$, $48.3^o$ and $56.1^o$ reveals a cubic (Zinc blend) lattice structure of ZnS. These peaks could be easily assigned to the planes (111), (220) and (311) respectively of the cubic phase[12]. The peak broadening in all the XRD patterns of ZnS samples clearly indicates the formation of ZnS nanocrystals of small size. The size of ZnS nanocrystals has been calculated using Debye-Scherrer formula using (111) reflection from the XRD pattern. The calculated size is found to be 1.72 nm of sodium hexametaphosphate capped ZnS nanoparticles prepared at room temperature.

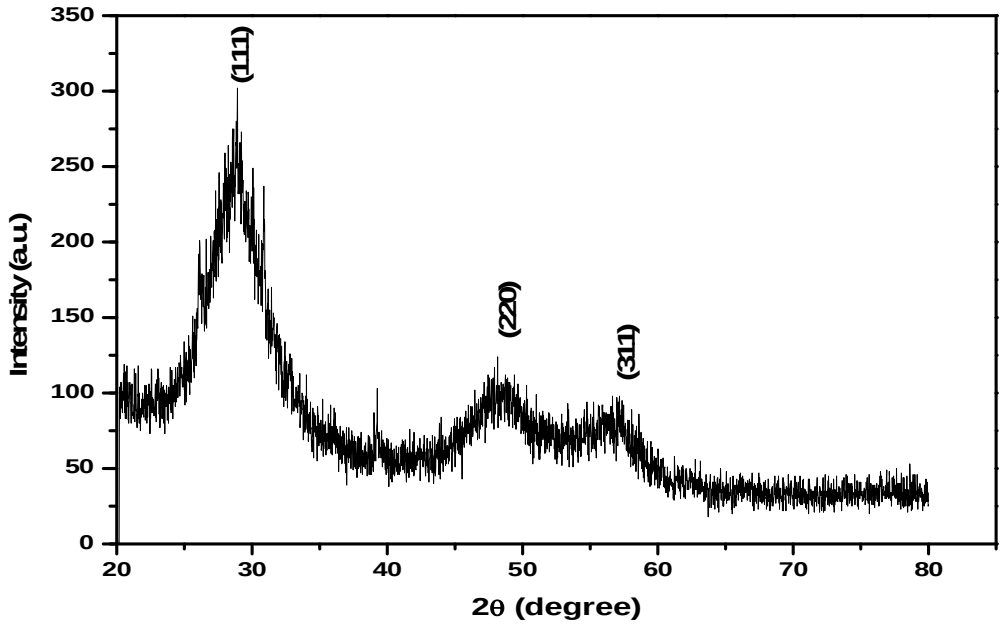


**Figure 1: XRD patterns of SHMP capped ZnS nanoparticles.**

## *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 edges for different sized nanocrystals arises from quantum confinement effect in the as-prepared ZnS nanoparticles. The fundamental absorption, which corresponds to electron excitation from the valance band to

conduction band, can be used to determine the value of optical band gap of the ZnS nanoparticles. Optical excitation of electrons across the band gap is strongly allowed transition thus it causes an abrupt increase in the absorbance at wavelength corresponding to gap energy. Blue shifting of absorption peak (bulk 344 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 (alpha) and the incident photon energy (hν) can be written by well known Tauc relation[13] $\alpha h\nu = A (h\nu - E_g)^n$ 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. Taking the logarithm of above Tauc's equation, we have

$\ln \alpha = \ln A - \ln h\nu + n \ln (h\nu - E_g)$

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[14]. 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 were 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 250-700 nm as shown in figure. 2. The blue shift of the absorption edges for different sized nanocrystals arises from quantum confinement effect in the as-prepared ZnS nanoparticles.

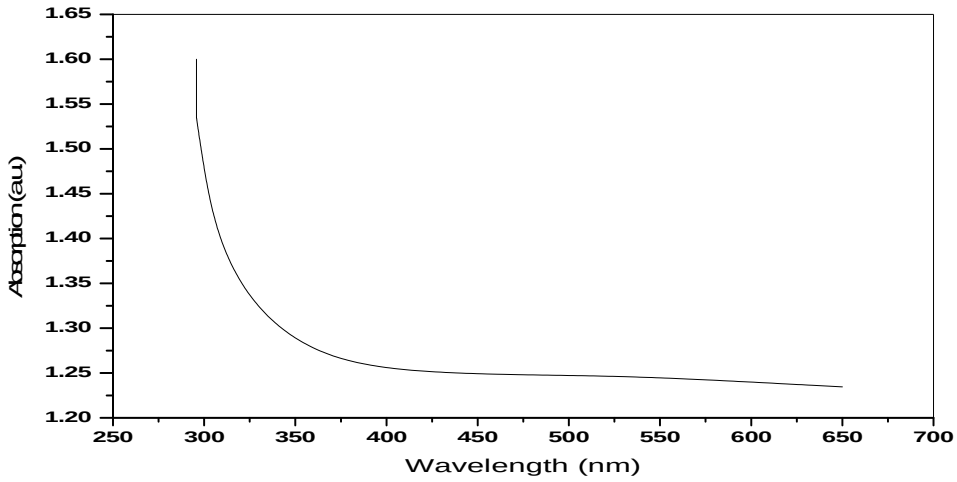


**Figure 2: Absorption spectra of SHMP capped ZnS nanoparticles with concentration of SHMP as 82 g $L^{-1}$.**

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 values of the samples is 3.71 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.

