Influence of CTAB surfactant on structural and optical properties of CuS and CdS nanoparticles by hydrothermal route

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ABSTRACT
Metal sulphide CuS and CdS nanoparticles capped with Cetyltrimethylammonium bromide (CTAB) were synthesized by hydrothermal method. Structural, morphological, chemical composition, optical and luminescent properties were evaluated by different analytical techniques. X-ray diffraction (XRD) analysis of the CTAB capped metal sulfide nanoparticles reveals the formation of hexagonal structure. High-resolution transmission electron microscopy (HRTEM) images show that the morphology of the capped copper sulphide samples consists of hexagonal structure and capped cadmium has spherical shape and also confirms the crystalline nature of the particles with distinct lattice fringes. In FTIR spectroscopy, the composition of the CTAB capped CuS and CdS nanoparticles have been confirmed. The analysis of photoluminescence (PL) and optical transition show a red shift due to the reduction of band gap energy and it is attributed to the low defects and high crystallinity. The optical studies indicate that CuS and CdS nanoparticles with CTAB can be suitable for optoelectronic devices and photovoltaic applications.

KEYWORDS
Nanoparticles; CTAB; X-ray diffraction; Optical properties and hydrothermal method

1. Introduction
Semiconductor materials have been widely investigated due to their distinctive properties [1,2]. Recent years have witnessed a greater interest in the area of metal sulphide semiconductors due to their interesting optical, electronic, luminescent, mechanical, quantum size effect and other important physical and chemical properties [3]. The synthesis of binary metal sulphide nanoparticles has been focused on II-VI [4] and I-VI [5] compounds, such as chalcogenides of cadmium and copper, as well as ZnS, HgS and MnS, on which a considerable degree of control upon their size and shape, and hence tunability of their optical and electronic properties has been achieved [6,7]. These materials exhibit interesting properties for modern industrial applications. Among these materials, CdS and CuS are typical direct band gap semiconductors having a band gap of 2.4 eV and 2.5 eV respectively for the hexagonal wurtzite phase at room temperature. Specifically, these materials
are excellent photocatalytic materials, sensitive to visible light and are widely used in field effect transistors, light-emitting diodes, electronics, photodiodes, solar cells, sensors, optical materials, various luminescence devices and so on [4,8]. It is found that the optical and optoelectronic properties of CdS and CuS nanoparticles largely depend on their size and morphology.

Herein, we report the CdS and CuS nanoparticles synthesized in aqueous media with cationic surfactant CTAB as passivating agent. The purpose of this paper is to explore the optical and PL properties of CdS and CuS nanoparticles during nucleation and growth in the presence of cationic surfactant that would be useful in understanding the origin of these properties. The reasons for this choice are as follows: (a) the surface of large CdS and CuS particles are negatively charged over a broad range of conditions and even down to quite low pH values. (b) Mehta and Sanjay [9] reported that the surfactants with charges on their head group to behave as lewis acid or base towards the ZnS nanoparticles surface. CTAB plays an important role as a stabilizer in modification and creation of surface recombination centers which can significantly change the optical properties of metal sulphide nanoparticles [10].(c) An important aspect of micellar medium is the ability to control the growth rate and prevents the self-aggregation of nanoparticles by capping the surface charge. However, 1) ligand control is not only on growth rate, but also the nucleation rate which is equally critical to final particle size [11]. 2) The surface charging is not the only factor on the optical properties [12]. A plethora of techniques including hydrothermal synthesis, chemical vapor deposition, electrochemical synthesis and solvothermal reaction have been exploited to prepare CdS and CuS in diverse appearances for various applications [5,8], among which the hydrothermal method is considered to be efficient, because it is demonstrated to be effective for controlling the shape and size of nanoparticles [10]. The in-house prepared nanoparticles were characterized by X-ray diffraction (XRD) analysis, Transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR), UV-visible spectroscopy and Photoluminescence spectroscopy.

