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Effect of trisodium citrate concentration on the particle growth of ZnS nanoparticles

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Abstract

ZnS nanoparticles were prepared by simple wet chemical precipitation method to study the effect of sulfur and trisodium citrate concentration on the particle growth of ZnS nanoparticles. Particle sizes were seen varied with the concentration of the sulfur and trisodium citrate. Compressive strain was observed only for a particular concentration of sulfur, and that sample was chosen for trisodium citrate experiments. Interestingly, the compressive strain was seen varied with the concentration of trisodium citrate and seen maximum for 2 M concentration of trisodium citrate. Further, interband transition was observed in the case of smaller crystallites prepared in the trisodium citrate. It is clearly observed from the FTIR analysis that the trisodium citrate acts as a stabilizer and capped the particles from further growth.

Keywords: ZnS nanoparticles; Wet chemical preparation; Crystallites

Background

It is a well-known fact that the semiconductor nanoparticles exhibit novel optical, electronic, and physical properties than their bulk counterparts. When the size of the nanoparticles becomes less or comparable to the Bohr diameter of the exciton [1], various size quantization effects such as widening of band gap and formation of discrete orbitals come into the picture as explained by the Heisenberg uncertainty principle. Among the semiconducting nanoparticles, ZnS is one of the most important materials in the electronic/optoelectronic industry with prominent applications [2] in flat-panel displays [3], white light LEDs [4], electroluminescence devices [5], sensors [6], lasers [7], infrared windows [8], ultraviolet (UV) lasers [9], antireflection coating on solar cells [10], solar cells [11], and for investigating cellular interactions [12], for both *in vivo-in vitro* imaging applications [13], and memristor applications [14]. Basically, ZnS has negative conductivity [15,16] and has a small exciton Bohr radius of about 2.5 nm [17]. Particle size reduction has a tremendous effect on the properties of the ZnS such as blueshift in the optical absorption spectrum, increased luminescence, enhanced oscillator strength, nonlinear optical effects, geometrical structure, chemical bonds, ionization

potential, mechanical strength, melting point, etc. Furthermore, ZnS relates more popular ZnO in terms of atomic structure and chemical properties. Certain properties of ZnS are unique and advantageous compared to that of ZnO. ZnS has a larger band gap than that of ZnO (approximately 3.4 eV [17]), and therefore, it is more suitable for visible UV light-based devices such as sensors/photodetectors. However, nano-sized ZnS has not been investigated in much detail compared to ZnO nanomaterials.

This paper discusses the preparation of zinc sulfide nanocrystals by simple wet chemical precipitation method [18-21] and studies the effect of the concentration of sulfur and trisodium citrate on nanoparticle formation since trisodium citrate acts as surfactant and capping agent that can modify the surface of ZnS particles and prevents the growth of the particles.

Results and discussion

ZnS nanocrystalline powder synthesis by varying sulfur concentration

The prepared ZnS powder samples were analyzed by powder X-ray diffraction technique, scanning electron microscopy (SEM), energy dispersive X-ray (EDX), and UV-visible absorption spectrophotometer. The X-ray diffractogram of the prepared samples well correlates with the American Society for Testing and Materials

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(ASTM) card no. 77–2100 of cubic structure, and the diffraction peaks from the planes (111), (220), and (311) were noted in the diffractograms shown in Figure 1. All of the X-ray diffractogram peaks and the peak broadening confirmed that the prepared sample contains pure cubic ZnS nanocrystallites. Their crystallite size and the strain were obtained from the

Williamson-Hall plot [22]. The crystallite size, strain, lattice parameters, and the volume of the crystals of each sample are shown in Table 1, and it is seen that the prepared ZnS powder consists of nanoparticles. Table 1 clearly shows the compressive strain for the samples ZnS_{0.1} and ZnS_{0.05}, a property of nanoparticles.

