

# A convenient noninjection one-pot synthesis of CdS nanoparticles and their studies

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## ABSTRACT

Water-dispersible CdS quantum dots (QDs) were synthesized in a simple one-pot noninjection route. The X-ray diffraction (XRD) pattern of the nanoparticles shows the cubic structure with particle size of the order 5-7 nm which was in good agreement with the transmission electron microscopic (TEM) studies. Selected area electron diffraction (SAED) recognized the cubic structure of CdS. The energy dispersive X-ray spectroscopy (EDAX) analysis confirms the presence of Cd and S elements in the samples. The optical properties are characterized by Ultraviolet-Visible (UV-Vis) absorption and Photoluminescence (PL) spectra. The synthesis parameters of this simple and rapid approach, including the reaction temperature and time, the pH of the reaction solution and the molar ratio of the 3-mercaptopropionic acid (MPA) stabilizer to Cd<sup>2+</sup>, have considerable influence on the particle size and photoluminescence of the CdS quantum dots. The 3-mercaptopropionic acid (MPA) stabilized CdS QDs can be used in solar cells, light emitting diodes, biological imaging etc. Copyright © 2013 VBRI press.

**Keywords:** CdS nanoparticles; thiourea; mercaptopropionic acid; optical properties; luminescence.



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## Introduction

Semiconductor quantum dots (QDs) have received considerable attention due to their size-dependent optical properties and applications in fields such as solar cells [1, 2], light-emitting diodes [3–6] and biological imaging [7–9]. For materials such as QDs that exhibit size-dependent properties, it is essential to understand the details of nucleation and grain growth and how they influence the evolution of particle size, size distribution and

consequently optical behavior [10, 11]. Among the reported semiconductor QDs, CdS stands out as a prominent material because of the possibility of wide tuning of its optical band gap to cover the whole visible range by varying the particle size [12, 13].

A survey of the literature shows that CdS QDs can be produced mainly via two synthetic routes, namely organic phase and aqueous phase approaches. Binding organic molecules to the surface atoms of nanoparticles is important in nanoscience for specific tailoring of physical and chemical properties of nanomaterials [14, 15]. Thus, trioctylphosphine oxide (TOPO) and trioctylphosphine (TOP) were applied as stabilizing agents [16, 17] in the traditional organic phase route. Nevertheless, QDs synthesized in organic solvents suffer from insolubility in the aqueous-phase and incompatibility with the biological system. The aqueous synthesis is reagent-effective, less toxic and more reproducible, which improves the water-stability and biological compatibility. This route is usually concerned with thiol-containing stabilizing reagents, including dihydrolipoic acid (DHLA) [18], 2 or 3-mercaptopropionic acid (MPA) [19-22], thioglycerol [23], thioglycolic acid (TGA) [24], and glutathione [25], which can interact with the surface of QDs as strongly as a covalent bond. Without sulfur atom bonding on CdS QDs, other stabilizing reagents have also been used to prepare CdS QDs, such as citrate [26], tyrosine [27] and carboxylic-functionalized PVA [28]. These surfactants or polymers are constructed around nanoparticles of various materials in order to add stability to them preventing their aggregations and minimizing surface energies [29].

A few reports have recently detailed one-pot noninjection routes to semiconductor nanocrystals [30-34]. Herein, we have developed a simple, one-pot, noninjection route for the synthesis of good-quality CdS QDs in water by simply mixing CdCl<sub>2</sub>, thiourea, and 3-mercaptopropionic acid and the structural and optical properties of CdS QDs were investigated. Synthesis in an aqueous phase is an alternative method for preparation of QDs. Compared with organic phase synthesis, the aqueous route exhibits good reproducibility, low toxicity, is inexpensive, and especially, the products have excellent water-solubility, stability, and preliminary biological compatibility.

## Experimental

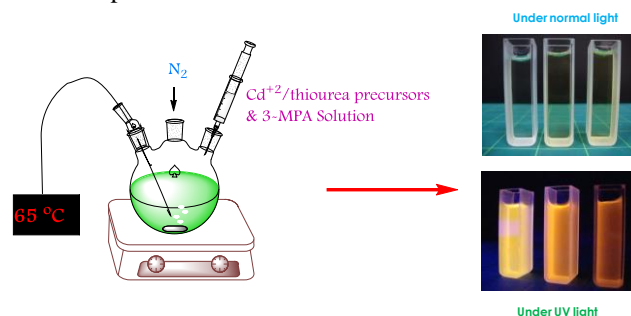
### Materials

CdCl<sub>2</sub>·2.5H<sub>2</sub>O, thiourea and mercaptopropionic acid (MPA) were purchased from Sigma Aldrich. All other chemicals used were also of analytical grade from Sigma Aldrich / Merck. Deionized water with resistivity of 18MΩ cm<sup>-1</sup> (ELGA, USA) was used for the preparation of aqueous solutions and buffers.