2. Materials and methods

2.1 Materials

Cadmium acetate \([\text{Cd(CH}_3\text{COO)}_2\cdot\text{H}_2\text{O}]\), Copper acetate \([\text{Cu(CH}_3\text{COO)}_2\cdot\text{H}_2\text{O}]\), Sodium hydroxide (NaOH), Cetyltrimethylammonium bromide (CTAB) were purchased from Loba (India). Thiourea (CH4N2S) and ethanol were obtained from Merck (India). The chemicals were of analytical grade and were utilized without additional purification. Deionized water was used as a solvent for all the experimental process. The glasswares were properly washed and sanitized.

2.2 Synthesis of CTAB capped CdS and CuS nanoparticles

In a typical preparation process, Cadmium acetate, Copper acetate and Thiourea were used as precursors and NaOH as the mineralizer for the hydrothermal synthesis of CdS or CuS nanoparticles. The process began with dissolving Cadmium acetate or Copper acetate (0.2 M) and CH4N2S (0.5 M) into 20 mL of double distilled water separately to form two transparent solutions. There after 20 mL NaOH (1 M) solution was added little by
little facilitated by magnetic stirring into the Cadmium acetate or Copper acetate transparent solution to maintain the pH around 8. Thiourea solution was added drop by drop into the above mixed solution under vigorous stirring using magnetic stirrer at room temperature and then CTAB (0.3 g) was added to this. Finally, the resulting solution was transferred into a 100 mL Teflon-lined stainless autoclave with a fill factor of approximately 70%. The autoclave was sealed and maintained in a furnace at 180 °C for 12 h. The autoclave was cooled naturally to room temperature. Thus, the uncapped yellow (greenish black) precipitate of CdS (CuS) was obtained and it was washed several times in double distilled water and ethanol by centrifugation and ultrasonication. Finally, the product was annealed in a furnace maintained at 150 °C for 10 h and used for further characterization. The annealing temperature was chosen considering the thermal steadiness of the compounds. The schematic diagram of the synthesis process is shown in Figure 1.

2.3 Characterization

The structural characterization of the samples were recorded by powder X-ray diffraction patterns (XRD) using X-ray Diffractometer (X’ Pert PAN Analytical) with CuKα radiation (\(\lambda = 1.5406 \text{ Å}\)) in 2θ ranging between 20° and 80°. The morphology and size were determined by the high-resolution transmission electron microscopy (HRTEM) and selected area electron diffraction (SAED) images were obtained on a microscope (JEOL, JEM-2100). TEM specimens were prepared by placing a few drops of liquid sample on a small copper grid and the sample was air dried at room temperature. The dried samples are investigated in the electron microscope. TEM images were operated at 200 kV acceleration voltages. FTIR spectra were recorded with KBr pellets on a Bruker Vector 22 FTIR spectrometer to identify the functional groups in the samples over the range of wave number 400–4000 cm\(^{-1}\). The UV-visible Diffuse Reflectance Spectral (UV-Vis/DRS) analyses
were measured in the range of 200–1200 nm on a (JASCO-V-670) UV-visible Spectrophotometer. The photoluminescence (PL) measurements were performed on a LS 45 fluorescence spectrometer equipped with Xenon lamp as the excitation source.

3. Results and discussions

3.1 X-ray diffraction analysis

To ascertain the crystalline nature of CTAB capped CuS and CdS nanocrystalline powders were explored by X-ray diffraction patterns shown in Figures 2 and 3. The diffraction peaks were found to be sharp and intense which implies the high crystallinity of the samples. The pronounced diffraction peaks indexed can be assigned to the hexagonal wurtzite phase of CdS (JCPDs card no: 75–1545) [11] and CuS (JCPDs card no: 06–0464) [8] respectively. In Figure 2, it is worth noting that the intensities of (103) and (006) diffraction peaks are lower than those of the standard patterns and however, the intensities of (110) diffraction peak in the XRD pattern was particularly strong, indicating the presence of preferential orientation in the sample [13]. In Figure 3, it can be noted that the (002) diffraction peak is comparatively strong and narrow, which can be ascribed to the preferential crystal growth orientation along the c-axis [14].