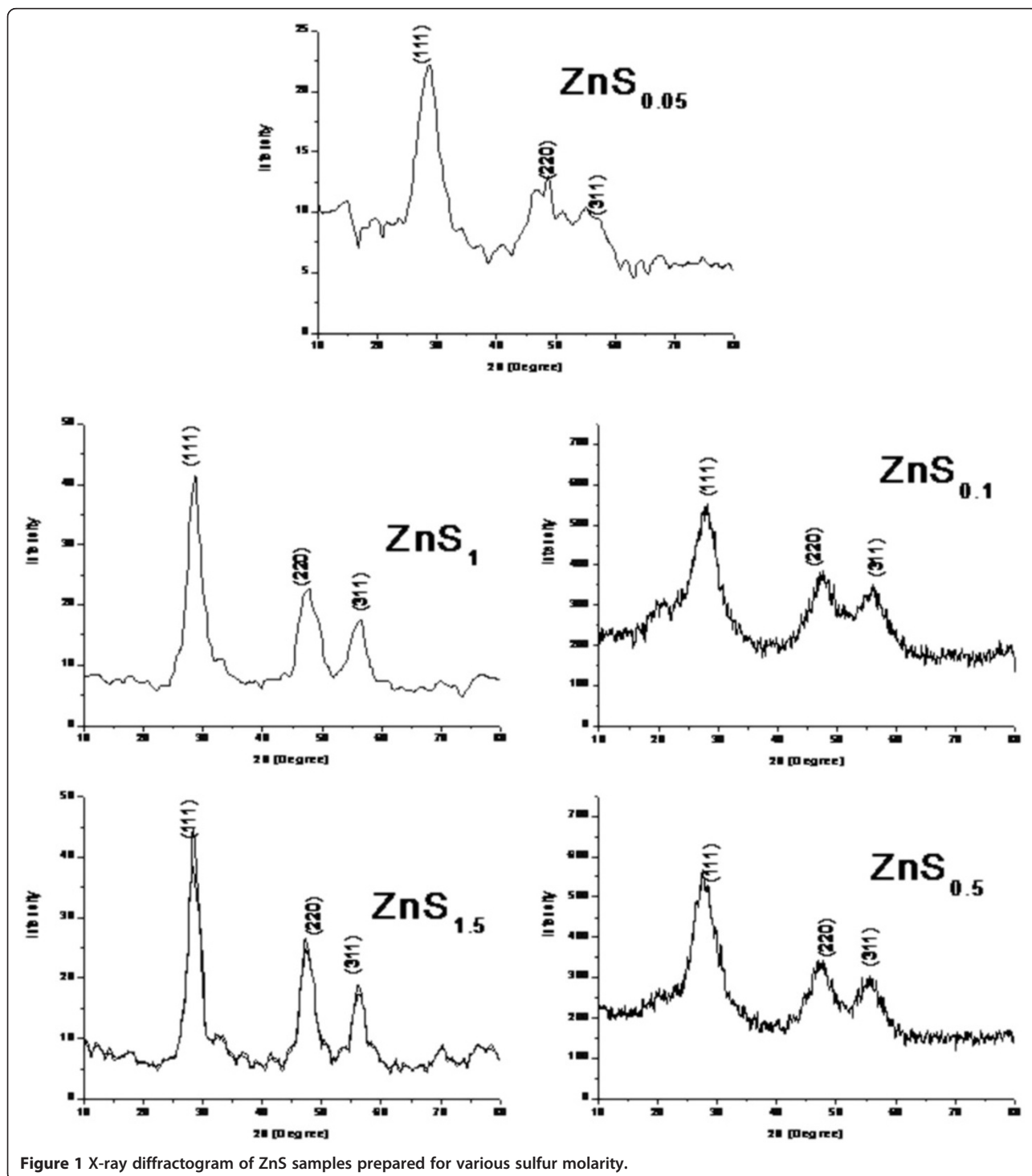


Figure 1 X-ray diffractogram of ZnS samples prepared for various sulfur molarity.

Table 1 Lattice parameters and volume of crystals

Samples	Lattice parameters (ASTM values are shown in bracket) (Å)			Average crystallite size (nm)	Average strain	Volume (10^{-28} m^3) (ASTM values are shown in bracket)
	a	b	c			
ZnS _{1.5}	5.4159 (5.4146)	-	-	6.91	0.0279	1.5905 (1.5875)
ZnS ₁	5.4106 (5.4146)	-	-	5.35	0.0441	1.5839 (1.5875)
ZnS _{0.5}	5.5138 (5.4146)	-	-	2.91	0.0265	1.6763 (1.5875)
ZnS _{0.1}	5.4289 (5.4146)	-	-	2.43	-0.0167	1.6001 (1.5875)
ZnS _{0.05}	5.3949 (5.4146)	-	-	2.90	-0.0351	1.5702 (1.5875)

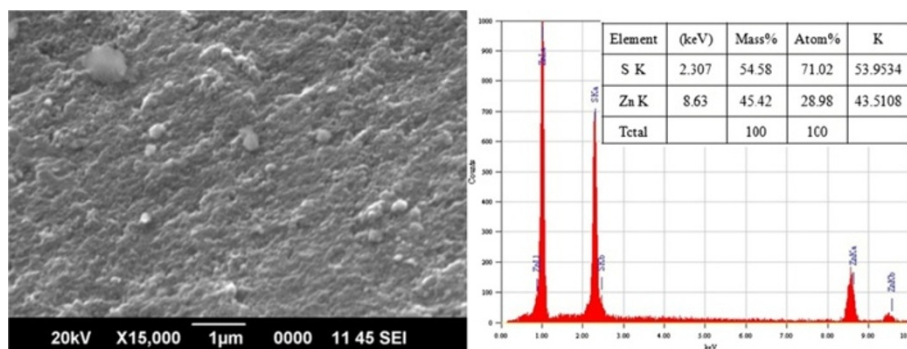


Figure 2 SEM micrograph and EDX of ZnS prepared with 1 M Na₂S solution (ZnS₁).

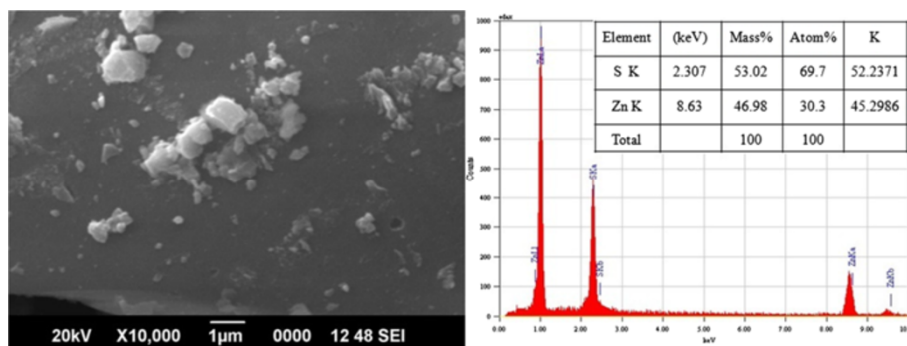


Figure 3 SEM micrograph and EDX of ZnS prepared with 0.5 M Na₂S solution (ZnS_{0.5}).

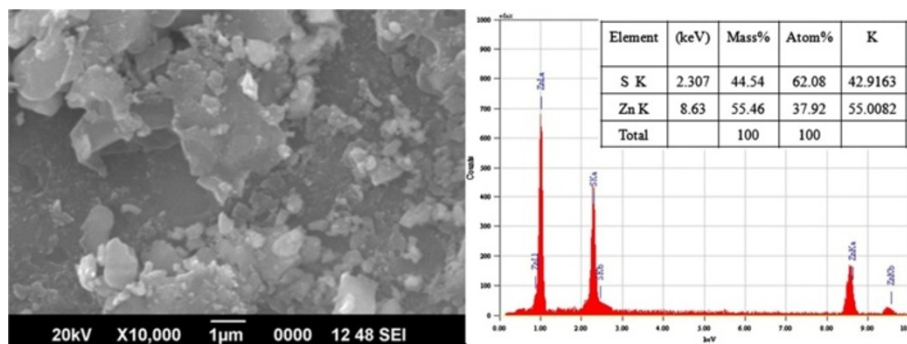


Figure 4 SEM micrograph and EDS of ZnS prepared with 0.1 M Na₂S solution (ZnS_{0.1}).

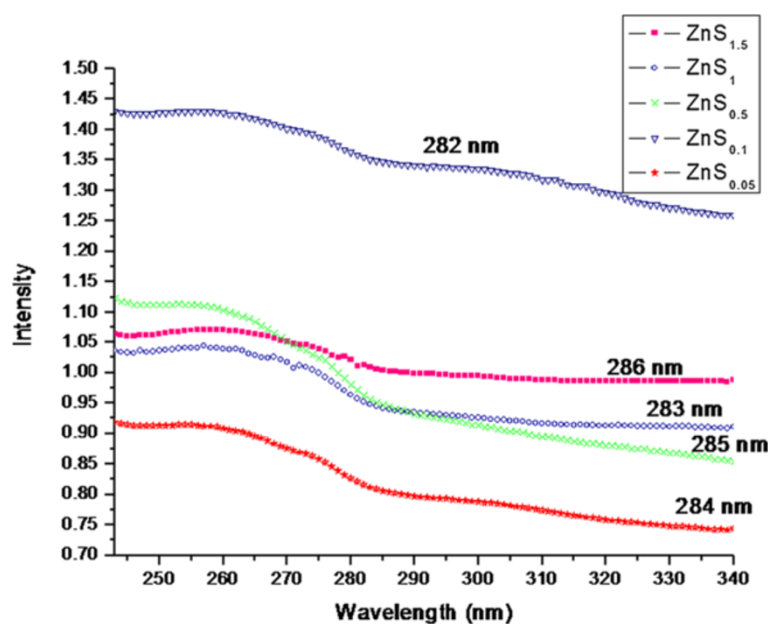


Figure 5 Absorption spectrograph of ZnS prepared in various concentrations of Na₂S solution.

