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Effect of polyvinyl alcohol concentration on the ZnS nanoparticles and wet chemical synthesis of wurtzite ZnS nanoparticles

Abdul Kareem Thottoli* and Anu Kaliani Achuthanunni

Abstract

ZnS is one of the most important semiconducting materials, and this paper discusses the wet chemical synthesis of ZnS nanoparticles in cubic and hexagonal structures. The effect of polyvinyl alcohol concentration on the ZnS particle formation is studied, and ZnS nanocrystals smaller than the Bohr exciton radius have been prepared using the capping agent polyvinyl alcohol. ZnS nanocrystals of about 1.54 nm have been prepared, and their optical absorption shows interband transitions. It is found that the polyvinyl alcohol concentration has a tremendous effect on the particle growth, since particle sizes were varied with the concentration of polyvinyl alcohol. The important result is that the strain was seen negative due to the contraction of the particles. The important finding of this experiment is that the preparation of the hexagonal ZnS nanoparticles is possible at lower temperature when a particular concentration of trisodium citrate and polyvinyl alcohol is used simultaneously with the ZnS precursors.

Keywords: ZnS, Polyvinyl alcohol, Semiconductor

Background

When the size of a semiconductor becomes less than 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 nanomaterials, ZnS is one of the most important semiconducting materials since it can be used in electronic or optoelectronic applications [2] such as in flat-panel displays [3], white light LEDs [4], electroluminescent devices [5], sensors [6], lasers [7], infrared windows [8], ultraviolet (UV) lasers [9], anti-reflection coating on solar cells [10], and solar cells [11]; for investigating cellular interactions [12]; for both *in vivo* and *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 ZnS, such as a blueshift in the optical absorption spectrum, increased luminescence, and enhanced oscillator strength,

non-linear optical effects, geometrical structure, chemical bonds, ionization potential, mechanical strength, and melting point. Furthermore, ZnS relates to the more popular ZnO in terms of atomic structure and chemical properties. Certain properties of ZnS are unique and advantageous compared to ZnO, that is, ZnS has a larger band gap than ZnO (approximately 3.4 eV [17]), and therefore, it is more suitable for visible-UV light-based devices such as sensors or photodetectors.

It is a well-known fact that bulk ZnS crystal is more stable at room temperature in zinc blende form, which transfers to a wurtzite structure at a higher temperature of about 1,020°C [18], even though nanostructures of ZnS take a wurtzite phase [19,20] at lower and higher temperatures. Further, wurtzite ZnS is much more attractive due to its optical properties than the sphalerite phase, so the low-temperature synthesis of wurtzite ZnS nanoparticles is extremely important. In this paper, through a series of designed experiments, we have synthesized wurtzite ZnS nanoparticles at low temperature by controlling the concentration of the polyvinyl alcohol and trisodium citrate. Since, polyvinyl alcohol and trisodium citrate act as surfactant and capping agent,

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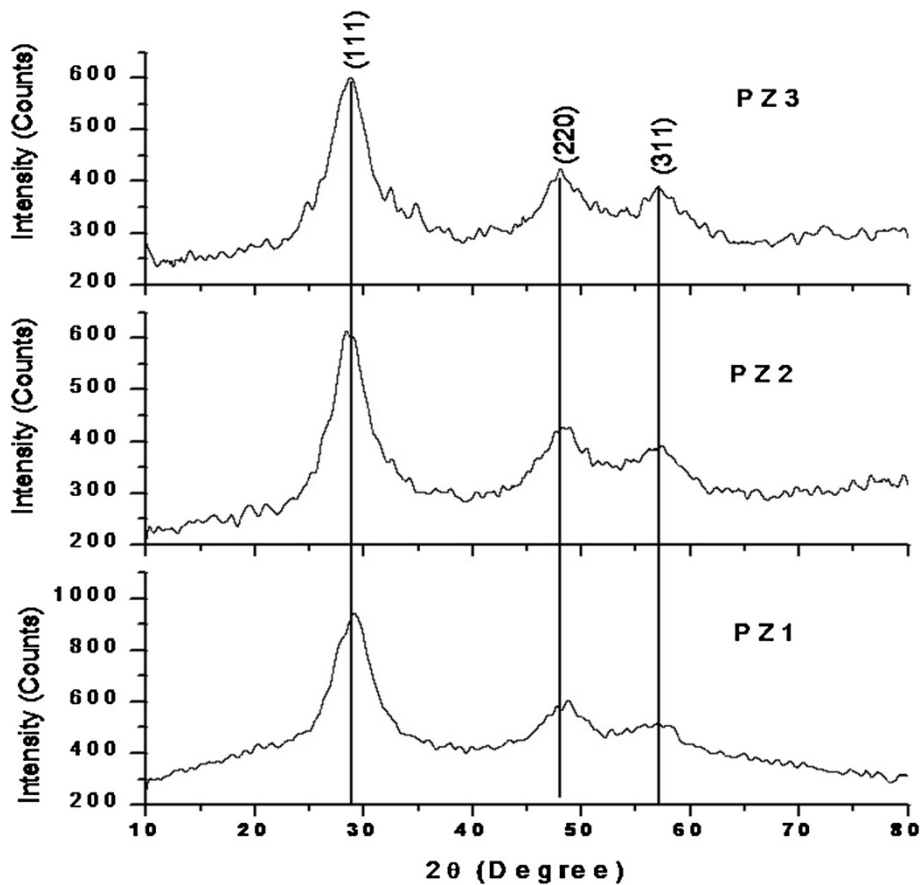


