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### Hydrothermal synthesis of CdTe quantum dots using ammonia as a reducing agent

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#### Abstract

Water-soluble CdTe quantum dots (QDs) have been applied in various fields, such as photoluminescent imaging, labeling agents in immune analysis, heavy metal ion sensing, and solar cells. The synthesis of CdTe QDs has relied vastly on reactions between Cd<sup>2+</sup> and Te<sup>2-</sup> ions in the presence of mercapto ligands. Te<sup>2-</sup> ions are usually formed *in situ* in the synthetic solution by reducing tellurite ions with hydride salts, such as NaBH<sub>4</sub> which gives rise to the emission of highly explosive hydrogen gas. Herein, we report a novel method to synthesize CdTe QDs by using ammonia as a reducing agent. We demonstrate that under hydrothermal conditions, ammonia can reduce tellurite forming telluride ions which react with cadmium ions in the presence of glutathione forming CdTe QDs. The resultant QDs exhibited high photoluminescence and resolvable absorption. The synthetic method demonstrated herein could reduce the cost of CdTe-based quantum dots for rapid deployment.

**Keywords:** Hydride-free, hydrothermal, CdTe quantum dots, water-soluble, ammonia

#### 1. Introduction

Because CdTe bulk semiconductor has a bandgap of 1.43 eV it allows one to tune the emission colors of CdTe quantum dots (QDs) in the visible and near-infrared regions based on the quantum confinement effects [1]. By adopting an aqueous synthetic method that was successful in preparing water-soluble CdS QDs A. Henglein and co-workers prepared CdTe QDs by the reaction between

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$\text{Cd}(\text{ClO}_4)_2$  and  $\text{Na}_2\text{Te}$  in the presence of hexametaphosphate stabilizer [2]. However, the absorption spectra of QDs were featureless and the QDs only showed weak photoluminescence in the excess of  $\text{Cd}^{2+}$ . T. Rajh and co-authors modified the synthetic protocol reported by A. Henglein by adding 3-mercaptopropionol (thioglycerol) as a co-stabilizer. They demonstrated that it could obtain CdTe QDs with a narrow-size distribution, well-resolved excitonic peak, and high photoluminescence [3]. Those early successes promoted numerous works demonstrating the use of different mercapto ligands in the synthesis of high-quality CdTe QDs that have narrow-size distribution and high emission quantum yields [4]–[7]. The synthetic protocol was further adopted to prepare other chalcogenide MX (M= Zn, Cd, Pb, Hg, Ag, Cu; X=S, Se, Te) QDs [8], [9], metal-doped CdTe QDs [10]–[13], and CdTe alloyed or core/shell QDs [14]–[18].

The formation of CdTe QDs is believed to undergo three states: monomer accumulation, nucleation, and growth [19]. A very fast monomer accumulation to facilitate instant nucleation and slow growth of nanocrystal seeds are important to narrow the size dispersion of resultant QDs. Practically, water-soluble cadmium salts and  $\text{Te}^{2-}$  precursors are usually used to synthesize high-quality CdTe QDs because  $\text{Te}^{2-}$  and mercapto ligands react quickly with  $\text{Cd}^{2+}$  ions to form complexes that coagulate to create QDs seeds in the subsequent heating process [14]. However, because  $\text{Te}^{2-}$  precursors are flammable ( $\text{H}_2\text{Te}$ ) or easily oxidized in ambient conditions, tellurite salts have been used widely to prepare CdTe QDs by a one-pot procedure [16], [19]–[21]. The "one-pot" characteristic is enabled by a parallel reaction between the tellurite salts and a strong reducing agent such as  $\text{NaBH}_4$  forming *in-situ*  $\text{Te}^{2-}$  ions. The use of hydrides brings about other safety requirements to deal with hydrogen emissions. To solve this issue, there have been several reports using ascorbic acid [22], [23], mercaptopropionic acid (MPA) [24], [25], and glutathione (GSH) as reducing agents instead of hydrides. In this study, we have demonstrated the use of ammonia as both a reducing agent and pH maintainer to synthesize CdTe QDs by a hydrothermal method. The results demonstrated herein offer a cost-effective method to prepare water-soluble and highly photoluminescent CdTe QDs by cutting down hydride salts.