EDX analysis of smaller crystallite samples were performed to find their elemental composition, and the results are shown in Figures 2, 3, and 4, where the elemental data are shown in the inset tables of Figures 2, 3, and 4. These data confirm that the prepared powder consist of only ZnS compound as seen from the X-ray diffractograms of those samples (Figure 1).

To obtain the visual image of the nanoparticles, these nanocrystalline powder samples were analyzed by SEM, and the micrographs are shown in Figures 2, 3, and 4. All of the SEM micrographs reveal that the nanograins are agglomerated due to the drying process, and the sizes of the particles are much larger than the values found from the XRD data. The discrepancy associated with the size of ZnS nanoparticles found from XRD and SEM may be because of the fact that SEM shows the lateral dimension of the particles whereas XRD gives the regularity in the atomic arrangement. Perhaps the lateral dimension is of the order of a few tens of nanometers, but the individual nanoparticle with regular atomic lattice extends only up to a few nanometers.

The study of optical absorption is important to understand the behavior of semiconductor nanocrystals since the fundamental property of semiconductors is the band gap that is the energy separation between the filled valence band and the empty conduction band. The optical excitation of electrons across the band gap is strongly allowed that produces an abrupt increase in absorption at the wavelength corresponding to the band gap energy. This feature of the optical spectrum is

known as the optical absorption edge. High-quality ZnS structures have good ultraviolet optical properties at room temperature, and Figure 5 shows the optical absorption spectra of the prepared ZnS nanoparticles which is in the range of 800 to 200 nm. The absorption edge was obtained at shorter wavelengths compared to the absorption edge of the bulk materials due to the increase in band gap as a result of the quantum confinement effect as well as the discrete energy bands. Strong confinement is observed when the crystallite or particle size is smaller than the Bohr exciton radius of ZnS (approximately 2.5 nm [23]) as a result of this strong confinement absorption peaks which appear in the absorption spectra due to subband transitions, so that in this case, band gap can be found from the maximum of this absorption peak. However, when the crystallite size increases above the Bohr radius, the peak structure disappears and attains the normal interband transition spectrum [24] having an absorption edge at a lower wavelength than the bulk

Table 2 Crystallite size and band gap

Samples	Crystallite size (nm)	Band gap from Brus equation (1) (eV)	Band gap from absorption spectra (eV)	Absorption edge (nm)
ZnS _{1.5}	6.91	3.75	4.33	286
ZnS ₁	5.35	3.83	4.38	283
ZnS _{0.5}	2.91	4.37	4.35	285
ZnS _{0.1}	2.43	4.71	4.40	282
ZnS _{0.05}	2.90	4.37	4.37	284

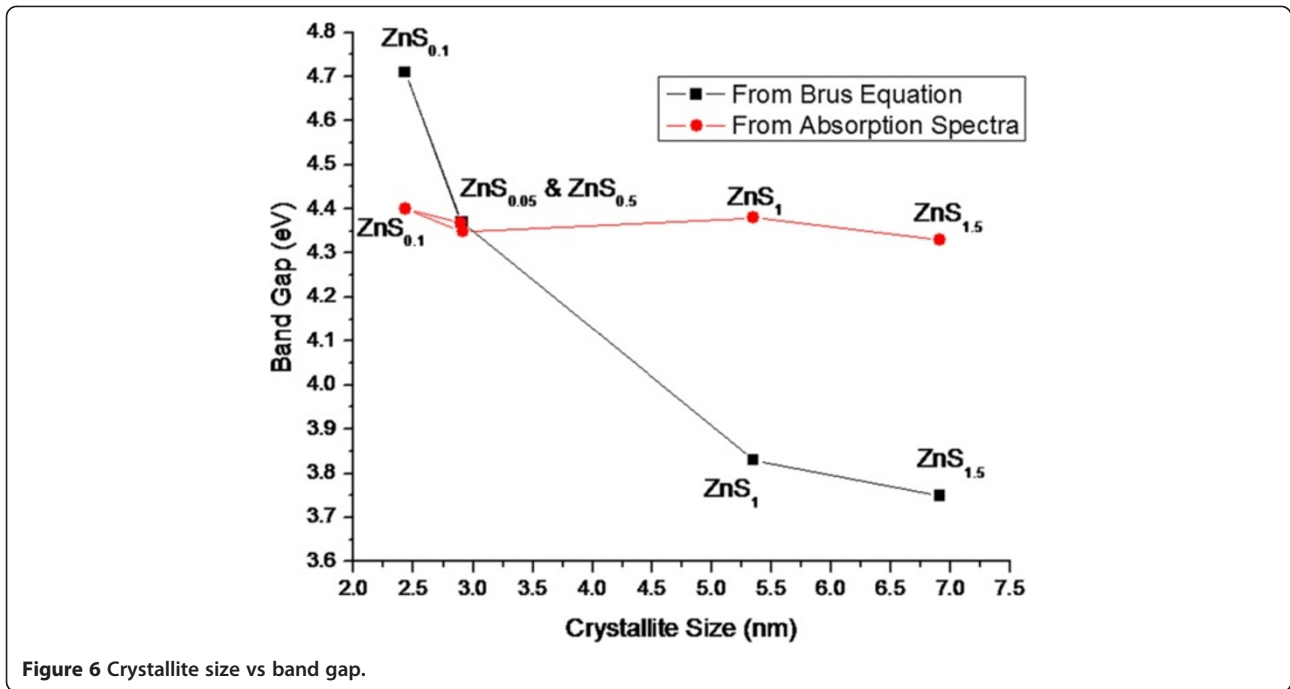


Figure 6 Crystallite size vs band gap.