### Methodology

The one pot synthesis of CdS nanoparticles by reflux process has been mentioned in the literature [33]. In a typical procedure, the Cd<sup>2+</sup>/thiourea precursors solution was prepared by mixing CdCl<sub>2</sub>·2.5H<sub>2</sub>O (0.15 mmol) and thiourea (0.60 mmol) in 14 mL of ultrapure water. 20 mL

of an aqueous solution of MPA (0.395 mmol) were then added and the pH of the mixture was adjusted to 10 with 1 M NaOH. The typical molar ratio of Cd<sup>2+</sup>/thiourea/MPA was 1/4/2.6 in our experiments. The mixture was then degassed with nitrogen bubbling for 30 min. After that, the solution was heated to 65°C at a heating rate of 5°C/min in the presence of N<sub>2</sub> flow under reflux and heated at this temperature for different time intervals of 1, 2 and 3 hrs to form different sized CdS nanoparticles. The nanocrystals were further washed with ethanol and finally dried in vacuum at room temperature. The synthesis of CdS nanoparticles was conducted in a three-neck flask. Fig. 1 describes the experimental setup of one-pot synthesis of CdS nanoparticles.



**Fig. 1.** Experimental setup for synthesis of CdS nanoparticles and their photographs of nanoparticles under normal light and UV light illuminations.

### Characterizations of CdS QD's

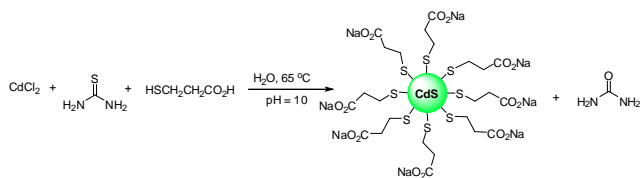
The crystalline phase of the prepared samples were characterized by X-ray diffraction analysis and the XRD patterns were obtained using a Phillips X'pert MPD 3040 X-ray diffractometer (XRD) with CuK $\alpha$  radiation ( $\lambda=1.5406$  Å). High resolution transmission electron microscope (HRTEM) images and the selected area electron diffraction (SAED) pattern were obtained using JEOL JEM-2100 at 200 keV. UV absorption spectra were recorded with Philips 7072 halogen lamp in the wavelength range 350-800 nm. The radiation from a He-Cd laser (325 nm) was used as the pump beam for photoluminescence (PL) measurements.

## Results and discussion

### Reaction mechanism

The overall chemical reactions involved in the synthesis of CdS nanoparticles are represented by the **Scheme 1**. In this scheme thiourea was selected as precursor of sulfide ions because of its easy decomposition into sulfide (S<sup>2-</sup>) or hydrogen sulfide (HS<sup>-</sup>) ions in water (at basic pH) and its relatively low activation energy which is favorable for the formation of nuclei and regular QDs. S<sup>2-</sup> or HS<sup>-</sup> anions formed in situ react with the Cd<sup>2+</sup> in the solution to form CdS nuclei that grow by heating to form CdS nanocrystals. The success of our strategy was found to strongly depend on the experimental conditions. Preliminary experiments indicated that CdS QDs could be prepared by using cadmium chloride and a 1.7-fold-excess of thiourea. The formation mechanism of CdS nanocrystals of cubic structure is due to the aqueous medium and the coordination of thiourea ligand as a molecular template

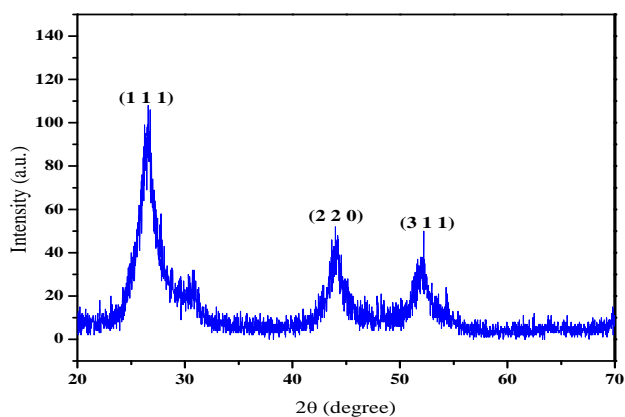
mechanism, wherein temperature and pH are critical conditions.