The crystallite size (Φ) was estimated from the highest intense peak (110) for CuS and (002) for CdS using Sherrer Equation (1):

$$\phi = \frac{K\lambda}{\beta \cos\theta}$$

Where, k is the proportionality constant (k = 0.89), λ is the wavelength of the Cu kα X-rays (1.5405 Å), β_{hkl} is the full width at half maximum (FWHM) of the diffraction peak in.
radians and \( \theta \) is the diffraction angle in degrees [15]. According to the calculation, the crystallite size of CTAB capped CuS and CdS was found to be 32 and 12 nm respectively. The lattice parameters \( a \) and \( c \), tabulated in Table 1, were calculated from the interplanar spacing of different (hkl) planes using the following Equation (2) [15].

\[
d_{hkl} = \frac{1}{\sqrt{\frac{4(h^2+k^2+hl)}{3a^2} + \frac{e^2}{c^2}}}
\]

The volume (V) of the unit cell for the hexagonal system was calculated using Equation (3) [15].

\[
V = \frac{3\sqrt{3}}{2} a^2 c \text{ for hexagonal structure}
\]

In order to understand the structural properties of the sample, crystallite size and strain calculations are required. The crystallite size and lattice strain are the two factors that contribute to the peak broadening. The peak broadening can be caused from defects like

![Figure 3. X-ray diffraction pattern of as prepared CTAB capped CdS nanoparticles.](image)

| Samples            | Crystallite Size (\( \Phi \)) (nm) | Lattice parameter \( a = b \) (Å) | \( c \) (Å) | Volume \( V \) (Å\(^3\)) | strain \( e \times 10^{-4} \) | Sherrer analysis (nm) | Williamson-Hall analysis (nm) | HRTEM analysis (nm) |
|--------------------|----------------------------------|---------------------------------|-----------|-----------------|----------------|-----------------|--------------------------|-----------------|-----------------|
| CTAB capped CuS    | 34 (110)                         | 3.626                           | 18.239    | 623             | 0.298          | 32              | 31.12                    | 34              |
| CTAB capped CdS    | 16 (002)                         | 4.221                           | 7.088     | 328             | 0.502          | 12              | 11.89                    | 18              |
residual stress, grain size, distortion and twinning present in the material [16]. The crystallite size and lattice strain were estimated by XPowder software according to Williamson-Hall (W-H) equation given by the following Equation (4) [16]

$$
\beta \cos \theta = \frac{K \lambda}{\varphi} + 4 \varepsilon \sin \theta
$$

where $\beta$ is full-width at half-maxima (FWHM) in radians, $\theta$ is the diffraction angle, $K$ is the shape factor ($K = 0.9$), $\lambda$ is the wavelength of X-rays, $\Phi$ is the crystallite size and $\varepsilon$ is strain. Figure 4 a and b depicts the Williamson-Hall plot for CTAB capped CuS and CdS nanoparticles.

**Figure 4.** (a) The Williamson-Hall Plot of CTAB capped CuS nanoparticles. (b) The Williamson-Hall Plot of CTAB capped CdS nanoparticles.
nanoparticles with the hexagonal phase respectively. The results of the W-H analysis for the samples give values of \( \Phi \) and \( \varepsilon \) as 31.12, 0.298, 11.89 and 0.502 nm for the CTAB capped CuS and CdS nanoparticles, respectively. The scattered points corresponding to each crystallographic plane in the W-H plot can be attributed to the anisotropy of the dislocation strain field in the elastic medium, as well as to the contribution of planar defects. The average crystallite size obtained from the Williamson-Hall analysis is in agreement with values obtained from Scherrer analysis and HRTEM analysis are listed in Table 1.