Figure 1 X-ray diffractograms of ZnS nanoparticles prepared in various concentrations of polyvinyl alcohol. PZ1, 0.03 g/ml PVA; PZ2, 0.003 g/ml PVA; PZ3, 0.0003 g/ml PVA.

they can modify the surface of ZnS particles and prevent the growth of the particles.

Results and discussion
ZnS nanocrystalline powder synthesis by varying polyvinyl alcohol concentration

The peak broadening in the X-ray diffraction (XRD) patterns of the samples shown in Figure 1 clearly indicates the formation of the ZnS nanocrystal in the samples [21], and their crystal information are tabled in Figures 2, 3, and 4.

All of the data are well correlated with the American Society for Testing and Materials (ASTM) card no. 77–2100 for cubic ZnS. The increase in the diffraction angle when comparing to the ASTM data (see tables in the Figures 2, 3, and 4) is clearly a result of lattice contraction which is expected to occur because of a higher surface-to-volume ratio [22], and it is also proven from the negative strain tabulated in Table 1 from the Williamson-Hall plot [23]. Their SEM images shown in Figures 2, 3, and 4 clearly show that the sample consists of agglomerated nanocrystalline

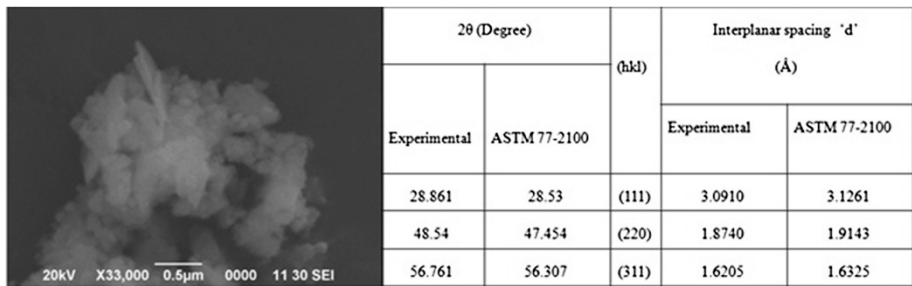


Figure 2 SEM micrograph of ZnS prepared in 0.03 g/ml concentration of PVA (PZ1).

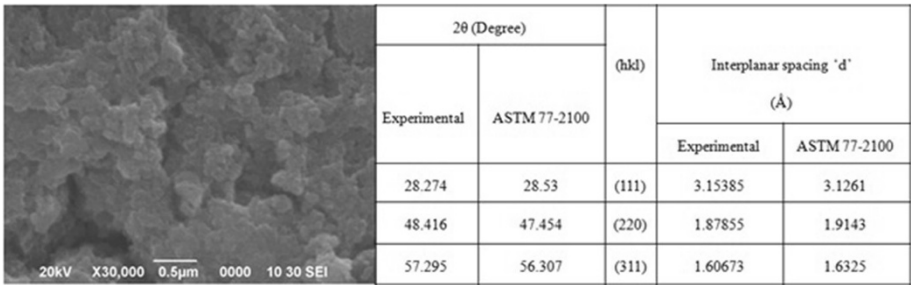


Figure 3 SEM micrograph of ZnS prepared in 0.003 g/ml concentration of PVA (PZ2).

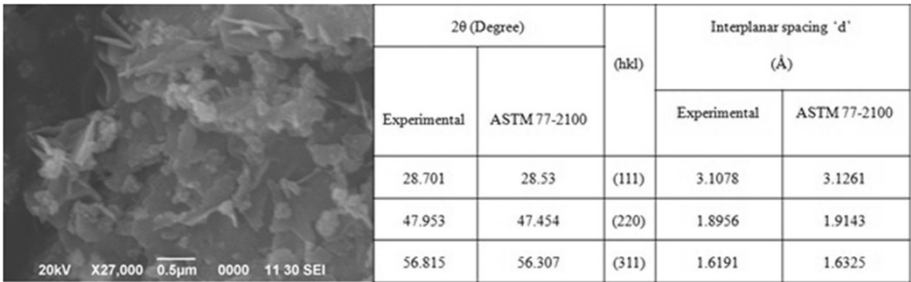


Figure 4 SEM micrograph of ZnS prepared in 0.0003 g/ml concentration of PVA (PZ3).