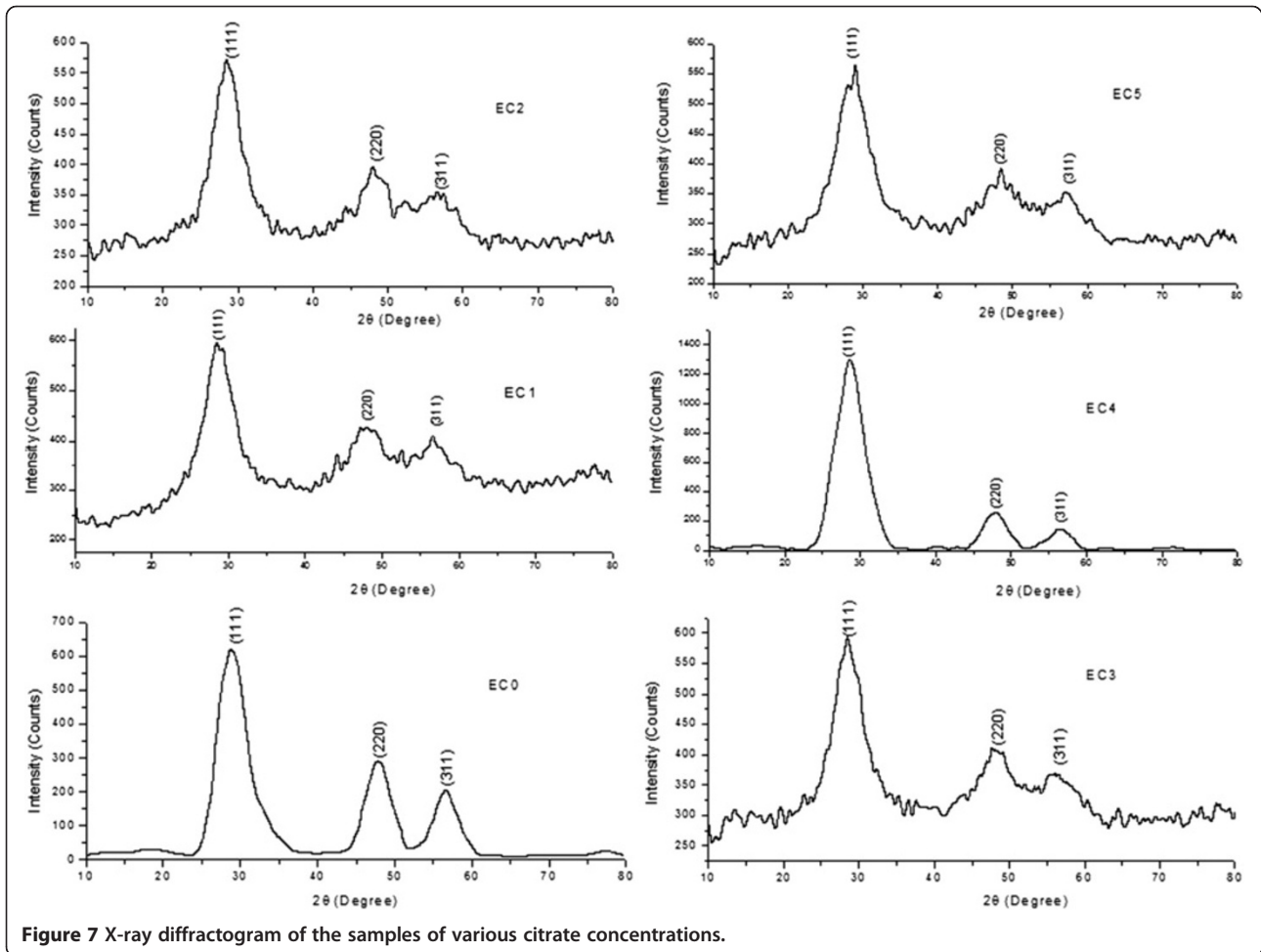
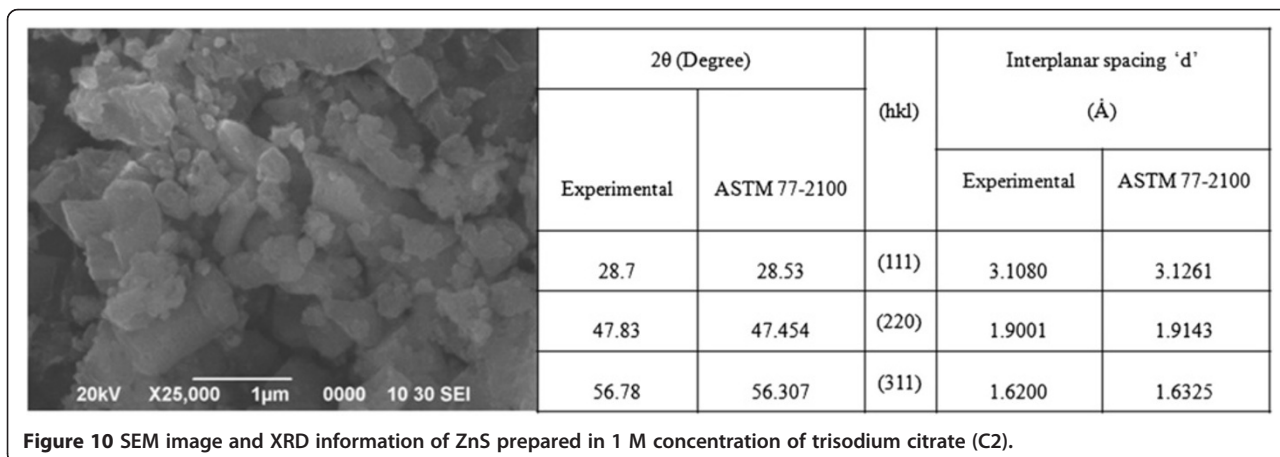
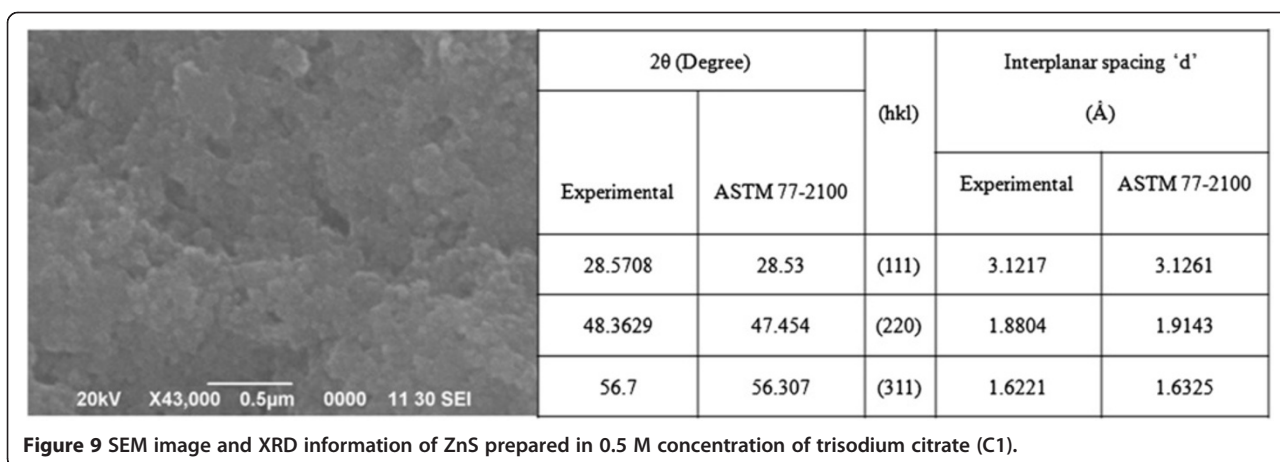
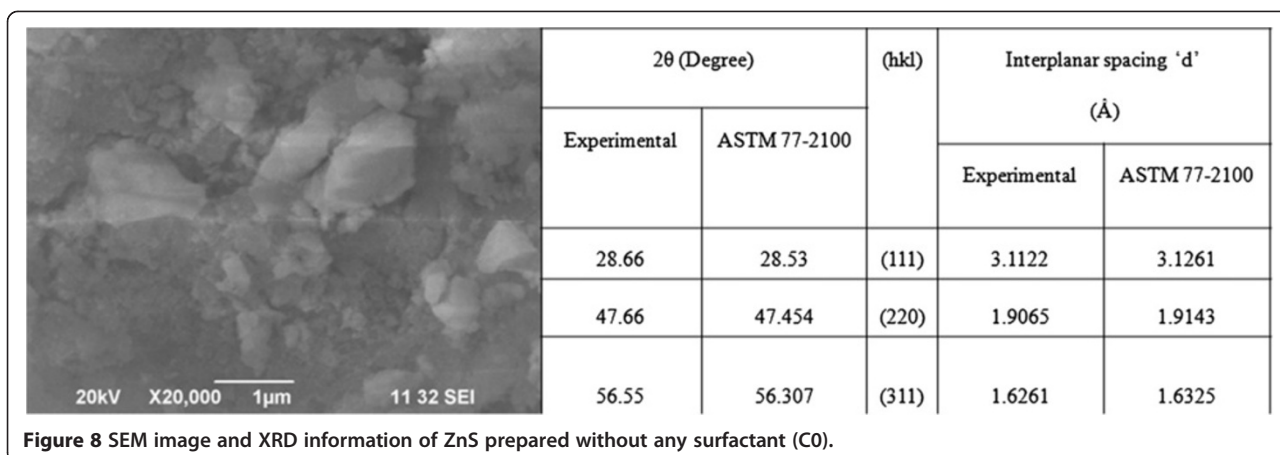