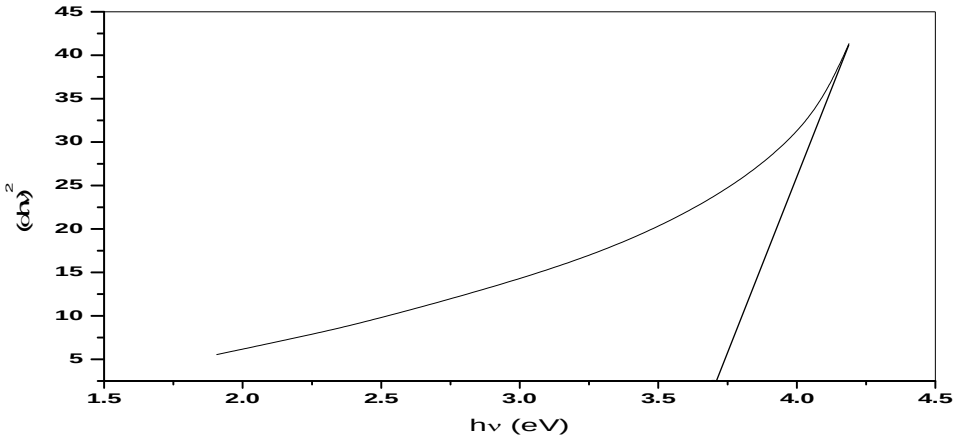


**Figure 3: Determination of energy band gap of SHMP capped ZnS nanoparticles with concentration of SHMP as 82 g $L^{-1}$.**

The average particle size present can also be determined by using the mathematical model of effective mass approximation[14] and it is 4.95 nm

## *Scanning electron microscopy (SEM)*

The morphology of the SHMP capped ZnS nanoparticles was studied by the SEM. Figure 4 shows the SEM image of SHMP capped ZnS nanoparticles.

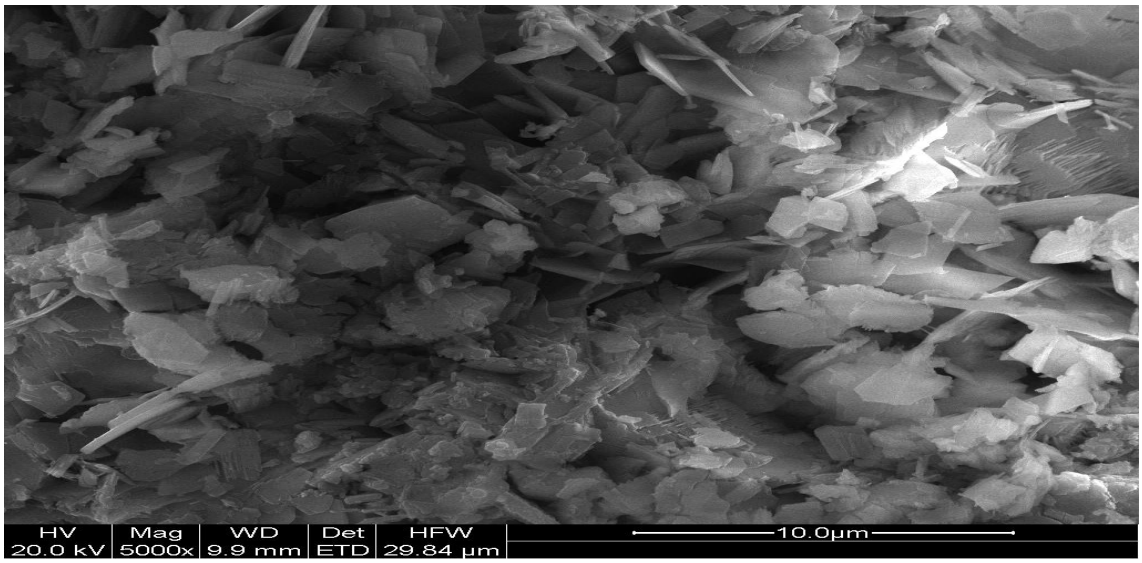


**Figure 4: Scanning electron microscope (SEM) image of SHMP capped ZnS nanoparticles.**

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