REFLUX CONDENSATION METHOD FOR THE PRODUCTION OF CADMIUM SULPHIDE NANOPARTICLES AND ITS CHARACTERIZATION

[1]Dr.T.Kiran Kumar, [2]P.Samatha, [3]P.Priyanka Reddy,
[1]Professor, [2,3]Assistant Professor, Department of H & S, Brilliant Institute of Engineering and Technology, Hyderabad, India

ABSTRACT
The straightforward and speedy reflux condensation approach has been used to successfully synthesize cadmium sulphide nanoparticles (CdS) with various cadmium (Cd) weight addition ratios. To ascertain the characteristics of the samples, X-Ray Diffraction analysis (XRD) and Field Emission Scanning Electron Microscope (FESEM) are used. According to the XRD pattern, the optimized sample shows a greater hexagonal crystanality. The uniform distribution of particle size and crystanality of cadmium sulphide nanoparticles are revealed by the structural investigation performed using FESEM.
Keywords: Cadmium sulfide; reflux condensation method; XRD; FESEM

1. INTRODUCTION

Due to the synergistic interaction between the components, which is essential for the material's application in numerous fields, nano composites made of two or more components with desirable qualities and performance are currently one of the most investigated topics. A yellow chemical compound called cadmium sulphide functions as an electrical semiconductor. In nature, it can be found as the rare minerals greenockite and hawleyite with two different crystal structures, although it is more common as an impurity substitute in the similarly structured zinc ores sphalerite and wurtzite, which are the main sources of cadmium on a commercial scale. It is simple to extract and purify this substance. By precipitating soluble cadmium (II) salts with the sulphide ion, cadmium sulphide can be made. This reaction has been used for gravimetric analysis and qualitative inorganic analysis. The preparative route and the subsequent treatment of the product, affects the polymorphic form that is produced (i.e., cubic vs. hexagonal). It has been asserted that chemical precipitation methods result in the cubic zinc blende form [1].

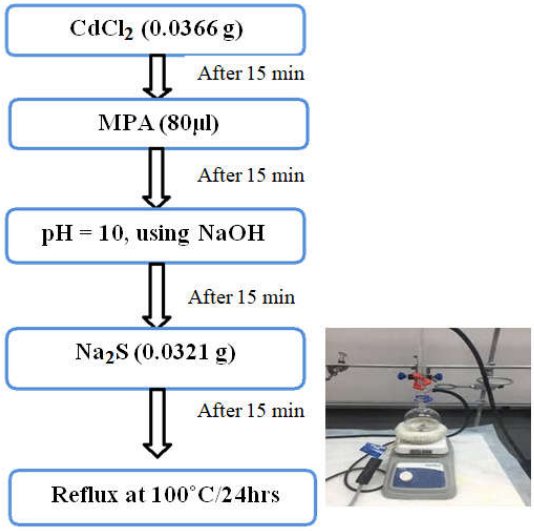
Pigment production usually involves the precipitation of CdS, the washing of the solid precipitate to remove soluble cadmium salts followed by calcination (roasting) to convert it to the hexagonal form followed by milling to produce a powder.[1]When cadmium sulfide selenides are required the CdS is co-precipitated with CdS and the cadmium sulfoselenide is created during the calcination step. Cadmium sulfide is sometimes associated with sulfate reducing bacteria. Cadmium sulfide is also used in the production of solar cells where it is used as a buffer layer in the manufacture of CIGS (Copper -Indium-Gallium-Selenite) solar cells. With an increasing interest and uptake of solar cells, this application for cadmium sulfide could also increase. And also used as light dependent resistors/photo resistors for light sensors and Screen printing using slurry containing dispersed CdS. While the main use for Cadmium sulfide has been as pigment, it is reasonable to expect that the solar cell market will begin to consume more Cadmium Sulfide in the future as the technology becomes more widely accepted and more affordable [2].

2. Materials and Methods

2.1 Preparation of CdS nanoparticles (CdS)

Cadmium sulfide nanoparticles are synthesized using a simple and inexpensive reflux condensation technique. For the synthesis of CdS nanoparticles, $CdCl_2$ (0.0366 g) was dissolved in double distilled water, secondly

Mercaptopropionic acid MPA (80µl) was added to the above mixture. Then the solution pH was adjusted to 10 using NaOH. Finally Na_2S was mixed to the above mixture. Then above mixture was transferred into reflux at 100˚C for different hours. Then the resultant product was collected and centrifuged, then dried at 60˚C. Similarly by varying amount of $CdCl_2$ (0.003M & 0.005M) for different hours the product was collected for further study.