### 3.2 Morphological characterization

Figure 5 shows the HRTEM micrographs of the hexagonal copper sulfide nanoparticles, which suggest that the hydrothermal synthesis is a potential method, with the advantages of large-scale production and good uniformity of the particles obtained. The statistical information of the mean particle size of nanoparticles was determined to be 34 nm shown in Figure 5b. In Figure 5c the measured lattice spacing of 1.960 Å, is consistent with the

![Figure 5](image)

**Figure 5.** (a) HRTEM micrograph of CTAB capped CuS hexagonal nanocrystal, (b) size distribution of the nanoparticles, (c) high resolution electron micrograph and (d) SAED pattern of as prepared nanoparticle.
spacing between [110] planes of CuS nanoparticles. Figure 5d presents the selected area electron diffraction [SAED] pattern and the ordered hexagonal–like spot arrays confirmed the formation of hexagonal structures of nanoparticles. On the other hand, CTAB played an important role in the formation of hexagonal CuS nanoparticles. Figure 6 shows the HRTEM images of CTAB capped CdS nanoparticles formed as spherical crystallites. By statistical means on the HRTEM image, Figure 6b reveals the CTAB capped CdS nanoparticles have a mean particle size of 18 nm. The nanoparticles were clearly well identified and no effective aggregations of bulk particles were found, which indicates the effective capping of CTAB on the nanoparticle surfaces. Lattice fringe pattern and size of CdS nanocrystallites are clearly revealed in Figure 6c. The SAED pattern confirms that the synthesized particles are crystalline in nature. There is another reason to have extra weak diffraction spots in SAED patterns. Some systematically absent diffraction spots appear, which do not change the unit cell, but seem to reduce the symmetry of the structure.

Figure 6. (a) HRTEM micrograph of CdS nanoparticles, (b) particle size distribution of nanoparticles, (c) HRTEM image showing lattice fringes and (d) corresponding SAED pattern.
Possible multiple scattering effect should be considered. If this is the case, these extra diffration peaks should not be observed in the corresponding XRD pattern, since no multiple scattering takes place in XRD. When the same SAED pattern is recorded at several areas with different specimen thickness, the relative intensities of these extra spots would be significantly changed. Specimen tilting would also notably change the relative intensities of the extra spots.

### 3.3 FTIR Spectroscopy

Figure 7 represents the IR spectra of CdS and CuS nanoparticles in the range of 4000–400 cm\(^{-1}\). For CTAB capped CuS nanoparticle, the peak at 1605 cm\(^{-1}\) corresponds to the H-O-H bending of absorbed water, which indicates the existing water molecules are absorbed by sulfur products [17]. The absorption band occurring at 1101 cm\(^{-1}\) is due to asymmetric stretching of carbonyl group (C = O). A vibration peak at 597 cm\(^{-1}\) signifies the presence of Cu-S bond [18,19]. For CTAB capped CdS nanoparticle, the peak at 3386.93 cm\(^{-1}\) is attributed to O-H stretching vibration of water molecules [20]. The CH\(_2\) symmetric (\(v_{\text{sym}}(-\text{CH}_2)\)) and asymmetric stretching (\(v_{\text{asym}}(-\text{CH}_2)\)) vibrations of CTAB bound on CdS nanoparticles are observed at 2917 and 2848 cm\(^{-1}\) respectively [21]. The peaks emerged at 1466.36 cm\(^{-1}\) and 1640.15 cm\(^{-1}\) corresponds to the C-C stretching vibrations and the stretching vibrations of sulphate group, traces of SO\(_4^{2-}\) ions respectively [18]. The peak at 1016.43 cm\(^{-1}\) is associated to the C = O stretching vibration. The band located at 717.90 cm\(^{-1}\) is correlated to the stretching frequency of Cd-S bond [22].

### 3.4 UV diffused reflectance spectral analysis

Optical characterization was examined by the UV-Vis diffuse reflectance spectrum (Figures 8 and 9) and the optical band gap energy is determined by using
Kubelka—Munk function $F(R)$ based on the following Equation (5) [23].

$$F(R) = \frac{(1 - R)^2}{2R} \tag{5}$$

where $R$ is the function of reflectance and $F(R)$ is proportional to the extinction coefficient ($\alpha$). The Kubelka—Munk function can be obtained by multiplying the $F(R)$ function by $h\nu$ using the corresponding coefficient (n) associated with an electronic transition.