Table 1 Lattice parameters and volume of crystals

Samples	Lattice parameters (ASTM value in brackets)			Average crystallite size (nm)	Average strain	Volume (10 ⁻²⁸ m ³)
	a (Å)	b (Å)	c (Å)			
PZ1	5.3431 (5.4146)	-	-	1.54	-0.1490	1.5253
PZ2	5.3683 (5.4146)	-	-	2.70	-0.0061	1.5470
PZ3	5.3715 (5.4146)	-	-	2.34	-0.0351	1.5499

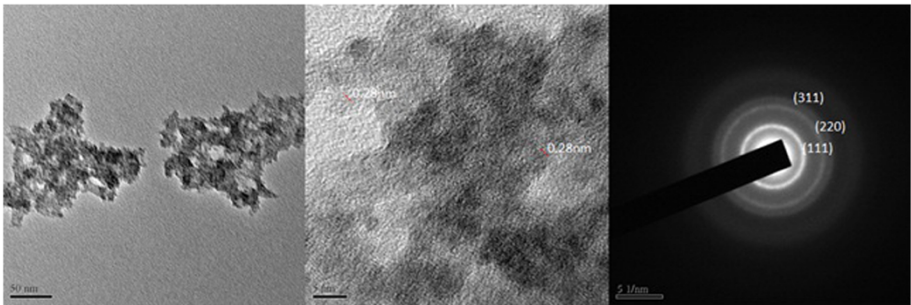


Figure 5 TEM, HRTEM, and SAED images of PZ1 sample.

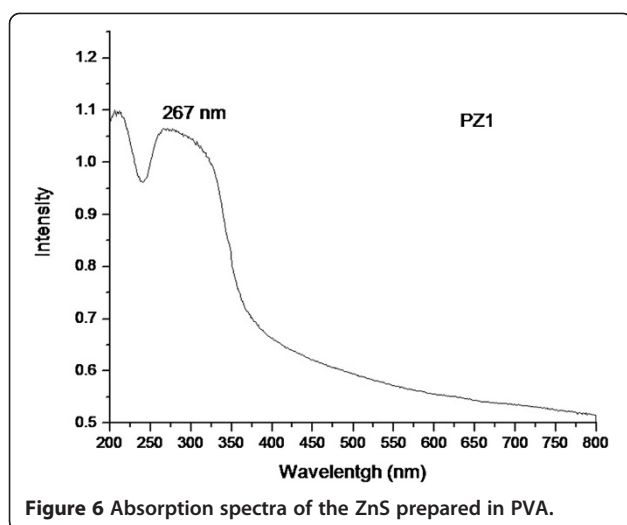


Figure 6 Absorption spectra of the ZnS prepared in PVA.

materials due to the heating of the samples for making powder. The crystallite sizes were found from the Williamson-Hall plot [23] as 1.54, 2.70, 2.34 nm for PZ1, PZ2, and PZ3 samples, respectively, (see Table 1). It is found that the sample made with 0.03 mg/ml PVA possessed smaller crystallites, so this sample was further analyzed by transmission electron microscopy (TEM) by dispersing the samples in ethanol. The TEM, high-resolution TEM (HRTEM), and selected area electron diffraction (SAED) images of the PZ1 sample are shown in Figure 5. It is very clear that the sample consists of nanosized particles, and the interplanar spacing was measured as 0.28 nm for the (111) plane. It was found that it is contracted due to the increased surface area-to-volume ratio when compared to the powder XRD data, and the polycrystalline diffraction rings were labeled to (111), (220), and (311) planes according to the ASTM card no. 77-2100 for cubic ZnS.