**Scheme 1.** Synthesis of MPA bound CdS nanoparticles.

### Structural analysis

In **Fig. 1**, the images of colloidal solutions were taken under normal light illumination and under long ultraviolet (365 nm) illumination; CdS QDs solutions display three PL colors from light yellow to dark orange with an increasing reaction time (1 to 3h) of preparation, which also clearly indicates size-dependent emission. The crystal growth of CdS QDs was noticeable as the reaction proceeded from Samples 1 to 3.



**Fig. 2.** XRD pattern of CdS QD's sample.

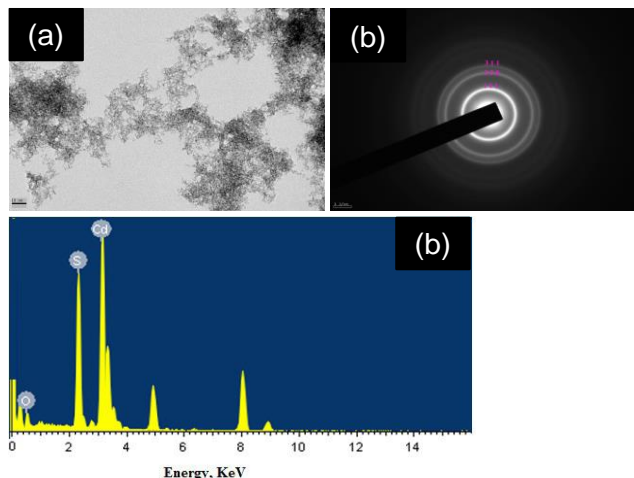
The structure of the as-synthesized CdS QDs was examined through XRD measurement (shown in **Fig. 2**). The diffraction peaks at  $2\theta$  are ca. 26.53, 43.86, and 51.77° can be readily assigned to (1 1 1), (2 2 0) and (3 1 1) planes, respectively. The diffraction peaks in this pattern can be indexed well to the cubic structure of CdS, which is in agreement with the literature (JCPDS 10-454). No peaks corresponding to impurities are detected, indicating the high purity of the product. The average particle size of the present samples was calculated using Debye-Scherrer's formula [35].

$$D = \frac{0.94 \lambda}{\beta \cos \theta}$$

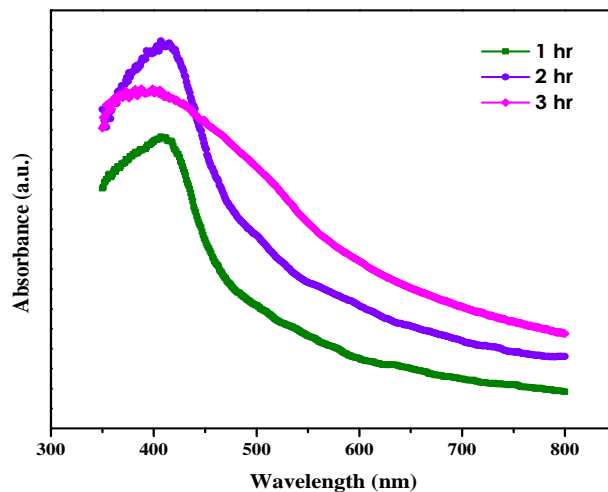
where  $D$  is the particle diameter,  $\lambda$  is the wavelength of X-ray used,  $\beta$  is the full-width at half-maximum and  $\theta$  is the scattering angle. From the XRD peaks for the CdS nanoparticles, the estimated particle size was in the range of 5-7 nm.

**Fig. 3a** shows the typical TEM image of the as-synthesized CdS nanoparticles at temperature 65°C for a reaction time of 3h. The TEM measurement confirmed that the size distribution of the as-synthesized CdS QDs was nearly mono-dispersed with the average size of 6.8 nm,

which is consistent with that obtained from XRD data. The SAED pattern of the CdS QD's confirms the cubic structure (**Fig. 3b**). Kumar et al [22] report the spherical morphology of CdS with cubic Zn-blend structure prepared in water at 100°C. The elemental analysis was performed by EDAX analysis. The EDAX spectrum of CdS nanocrystals prepared at 65°C for a reaction time of 3h is illustrated in **Fig. 3c**. These results indicate that the Cd and S elements are found in a near stoichiometric ratio.