Figure 7 X-ray diffractogram of the samples of various citrate concentrations.



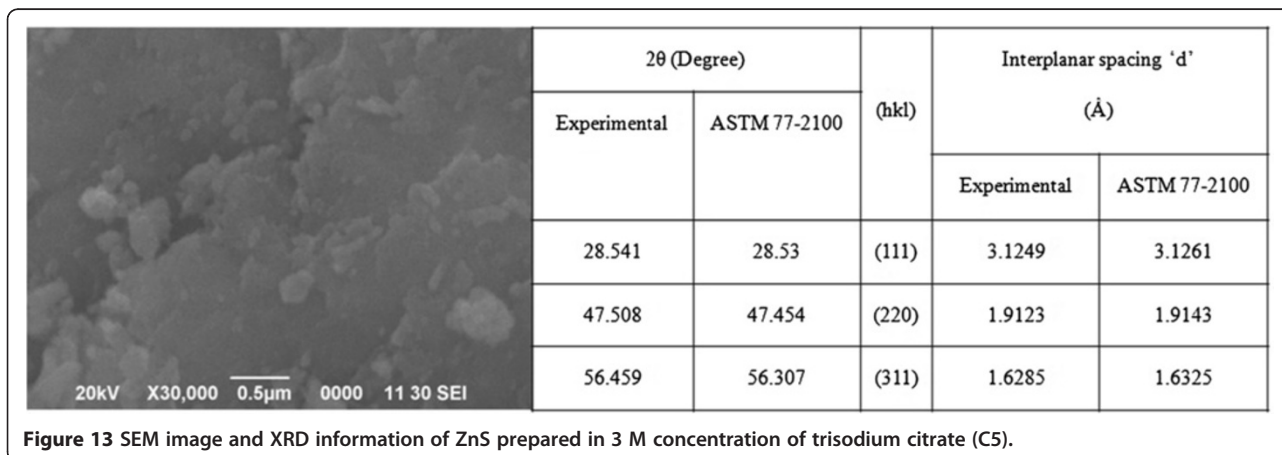
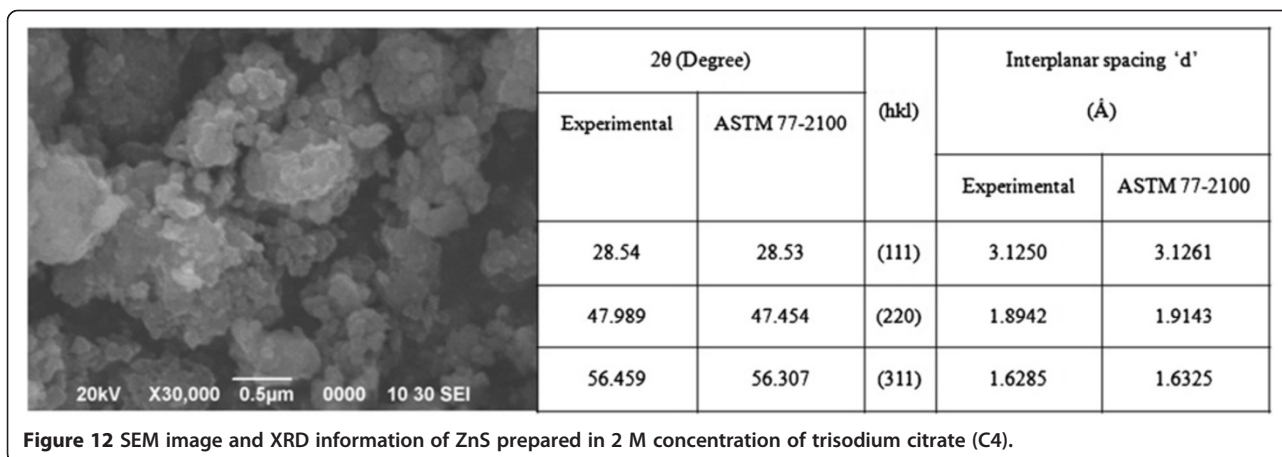
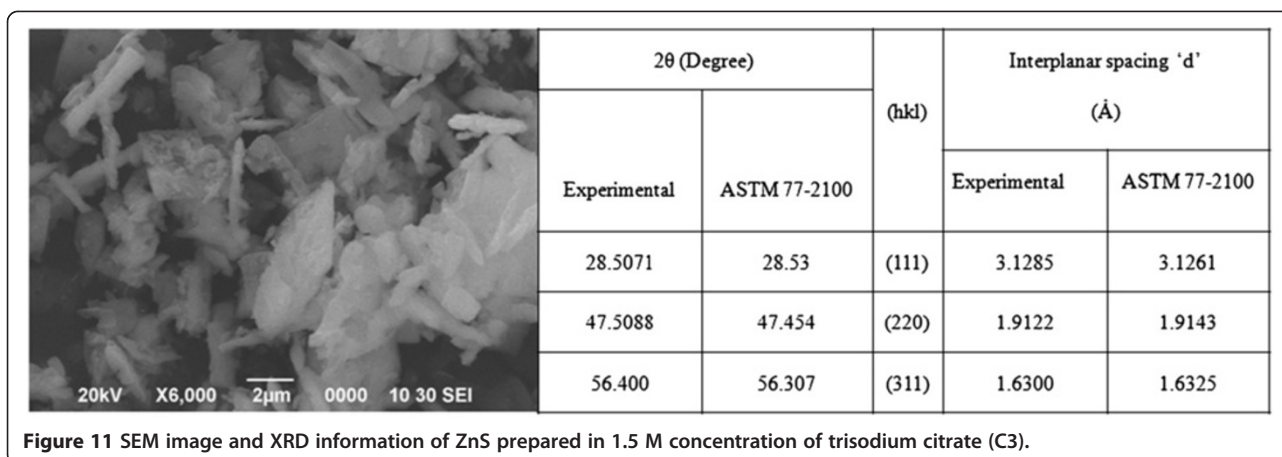