3. Results and Discussion 3.1 XRD study

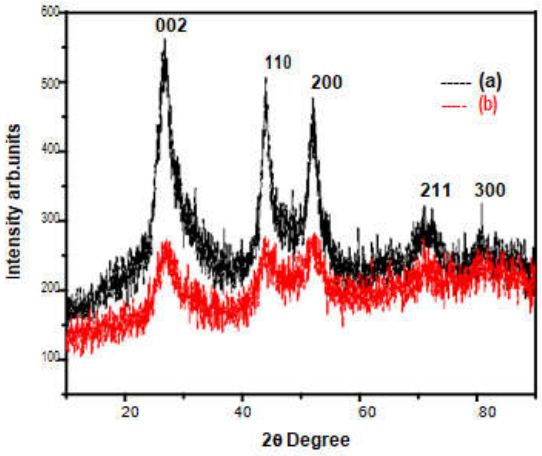


Figure 1. XRD patterns for CdS nanoparticles with different mole concentration (a) Cd (0.003 M)/ S (0.004 M), (b) Cd (0.005 M)/S (0.004 M).

The XRD spectra of Cadmium Sulphide (CdS) nanoparticles are as shown in Figure (1). XRD studies reveal that the samples are nano sized and crystalline. The fine particle nature of the samples are reflected in the X-ray line broadening. It shows the five major broaden peaks corresponds to (002), (110), (200), (211) and (300) planes respectively. The peaks are directly well indexed with the JCPDS data (Card No: 80-0006), which confirms the hexagonal crystal structure of CdS and also proves the existence of Cd and S ions present in the as-prepared samples [3,4]. No other peaks are identified in the XRD plot, indicating the phase purity of the as prepared sample as evident from the high intensity of the diffraction peaks. From the results the Grain size, Dislocation density and Lattice constants of CdS nanoparticles are calculated using Scherrer equation and summarized in

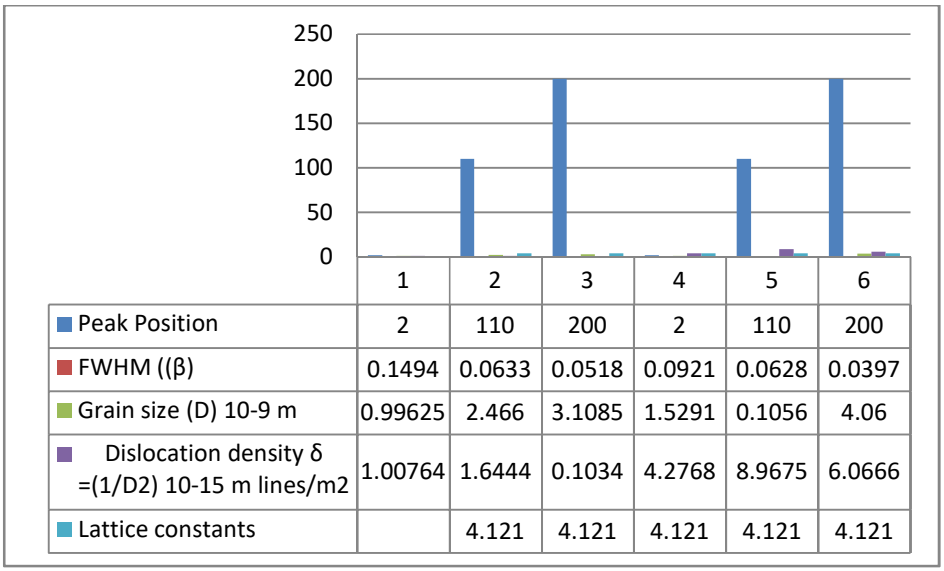
Table (1) as given below. The lattice parameters for hexagonal form may shows the results as a = 4.121 Å and c = 6.682 Å

Scherrer Equation $D = 0.9 \lambda / \beta \cos \Theta$

Where λ represents wavelength of X-rays, β represents half width at full maximum and Θ is the diffraction angle [5-7].

The dislocation density is defined as the length of dislocation lines per unit volume of the crystal. In material science, a dislocation is a crystallographic defect or irregularity within a crystal structure. The presence of dislocations strongly influences many of the properties of materials. Thus, a larger dislocation density implies a larger hardness.