The absorption spectrum of the PZ1 sample is shown in Figure 6 with the absorption peak. It is evident from the absorption peak that the interband transitions are happening here due to the strong quantum confinement effect since the crystallite size is smaller than the Bohr exciton radius of ZnS. The band gap of the sample is also calculated using the Brus equation [24] and shown in Table 2 with the crystallite size. It is seen that the band gap calculated from the Brus equation is larger than the band gap obtained from absorption spectra,

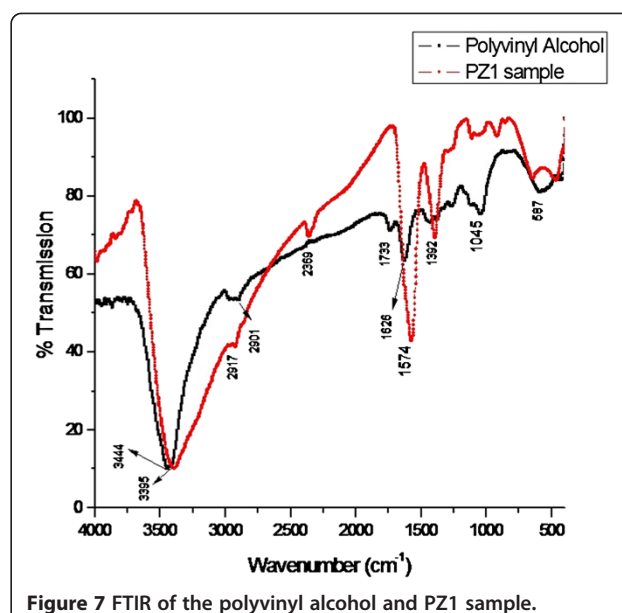


Figure 7 FTIR of the polyvinyl alcohol and PZ1 sample.

which shows that the Brus equation cannot be expected to be quantitatively correct for very small particles [25], since the absorption in the case of small particles having a size smaller than the exciton Bohr radius is due to the interband transition. Further, the agglomeration of the nanoparticles also has an effect in their absorption spectra.

Further, the PZ1 sample was analyzed by FTIR to find the presence of PVA in the final powder sample. Figure 7 shows the infrared spectrum of pure PVA and PVA-ZnS samples between 500 and 4,000 cm^{-1} . The spectrum of PVA seems to be consistent with that previously reported in literatures [26,27]. A relatively broad and intense $\nu(\text{OH})$ absorption stretching band is observed at 3,444 and 3,395 cm^{-1} , indicating the presence of polymeric association of the free hydroxyl groups and bonded OH stretching vibration. The slight shift of the peaks of the PVA-ZnS sample when comparing to the PVA peaks undoubtedly shows that ZnS is well incorporated in the PVA matrix. The broad O-H absorption stretching vibration is observed at around 3,444 cm^{-1} for PVA results from the superposition of multiple polymeric H bonds associated with the crystalline phase and dimeric H bonds associated with the amorphous phase [26]. The absorption bands occurring at 2,901 and 2,917 cm^{-1} resulted from anti-symmetric CH_2

Table 2 Crystallite size and band gap of ZnS prepared with PVA

Crystallite size (nm)	Band gap from the Brus equation ^a (eV)	Band gap from absorption spectra (eV)
1.54	6.45	4.64

^aPlease refer to the 'ZnS nanocrystalline powder synthesis by varying polyvinyl alcohol concentration' section.

Table 3 FTIR peak assignment of polyvinyl alcohol and ZnS prepared in polyvinyl alcohol

Polyvinyl alcohol (cm ⁻¹)	Band assignment	ZnS prepared in polyvinyl alcohol (cm ⁻¹)	Band assignment
3,444	O-H stretching	3,395	O-H stretching
2,901	C-H stretching	2,917	C-H stretching
1,733	C=O stretching	2,369	May be due to S-H interaction
1,626	C=C stretching	1,574	C=C stretching
1,428	Symmetric bending of CH ₂	1,392	C-H bending
1,263	C-H bending	1,114	-C-O-H stretching
1,045	C-O stretching	915	C-C stretching
587	C-H bending	854.23	CH ₂ stretching
-	-	642	OH stretching