Table 3 Lattice parameters, crystallite size, strain, and volume of crystals

Samples	Lattice parameters (ASTM value in brackets) (Å)			Average crystallite size (nm)	Average strain	Volume (10^{-28} m^3) (ASTM value in brackets)
	a	b	c			
C0	5.3921 (5.4146)	-	-	2.94	-0.0136	1.5677 (1.5874)
C1	5.3686 (5.4146)	-	-	1.74	-0.0247	1.5474 (1.5874)
C2	5.3769 (5.4146)	-	-	1.73	-0.0872	1.5545 (1.5874)
C3	5.4113 (5.4146)	-	-	2.25	-0.0341	1.5846 (1.5874)
C4	5.3906 (5.4146)	-	-	1.46	-0.0119	1.5664 (1.5874)
C5	5.4075 (5.4146)	-	-	1.95	-0.0531	1.5812 (1.5874)

materials. In the bulk material or in thin films, the band gap can be found from the plot between $(\alpha h\nu)^2$ vs $h\nu$, whereas in the nanomaterials of sizes above Bohr radius, the band gap is obtained from the absorption edge, but when the nanomaterial size is less than Bohr exciton radius of the materials, it is obtained from the absorption peak.

When comparing the absorption spectrogram of each of the samples, it is well clear that the ZnS nanocrystalline powder prepared with 0.1 M of Na_2S has the highest absorption intensity. Highest intensity of absorption means that there is a higher number of particles to absorb as well as this sample has the highest capacity to absorb the incident light. Each of the samples has the absorption around 280 nm (4.4 eV) which was blueshifted from 336 nm (3.7 eV) of the bulk ZnS [25]. The blueshifted absorption also confirms that the results obtained from the X-ray diffractograms and the SEM micrographs, that the powder samples are made up of nanocrystals, since the reduction in the size of particles leads to widening of the band gap and this widening is observable in the optical absorption spectra, where a blueshift is observed with the reduction in particle size. Brus [26] has proposed a theoretical model

relating the effective band gap of a material with the particle size. The simple model predicting the variation of exciton energy with particle size is based on the effective mass approximation. The lowest excited state of the crystallite is assumed to be the ground state of an electron hole pair. The ground state energy of an exciton or the increase in effective band gap as a function of crystalline size is estimated as

$$E_g(r) = E_{g,\text{bulk}} + \frac{h^2}{8r^2} \left(\frac{1}{m_e} + \frac{1}{m_h} \right) - 1.8 \frac{e^2}{4\pi\epsilon_r\epsilon_0 r} \quad (1)$$

where $E_g(r)$ is the band gap of the nanoparticle, $E_{g,\text{bulk}}$ is the bulk semiconductor band gap (approximately 3.68 eV for Cubic ZnS [25], approximately 3.9 eV for Wurtzite ZnS [25]), h is Planck's constant ($6.6260 \times 10^{-34} \text{ m}^2 \text{ kg/s}$ or $4.1356 \times 10^{-15} \text{ eV} \cdot \text{s}$), ϵ_r is the relative dielectric constant of the material (8.6 [25]), ϵ_0 is the permittivity of free space, r is the radius of the particle, and m_e and m_h are the effective masses of the electron (approximately $0.3m_e$ for ZnS [27]) and hole (approximately $0.6m_e$ for ZnS [27]), respectively. Using the average crystallite size obtained from X-ray diffractograms, the band gap for each sample is calculated in Table 2. It is clear from Table 2 and Figure 6 that the band gap increases as the crystallite size decreases, and it is found that the sample $\text{ZnS}_{0.1}$ possesses the smallest crystallites and has high absorption as well as higher band gap when compared with other samples, so that the stoichiometry of the $\text{ZnS}_{0.1}$ was selected for further experiments.

Effect of trisodium citrate concentration

Here, trisodium citrate acts as a surfactant, and it allows easy spreading and lowering the surface tension of the solvent. Further, it acts as a microreactor for hosting the reaction and a steric stabilizer to inhibit the aggregation. X-ray diffractogram of the samples was compared with the standard ASTM cards of ZnS and ZnO, and it is found