Dislocation density $\delta = 1 / D^2$



	1	2	3	4	5	6
Peak Position	2	110	200	2	110	200
FWHM ((β)	0.1494	0.0633	0.0518	0.0921	0.0628	0.0397
Grain size (D) 10-9 m	0.99625	2.466	3.1085	1.5291	0.1056	4.06
Dislocation density δ =(1/D2) 10-15 m lines/m2	1.00764	1.6444	0.1034	4.2768	8.9675	6.0666
Lattice constants		4.121	4.121	4.121	4.121	4.121

3.2 FESEM image

The surface morphology and structure characteristics of as-prepared CdS nanoparticles with different precursor concentrations are analyzed through FESEM analysis as shown in Figure (2) [8-10]. The CdS nanoparticles prepared with low mole concentration [Cd (0.003 M)/S (0.004 M)] were small sized and less agglomerated compared with those prepared with high mole concentration [Cd (0.005 M)/S (0.004 M].

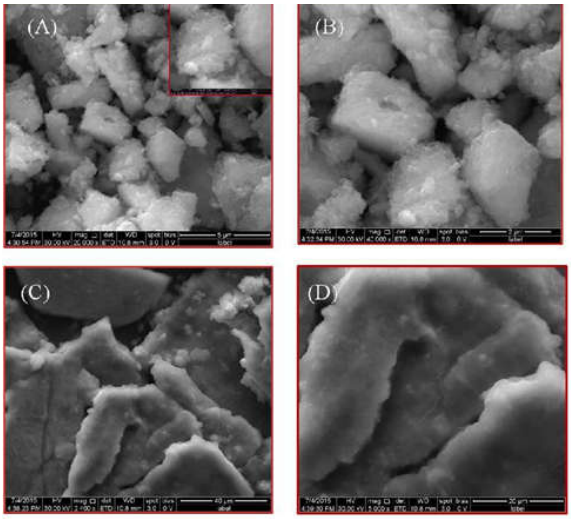


Fig 2. FESEM images for CdS nanoparticles with different mole concentrations (a), (b) Cd (0.003 M)/ S (0.004 M), (c) and (d) Cd (0.005 M)/S (0.004 M).

Conclusion

In conclusion, we have created CdS nanoparticles using a straightforward chemical process (reflux condensation technique). By employing XRD, the crystal structure and grain size of the nanoparticles are determined. This verifies that the nanoparticles have hexagonal crystal structures. FESEM analysis was also used to determine the surface morphology, which revealed that low mole concentration CdS nanoparticles [Cd (0.003 M)/S (0.004 M)] were smaller and less agglomerated than high mole concentration Cd (0.005 M)/S (0.004 M) nanoparticles.

References

1. 'Synthesis and characterization of CdS nanoparticles' by R. Bhattacharya et.al. J. Matter Sci (2011) 22:1761-1765.
2. 'Synthesis and optical properties of water soluble CdS nanoparticles for biological applications' by Viet Ha Chu et.al. Adv. Nat. Sci. (2012) 02517.
3. 'Novel method for synthesis of CdS nanoparticles' by N. Ghows et.al. ultrasonics sonochemistry 18 (2011) 269-275.
4. 'Photo conductive Characteristics of Single Crystal CdSNanoribbons' by Jie, J, S.Zhang, et.al. Nano Letters 2006, 6, 1887-1892.
5. 'Enhancement-Mode Metal Semiconductor Field Effect Transistors Basedon Single CdSnanowires', by Ma.R.M. et.al, Applied Physics Letters 2007,90,093109-093109-3.
6. 'Growth of CdS Nanoparticles byChemical Method and its Characterization' by Bhattacharya, R.; Saha,S.et.al., Pramana-Journal ofPhysics 2008, 71, 187-192.
7. 'Surface Properties of CdS Nanoparticles'. By Favero, P. P.et.al., BrazilianJournal of Physics 2006,36,1032-1034.
8. 'Biologically Synthesized Fluorescent CdSNPs Encapsulated by PHB', byPandian,S.et.al.,Enzyme and Microbial Technology 2011,48,319-325.
9. 'Synthesis and Structural properties ofWurtzite TypeCdS Nanoparticle', by Singh,V,; Chauhan, P,er.al., Chalcogenide Letters 2009,6,421-426.
10. 'Application of Pt /CdS forthe Photo catalytic Flue Gas Desulfurization' by Song X et.al., International