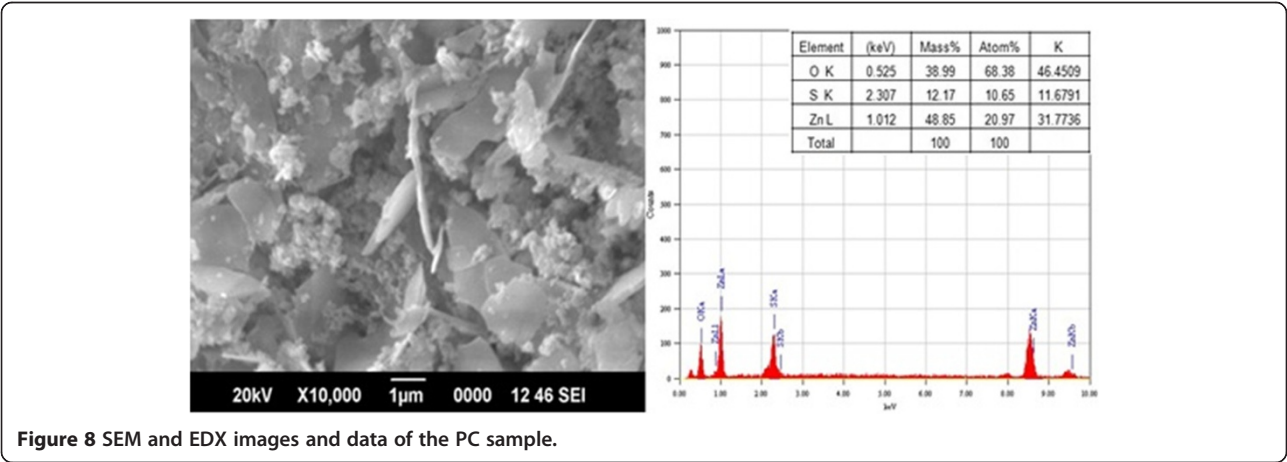


Figure 8 SEM and EDX images and data of the PC sample.

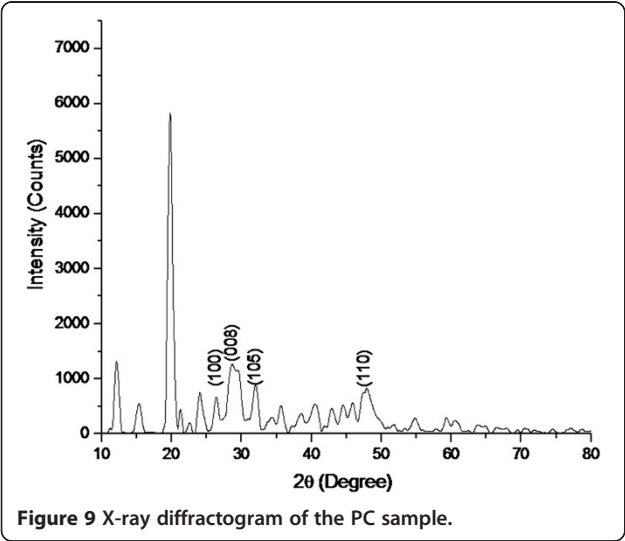


Figure 9 X-ray diffractogram of the PC sample.

stretching and C-H stretching of CH₂ groups, respectively. The bands at 1,733 and 1,626 cm⁻¹ are due to the C=O stretching and C=C stretching. These carbonyl groups are due to the absorption of the residual acetate groups due to the manufacture of PVA from hydrolysis of polyvinyl acetate [26]. The symmetric bending mode of CH₂ is found at 1,428 cm⁻¹. The band at about 1,263 cm⁻¹ results from the vibration of CH. The band at about 1,045 cm⁻¹ is assigned to C-O stretching vibration. The IR band positions and their assignments are presented in Table 3.

Wurtzite ZnS nanoparticles

The SEM image of the prepared white powder is shown in Figure 8 with the energy-dispersive X-ray spectroscopy (EDX) data. The SEM image shows that the sample consists of nanosized particles and nanosheets. The elemental analysis shows that the sample possessed Zn, S, and oxygen. So, the sample was studied using its X-ray diffractogram (Figure 9) to confirm the compound and to find the crystal information. Also, the X-ray diffractogram of the sample

Table 4 XRD data of the PC sample

2θ (°)		(hkl)	Interplanar spacing 'd' (Å)	
Experimental	ASTM 39-1363		Experimental	ASTM 39-1363
26.399	26.914	(100)	3.3734	3.3100
28.577	28.586	(008)	3.1210	3.1201
29.788	29.257		2.9969	3.0500
32.208	32.411	(105)	2.7770	2.7601
35.597	35.597		2.5200	2.5200
46.006	46.865		1.9711	1.9370
47.7	47.727	(110)	1.9050	1.9040

was compared with the ASTM data of ZnO and ZnS due to the presence of oxygen as seen from EDX. It is found that the obtained the X-ray diffractogram correlates well with the ASTM 39-1363 for hexagonal ZnS. The crystals of ZnS were seen aligned along (100), (008), (105), and (110) planes. Further, the peaks for PVA was also seen at 19.72 [28,29] for (101) reflections due to the crystallization of PVA, and other reflection peaks from unknown planes were seen at 2θ , 11.96, 15.36, 21.16, 24.06, 34.48, 35.45, 40.53, 42.7, 44.6, 45.85, 54.81, and 59.4. Crystal data are shown in Tables 4 and 5, and it is found that the data are well correlated with the hexagonal ZnS. The average crystallite size was calculated using the Williamson-Hall plot [23] as 5.70 nm.