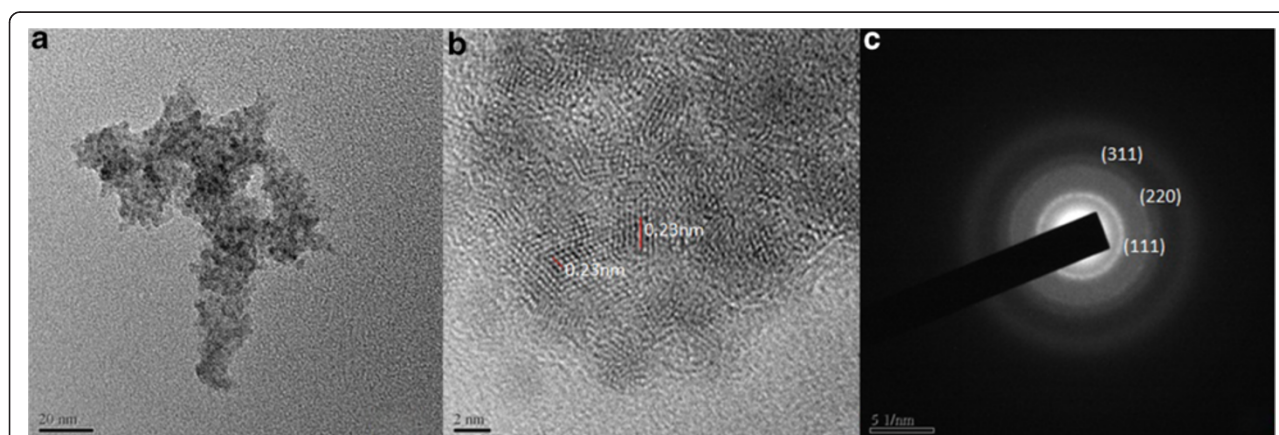


Figure 14 (a) TEM, (b) HRTEM, and (c) SAED images of ZnS prepared in 2 M concentration of citrate (C4).

Table 4 Comparison of interplanar spacing of sample C4 from different analyses

Sample	ASTM 77-2100 (nm)	XRD (nm)	SAED (nm)	HRTEM (nm)	Planes
C4	0.3126	0.3125	0.30	0.23	(111)
	0.1914	0.1894	0.18	-	(220)
	0.1632	0.1628	0.15	-	(311)

that all the diffractograms correlate with the ASTM card of ZnS 77-2100 (cubic). Their X-ray diffractograms are shown in Figure 7, and their broadened peaks reveal that the sample consisted of nano-sized crystallites. Interplanar spacing of the crystals was calculated and compared with ASTM data in the table given with Figures 8, 9, 10, 11, 12, and 13. Average crystallite sizes and strain were obtained from Williamson-Hall plot [22], and other crystal information is given in Table 3. It is seen from Table 3 that the ZnS crystals prepared in trisodium citrate have smaller crystal size when compared to the sample without this surfactant and formed various sizes of crystallites depending on the concentrations of the surfactants. Further, all of the samples show compressive strain, a property of nanomaterials. From all these experiments, ZnS prepared in 2 M of citrate has the lowest size of crystallites which is evident from Table 3. SEM micrographs of the samples shown in Figures 8, 9, 10, 11, 12, and 13 and the images reveal that well-dispersed particles of nanocrystallites have been formed as observed from the X-ray diffractogram. From this analysis, it is obvious that the ZnS particle growth can be controlled by the concentration of the surfactant sodium citrate.

Reported works of John et al. [28] and Yuan-yuan [20] on ZnS nanostructures without any surfactants show larger particles (12 and 35 nm, respectively) when compared to

Table 5 Crystallite size and band gap of the ZnS prepared in 2 M trisodium citrate

Crystallite size (nm)	Band gap from Brus equation (1) (eV)	Band gap from absorption spectra (eV)
1.46	6.82	4.38

the work explained in this paper (1.46 to 2.25 nm). This comparison on particle size with the previous works [20,28] also showed that surfactant sodium citrate has influence on the crystallization of ZnS nanocrystals.

Further, the sample prepared in 2 M of trisodium citrate was analyzed by transmission electron microscopy (TEM) by dispersing the powder in ethanol. Figure 14 shows the TEM, high-resolution transmission electron microscopy (HRTEM), and selected area electron diffraction (SAED) images of this sample, and it is explicitly shown that the particles are well below 10 nm. The polycrystalline diffraction rings of the SAED images are labeled to the corresponding planes according to the ASTM card of ZnS 77-2100 (cubic). The interplanar spacing of 0.23 nm found from HRTEM matches with the (111) planes which is smaller than the XRD data since the powder was dispersed in ethanol for TEM analysis that disintegrate the agglomerated particles and that further compresses the crystallites. The interplanar spacing (d) obtained from different analysis are compared in Table 4.

Optical absorption spectrum of the ZnS prepared in 2 M citrate is shown in Figure 15, and the appearance of the peak shows interband transitions due to the strong quantum confinement effect. The band gap of the sample is calculated and shown in Table 5 with the crystallite size. It is seen that the band gap calculated from the Brus equation is larger than the band gap obtained from the absorption spectra, which shows that the Brus equation cannot be expected to be quantitatively correct for very small particles [29], since the absorption in the case of small particles having size smaller than their exciton Bohr radius is due to the interband transition. Further, the agglomeration of the nanoparticles also has an effect in their absorption spectra.

Fourier transform infrared spectroscopy (FTIR) analysis was performed after dispersing the powder in ethanol to find the presence of trisodium citrate in the final product that helps to differentiate the properties of the pure ZnS nanocrystals with the ZnS nanocrystals with trisodium citrate. The FTIR spectra of the trisodium citrate, ZnS prepared without trisodium citrate, and ZnS prepared with citrate (sample C4) are shown in Figure 16, and the absorption bands are assigned to the respective vibrations as shown in Table 6. The FTIR spectra show that ZnS is covered with the surfactant trisodium citrate.

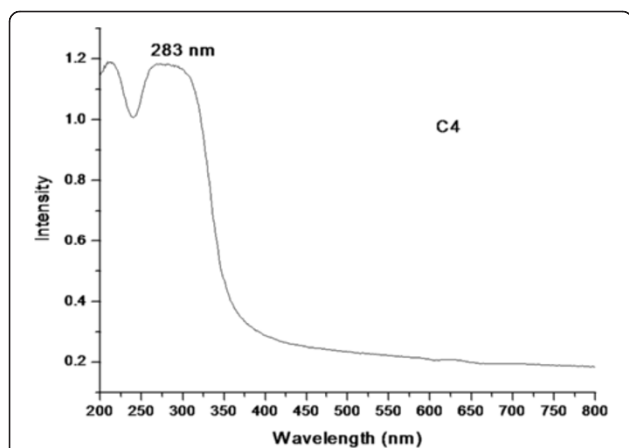


Figure 15 Absorption spectra of the ZnS prepared in 2 M trisodium citrate.