Figure 8. UV-Vis diffuse reflectance spectra of CTAB capped CdS NPs and the inset, shows the The Kubelka Munk plot for CTAB capped CdS NPs.

Figure 9. UV-Vis diffuse reflectance spectra of CTAB capped CuS NPs and the inset, shows the The Kubelka Munk plot for CTAB capped CuS NPs.
as follows [24]:

\[(h\nu F(R))^n = A(h\nu - E_g)\]  \hspace{1cm} (6)

Where, \(h\) is the Planck’s constant, \(\nu\) is the frequency of vibration, \(E_g\) is the Energy gap, \(A\) is the proportionality constant and \(F(R)\) is the function of Reflectance and \(n = 1/2\) for a direct allowed band gap transition. In Figures. 8 and 9 the inset shows the Kubelka – Munk plot for the calculation of \(E_g\) by plotting \((F(R)h\nu)^2\) versus \(h\nu\), such a representation is known as the Tauc Method. The value of \(E_{phot\,\,extrapolated}\) to \(F(R)E = 0\), which gives an absorption energy, corresponds to a band gap \(E_g\). The band gaps of the CTAB capped CdS and CuS samples are approximately equal and are estimated to be 2.35 and 1.8 eV respectively, which is lower than that of the standard bulk CdS and CuS materials. It can be seen that by capping CTAB in CdS and CuS, the band gap energy has decreased, which was similar to the earlier reports [25,26]. The red shifts can be attributed to the charge transfer transitions between the conduction or valence band of CdS and CuS nanoparticles. Thus it is speculated that this sample may have the highest visible-light driven photocatalytic activity and solar cell application.

3.5 Photoluminescence spectroscopy

The optical spectra of CdS and CuS nanoparticles are portrayed in Figure 10a and b. Figure 10(a) shows the emission spectra of CTAB capped CuS nanoparticles excited at 518 nm. The sharp intense emission peak is located in 657 nm, corresponding to the red emission. The red emission band may be ascribed to the recombination of trapped electron-hole pair, similar behavior was observed by Sharma et al [27] and Deori et al [28]. It is observed that the narrow emission peaks shows better electronic passivation of the CuS nanoparticles by capping CTAB and the reduced broadness of the emission curve may imply a narrow size distribution. The absorption spectrum of Figure 10(b) shows an excitation peak at 493 nm and the photoluminescence spectrum shows a broad emission peak
at 605 nm in red region is observed due to the defects consist of cadmium interstitials or sulfur vacancies in CdS nanoparticles [29,30]. U. Reschet al [31] and Y. Linet al [32] suggested that the sharp emission band is attributed to the radiative recombination of free charge carriers at Cd atom with trapped holes at the sulphur vacancies. The cationic surfactant acting as Lewis acids and depending on their structure, it can stabilize the surface states of the CdS nanoparticles [33]. Several researchers have reported that the adsorbed surfactant acts as, surface passivating agent, which draws more electronic transitions and enhances the PL intensity of the CdS nanoparticles [34–36]. The higher PL intensity of CTAB capped CuS and CdS nanoparticles confirm the high crystallinity and control over size distribution.

4. Conclusions

CuS and CdS nanoparticles capped with CTAB have been successfully synthesized using hydrothermal route. XRD pattern showed that the CTAB capped CuS and CdS nanoparticles possess hexagonal phase. The average size of nanocrystallites was about 31 nm for the CuS and 12 nm for CdS. HRTEM images and SAED pattern adequately confirmed the well dispersed and crystalline nature of the metal sulphide nanoparticles. The presence of all the functional groups was observed from FTIR analysis and this study confirmed the presence of Cu-S bond and Cd-S bond. The UV-Vis measurements showed that the band gap of 1.8 eV and 2.3 eV for CuS and CdS nanoparticles, respectively. Emission spectra of the both as-prepared samples were significantly red shifted and the higher PL intensities are attributed to the low defects and high crystallinity. Enhancing the optical properties of the CTAB capped CuS and CdS nanoparticles revealed that these samples can be used in solar cells and optoelectronics.

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Disclosure statement

All the authors have declared that no competing financial interests in this article.

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