The sample was further analyzed by its TEM, HRTEM, and SAED images shown in Figure 10. It is seen that the sample consists of nanosized ZnS particles of about 4.17 nm. The interplanar spacing of the crystals found from the HRTEM is about 0.19 nm, and it may be along the (110) plane. The polycrystalline diffraction rings shown in the SAED images are labeled to the (110), (008), and (105) planes after comparing with the XRD data of the sample.

The absorption spectrum of the polycrystalline PC sample is shown in Figure 11, and the band gap of the sample was found at 4.07 eV from the absorption edge (305 nm) which is blueshifted when compared to the bulk value of 3.9 eV. Absorption peaks are not observed in this sample which may be due to the larger crystallite size than the Bohr exciton radius of ZnS. The band gap of the sample with particle and crystallite sizes is shown in Table 6. It is also seen that the band gap found from the Brus equation [24] is larger than the observed band gap from the absorption spectra, and the reason for the difference is already well explained before.

The FTIR spectra of the ZnS sample prepared with PVA and trisodium citrate are given in Figure 12 that shows C-H, C-O, and O-H stretching vibrations and C-H bending vibrations. All of the other observed peaks are assigned to their respective values as given in Table 7. The C=C bending vibrations are also seen in the spectra, and that may be due to the presence of esters from the manufacture of PVA.

Conclusion

ZnS nanoparticle growth using the concentration of the polymer capping agent polyvinyl alcohol was studied in this paper. ZnS nanocrystals of about 1.54 nm have been prepared, and their optical absorption shows interband transitions. Wurtzite ZnS nanocrystals have been prepared by low-temperature wet chemical method. It is found that the polyvinyl alcohol concentration has a tremendous effect in the particle growth. Particle sizes were varied with the concentration of PVA. The important factor is that the strain was seen negative due to contraction of the particles. The important finding of this experiment is that the preparation of the hexagonal ZnS nanoparticles is possible in lower temperatures when both the trisodium citrate and PVA are used simultaneously with the ZnS precursors.

Methods

ZnS nanocrystalline powder synthesis by varying polyvinyl alcohol concentration

The exact concentration of the ZnS precursor solutions and trisodium citrate for ZnS was found by fixing ZnCl_2 concentration and by varying Na_2S and trisodium concentration, but those experimental results are not explained

Table 5 Crystal information of prepared hexagonal ZnS

Crystallite size (nm)	Average strain	Observed lattice parameters (Å)		Lattice parameters (ASTM card no. 39-1363) (Å)		Volume of the crystal (10^{-28} m^3)	
		a	c	a	c	Observed	ASTM card no. 39-1363
3.2	-0.0668	3.8953	24.9687	3.8221	24.9611	3.2809	3.1578

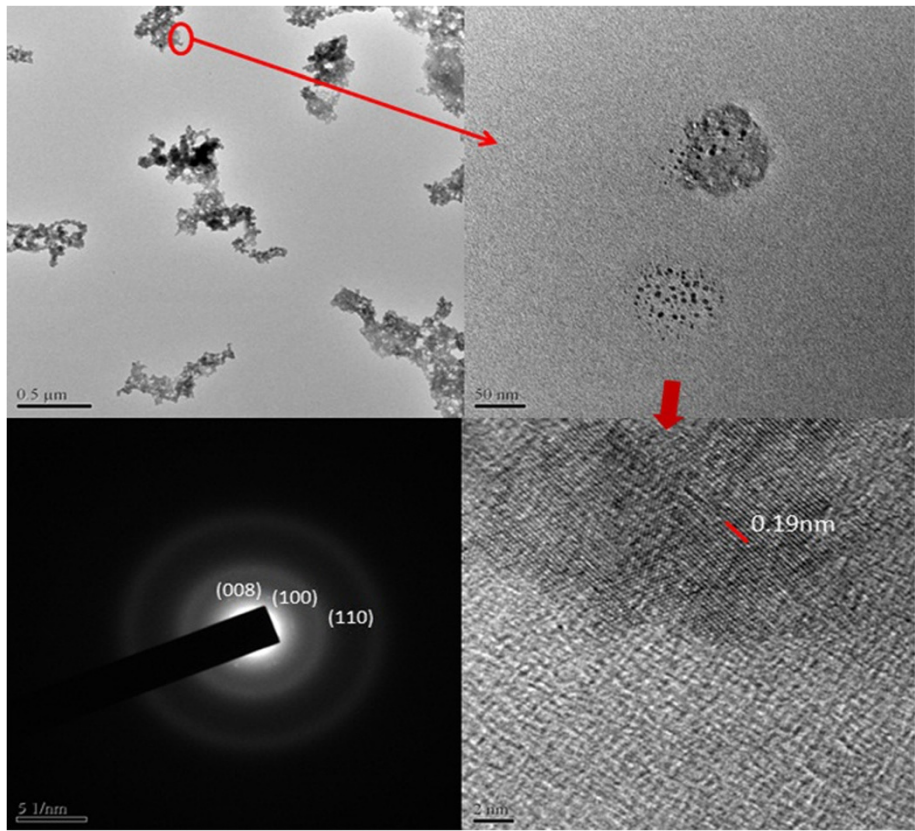


Figure 10 TEM, HRTEM, and SAED images of the PC sample.