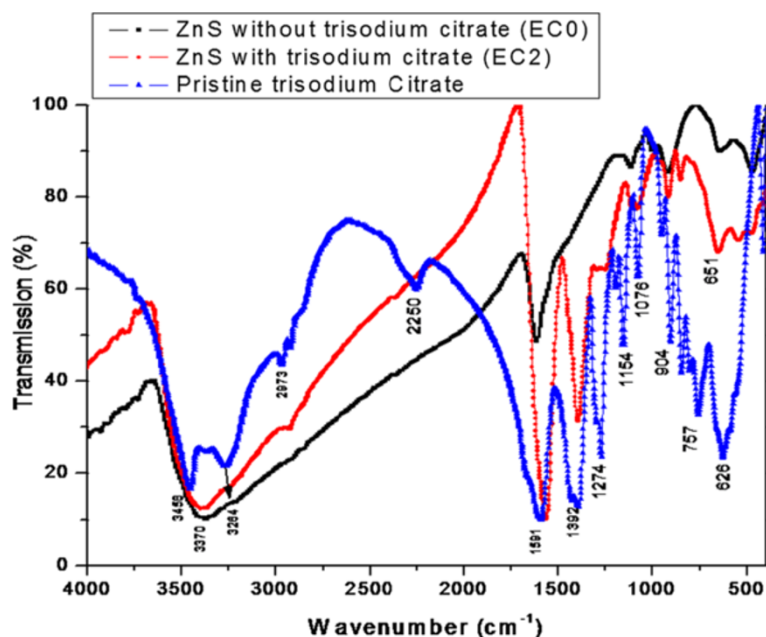


Figure 16 FTIR spectra of Trisodium citrate and ZnS with and without trisodium citrate.

Conclusions

The preparation of zinc sulfide nanocrystals by simple wet chemical precipitation method is discussed here. It is found that the concentration of sulfur and trisodium citrate has tremendous effect on ZnS nanoparticle formation. The particle size was seen to depend on the concentration of trisodium citrate, and the dependence of the optical behavior on Bohr exciton radius of the ZnS nanoparticles was observed.

Methods

ZnS nanocrystalline powder synthesis by varying sulfur concentration

It is essential to find a suitable reagent concentration and stoichiometry for ZnS nanoparticles preparation, and therefore, it was decided to fix the concentration of Zn precursor ($ZnCl_2$) and the surfactant trisodium citrate

and to vary the concentration of the sulfur precursor (Na_2S). These studies revealed the suitable Zn precursor and sulfur precursor concentrations for processing ZnS nanocrystallites.

ZnS samples were prepared by varying the sulfur concentration from 0.05 to 1.5 M, and the samples were named as $ZnS_{0.05}$, $ZnS_{0.1}$, $ZnS_{0.5}$, ZnS_1 , and $ZnS_{1.5}$. For this experiment, 50-ml stock solution of 1 M $ZnCl_2$ and 0.5 M trisodium citrate was prepared in deionized (DI) water and concentration of Na_2S was varied as shown in Table 7. Trisodium citrate solution (0.5 M) was added to each of the Na_2S solution and stirred well for 1 hr at room temperature. Then, each of the 10.5-ml Na_2S trisodium citrate solution was added dropwise using a burette to the 10-ml $ZnCl_2$ solution while continuing the stirring (200 rpm) at room temperature. It was found that the color of the solution changed from a transparent

Table 6 FTIR peak assignments of trisodium citrate and ZnS with and without trisodium citrate

Peaks	Citrate		ZnS without citrate (C0)	Peak assignment	ZnS with citrate (C4)	Peak assignments	
	Peak assignment	Peaks					Peak assignment
3458	O-H stretching	1193	C-O stretching	3370	O-H stretching	3399	O-H stretching
3264	O-H stretching	1154	C-O stretching	1615	C-C	1563	C=O stretching
2973	C-H bending	1076	C-O stretching	1115	C-O stretching	1394	C-H bending
2921	C-H bending	949	C-O stretching	1002	C-O stretching	1293	C-O asymmetrical stretching
2250	C=O stretching	904	C-H bending	920	C-H bending	1077	C-O asymmetrical stretching
1591	C=O stretching	845	C-H bending	642	O-H bending	913	C-H bending
1392	C-H bending	757	C-H bending	-	-	849	C-H bending
1274	C-O stretching	626	C-H deformation	-	-	651	C-H bending

Table 7 Concentration of Na₂S for finding suitable stoichiometry

Samples	Number of moles	Molarity (M)	Color of the product
ZnS _{1.5}	0.015	1.5	light yellow
ZnS ₁	0.01	1	light sandal
ZnS _{0.5}	0.005	0.5	light white
ZnS _{0.1}	0.001	0.1	pure white
ZnS _{0.05}	0.0005	0.05	light brown

solution to milky; then, a white precipitate was formed. The excess water with the dissolved by-product NaCl (ZnCl₂ + Na₂S → ZnS+2NaCl) was decanted, and the precipitate was washed in ethanol and then washed four times with deionized water. Since, ethanol can avoid the new formation of ZnS nanocrystals from undissolved Na₂S and ZnCl₂ (ZnCl₂ partially dissolves in ethanol). After washing four times in DI water, the precipitate was again washed in ethanol and the excess ethanol was decanted, and then the precipitate was dried at 60°C in air since ethanol evaporates at around 50°C. After evaporation of the ethanol, a fine powder of ZnS was obtained and the color of the final powder was seen different for each sample as shown in Table 7.

Effect of trisodium citrate concentration

The effect of the concentrations of trisodium citrate on ZnS particle growth was studied by varying trisodium citrate concentration. Here, ZnCl₂ and Na₂S concentrations were fixed similar to the concentration of the sample ZnS_{0.1} in the 'ZnS nanocrystalline powder synthesis by varying sulfur concentration' section based on the results discussed in the 'Results and discussion' section. Stock solutions of 1 M ZnCl₂ and 0.1 M Na₂S were prepared as in the 'ZnS nanocrystalline powder synthesis by varying sulfur concentration' section, and then the solutions of trisodium citrate for various concentrations were prepared as shown in Table 8.

Each concentration of trisodium citrate solution was made under magnetic stirring for 10 min to make it a clear solution, and then each of the trisodium citrate solution was mixed with 10 ml Na₂S solution. The stirring

Table 8 Variation of citrate concentration for ZnS nanopowder synthesis

Samples	Molarity (M)
C0	0
C1	0.5
C2	1
C3	1.5
C4	2
C5	3

continued for 1 h until the solution became light blue, and then the Na₂S-trisodium citrate solution was added drop by drop using a burette to 10 ml ZnCl₂ solution while maintaining the stirring (about 200 rpm). It was seen that the solution became milky for each drop of Na₂S-trisodium citrate, and after mixing all the Na₂S-trisodium citrate solution, a white precipitate was obtained. Then, stirring was stopped to let the white precipitate settle at the bottom of the flask. The excess liquid was decanted, and then the settled white powder was washed in ethanol and then washed four times using DI water as explained in the 'ZnS nanocrystalline powder synthesis by varying sulfur concentration' section. After washing again with ethanol, the precipitate was taken into a heater which was maintained at about 60°C in air, and then finally, a white fine powder was obtained.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

AKT carried out the experiments, examined the results, and drafted the manuscript. AKAU coordinated the project and discussed the results. Both authors read and approved the final manuscript.

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