**Fig. 3.** Images of TEM (a), SAED (b) and EDAX spectrum (c) of CdS nanoparticles synthesized at 65°C/3h.



**Fig. 4.** UV absorbance spectra of CdS nanoparticles obtained at different reaction times.

### Optical studies

**Fig. 4** depicts the temporal evolution of the absorption spectra of CdS nanoparticles synthesized at 65°C for different reaction times (1, 2 and 3 hr). These spectra exhibits a broad absorption band between 350-500 nm which was assigned to the optical transition of the first excitonic state, i.e. this absorption band was due to the first optically allowed transition of CdS between the electron state in the conduction band and the hole state in the valence band [28, 37, 38]. The shift in the absorption peak corresponds to the increase in particle diameter of the CdS QDs with duration of the heating process. The variation of the absorption peaks indicates that the particles grow

rapidly as the reaction time increased, while the absorption spectrum for the CdS sample is broadened. The broadening indicates the onset of conventional Ostwald ripening, which causes a slow defocusing of the size distribution [39].

Fig. 5 shows the PL spectra of CdS nanoparticles synthesized at 65°C for different reaction times (1, 2 and 3 hr). It can be seen that the peak position shifts towards the longer wavelengths side with the increase of reaction time i.e., from 590 to 670 nm when the reaction time rises from 1h to 3h respectively, attributed to  $1s_h-1s_e$  excitonic transitions [39]. The observed broad PL peak is commonly attributed to the recombination of charge carriers trapped in the surface states and is related to the size of CdS nanocrystals [40–43]. It can be observed that the low enough temperature of 65°C shows a largest variation of luminescence features and thus endowed the QDs with the widest optical properties. From the viewpoint of control of fluorescence color and intensity over a wide range, a reaction temperature of 65°C was appropriate for the synthesis of the CdS QDs. One can notice that during the whole reaction process, the growth of the CdS QDs was clearly evidenced by the shift of both absorption (towards shorter wavelengths) and emission (towards longer wavelengths) spectra as a consequence of the quantum confinement effect.

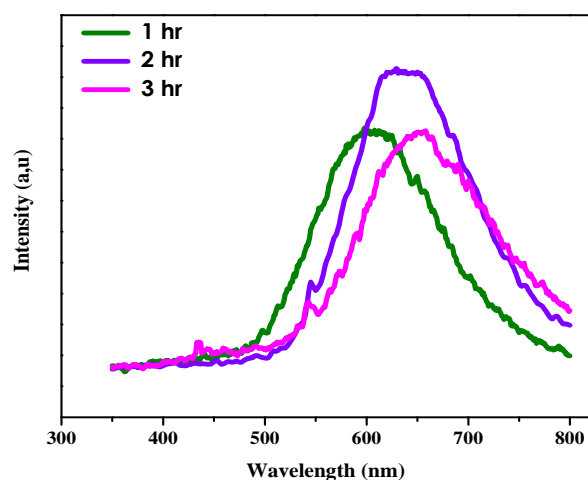


Fig. 5. PL spectra of CdS nanoparticles obtained at different reaction times.

It can also be seen that the full-width-at-half-maximum (fwhm) of PL emission spectra prepared through our method is ca. 110 nm, which is 3–4 times wider than the emission peaks of CdS QDs prepared in organic medium at elevated temperature [44–47]. This is typical of CdS QDs obtained through synthetic routes in aqueous media. Compared to aqueous syntheses of CdS QDs that use meso-2,3-dimercaptosuccinic acid [48], thioglycolic acid or 3-mercaptopropanoic acid [49, 33], thioglycerol [50], 2-mercaptoethanol [51], dendrimers [28], poly(vinyl alcohol) [52], or poly(acrylic acid) [53] as surface ligands, our method provides QDs with greater color tunability and higher PL QYs than those previously reported. Moreover, the shape of the PL band for our nanocrystals is also simpler, indicating particles with fewer defects. Finally, the time evolution of the optical properties of the as-prepared nanocrystal solutions after synthesis indicates highly

reproducible behavior up to (at least) 3 months. A novel and simple technology for preparing CdS QDs using thiourea as sulfur source has been described. The advantages of the method are as following: firstly, the preparation condition is mild, and the procedure is simple; secondly, it is a cheap and green route for synthesis of CdS QDs; thirdly, this method may be extended for the preparation of other sulfide QDs such as ZnS, CuS, and so on.

## Conclusion

In summary, we report a one-pot noninjection route for producing homogeneous CdS nanoparticles from cadmium chloride and thiourea using mercaptopropionic acid as stabilizing agent. The XRD pattern shows a cubic structure for CdS nanoparticles. The TEM result confirmed the spherical shape of nanoparticles. The average size of CdS nanoparticles was estimated to be 5–7 nm. A shift in optical absorption and PL spectra of the CdS nanoparticles with the increase of reaction time is mainly due to the quantum confinement effect. Based on the results we suggest that the present non-injection one-pot route may provide a more simplified, environmental friendly and low-cost synthesis approach, which would have potential advantages in preparing high-quality semiconductor QDs with desired sizes for both fundamental research and industrial applications.

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