here since they do not belong under the scope of this paper. A systematic study was performed to find the influence of polymer polyvinyl alcohol concentration on ZnS particle growth. Polyvinyl alcohol is a water-soluble polymer, and it can be used as a capping and reducing agent in nanoparticles preparation. Here, ZnS powder samples were prepared for various concentrations of polyvinyl

alcohol and compared with the ZnS sample prepared without any surfactant or capping or reducing agent.

Polyvinyl alcohol (PVA) (80% hydrolyzed) was purchased from Sigma-Aldrich Chemicals (Bangalore, India) and used without any further purification. PVA solution of different concentrations (Table 8) was prepared at 60°C under magnetic stirring for 1 h to make it a clear solution. Then, 1 M ZnCl₂ solution was slowly mixed with this hot PVA solution. The stirring continued for another 1 h, and then the 0.1 M Na₂S solution was added drop by drop slowly using a burette while continuing the stirring (about 200 rpm). The formation of white powder for each drop of Na₂S was seen, and it was taken out from the heater after mixing all of the Na₂S solution. When the white precipitate settled at the bottom of the flask, the excess liquid was decanted, and the settled white powder was washed in ethanol then with deionized (DI) water four times, then

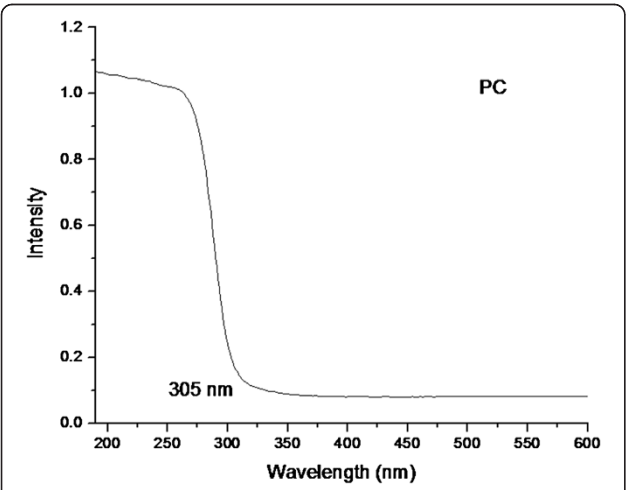


Figure 11 Absorption spectrum of the prepared wurtzite ZnS.

Table 6 Crystallite size, particle size, and band gap of the PC sample

Crystallite size (nm)	Particle size from TEM (nm)	Band gap from the Brus equation ^a (eV)	Band gap from absorption spectrum (eV)
3.2	4.17	4.45	4.07

^aPlease refer to the 'Wurtzite ZnS nanoparticles' section.

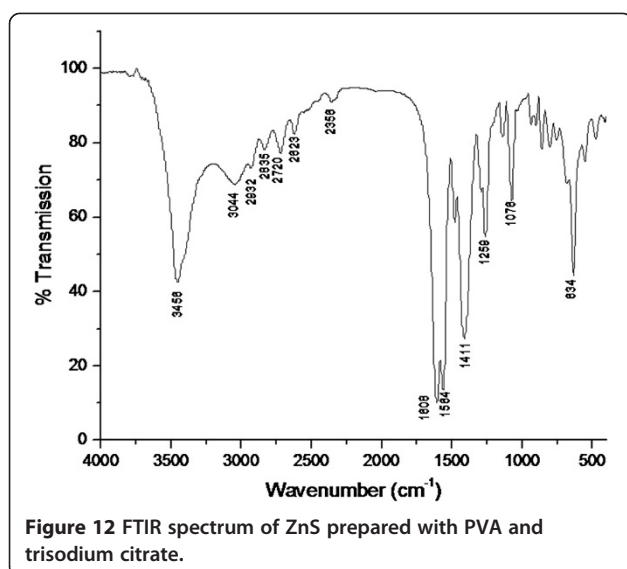


Figure 12 FTIR spectrum of ZnS prepared with PVA and trisodium citrate.

washed again with ethanol. After washing with ethanol, the precipitated white powder was taken into a heater maintained at about 60°C to dry it and to make it into a fine powder. Then, the powder samples were analyzed

Table 7 FTIR peak assignment of ZnS sample prepared with PVA and trisodium citrate

Peak (cm ⁻¹)	Band assignment
3,456	Dimeric O-H stretch
3,044	C-H stretch
2,932	C-H asymmetrical stretching
2,835	C-H stretch
2,720	C-H stretch.
2,623	S-H stretch
2,356	May be due to S-H interaction
1,608	Conjugated C=C
1,564	C=C bending
1,480	C-H bending
1,411	C-H inplane bend
1,291	C-H in plane bend
1,259	O-H in plane bend
1,138	C-H bend
1,076	C-O symmetrical stretch
935	C-H bending
902	C-H bending
859	C-H bending
803	C-H bending
753	C-H bending
681	C-H bending
634	C-H bending

Table 8 Variation of PVA concentration on ZnS nanopowder synthesis

Name of sample	Material	Density (gm/ml)
PZ1	PVA	0.03
PZ2	PVA	0.003
PZ3	PVA	0.0003

using a Bruker AXS D8 Advance powder X-ray diffractometer (Bruker Optik GmbH, Ettlingen, Germany), Thermo Nicolet Avatar 370 Fourier transform infrared spectrometer (FTIR; Thermo Nicolet Corporation, Madison, WI, USA), JEOL model JSM-6390LV scanning electron microscope (JEOL Ltd., Akishima, Tokyo, Japan), JED-2300 energy-dispersive spectrometer (JEOL), and JEM-2100 transmission electron microscope (JEOL).

Wet chemical synthesis of wurtzite ZnS particle

Mutual effect of the PVA and trisodium citrate concentration was studied by adding them together after finding their best concentration from the experiments explained in the above section. So, polyvinyl alcohol solution of 0.03 mg/ml concentration was made at 60°C under magnetic stirring for 1 h to make it a clear solution. Then, 1 M ZnCl₂ solution was slowly mixed with this hot PVA solution while continuing the stirring. Another solution of 0.1 M Na₂S and 2 M trisodium citrate were prepared and mixed together and stirred for 1 h. The Na₂S-trisodium citrate solution was added drop by drop slowly using a burette while continuing the stirring (about 200 rpm). The formation of white powder for each drop of Na₂S-trisodium citrate was seen, and it was taken out from the heater after mixing all of the Na₂S-trisodium citrate solution. When the white precipitate settled at the bottom of the flask, the excess liquid was decanted, and the settled white powder was washed in ethanol, then four times using DI water, and then washed again with ethanol. After washing with ethanol, the precipitated white powder was taken into a heater maintained at about 60°C to dry it and to make it into a fine powder. Then, the powder sample was named as PC and then analyzed using a Bruker AXS D8 Advance powder X-ray diffractometer, Thermo Nicolet Avatar 370 FTIR, JEOL model JSM-6390LV scanning electron microscope, JED-2300 energy-dispersive spectrometer, and JEM-2100 transmission electron microscope.

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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AKT has M.Sc. and M.Phil degrees and is a research scholar at the Department of Physics, Kongunadu Arts and Science College, Coimbatore. He holds a Bachelor's degree in Physics from the University of Calicut, Malappuram, Kerala, India and M. Sc. and M.Phil. in Physics from Bharathiar University, Coimbatore, Tamilnadu, India. Currently, he is doing a Ph.D. degree at the Kongunadu Arts and Science College (affiliated to Bharathiar University), Coimbatore in Physics. He specializes in the plasma electrochemical synthesis of ZnS nanoparticles and wet chemical synthesis of ZnS and Ag nanoparticles for P3HT bulk heterojunction solar cells. He is also the recipient of the Government of Tamilnadu State Collegiate Education Scholarship for Ph.D.; US National Science Foundation I2CAM International Materials Institute Travel Grant for I-CAMP Summer School 2012 held at University of Colorado, Boulder, USA; Centre for International Co-operation in Science (promoted by the Indian National Science Academy), Chennai Travel Fellowship for I-CAMP Summer School 2012 held at University of Colorado, Boulder, USA; US National Science Foundation I2CAM International Materials Institute Travel Grant for I-CAMP Summer School 2009 held in China; and Department of Science and Technology (DST), Government of India Travel Fellowship for I-CAMP Summer School 2009 held in China. He is a life member of the Plasma Science Society of India (PSSI); full ICAM member-JNCASR, Bangalore Branch; member of the International Union of Crystallography (IUCr); regular fellow of the Optical Society of India (F1082); and life member of the Materials Research Society of India. Currently, he is credited for ten papers in international journals, and he has visited the Kingdom of Saudi Arabia, Republic of China, and the United States of America, where he presented his research results.

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