## 2. Experimental section

### 2.1. Chemicals and facilities

Cadmium chloride hemipentahydrate (99.95%), zinc chloride (99.99%), sodium tellurite (99.9%), reduced glutathione (GSH, 98 %), acetone (HPLC grade), and ammonia solution (25% in water) were purchased from Aladdin chemicals and used without any purifications. Double-distilled water was freshly prepared in the laboratory and used as a synthetic solvent. Hydrothermal reactors were 100 ml PPL-liner autoclave and a Binder ED115 electric oven was used as a temperature-controlled environment.

### 2.2. The synthesis of CdTe quantum dots

Mixture solution A was made by mixing 25.5 ml of  $\text{CdCl}_2$  solution (0.02 M), 2.5 ml of  $\text{ZnCl}_2$  solution (0.02 M), 3 ml of  $\text{NH}_3$  solution (25%), and 0.19 g of GSH dissolved in 45 ml of water. Solution B was made by dissolving 0.02 g of  $\text{Na}_2\text{TeO}_3$  in 10 ml of  $\text{NH}_3$  solution. B was heated at 70°C for 1 hour followed by being transferred into A and the total solution was placed in a PPL autoclave. All processes above were conducted in an argon-filled environment. The hydrothermal treatment was conducted at 120°C in an electric oven. After being cooled to room temperature, CdTe QDs were

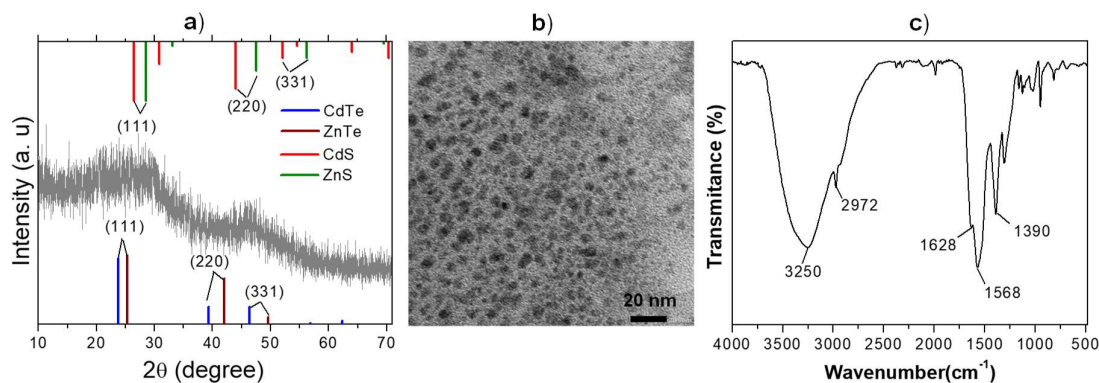
precipitated by adding acetone and collected by centrifugation at a speed of 5000 rounds per minute for 7 minutes.

### 2.3. Characterizations

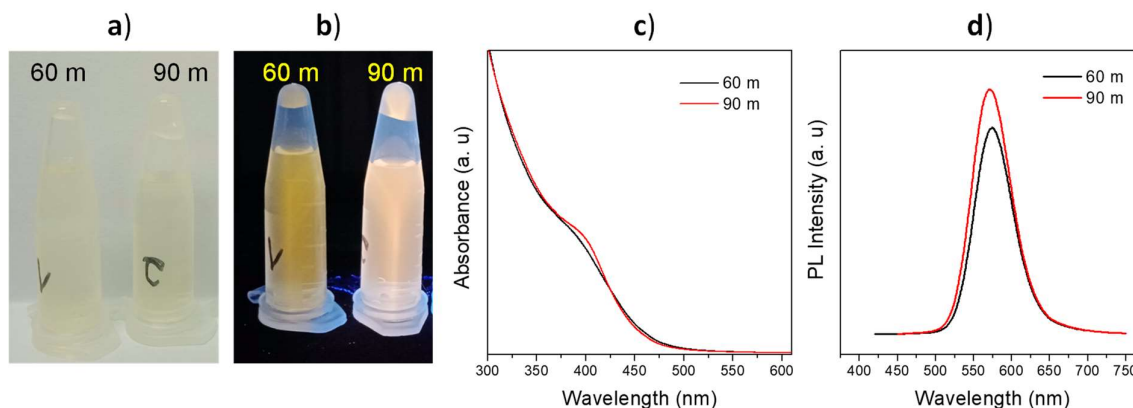
UV-vis absorption spectra were carried out on a UV-2450 (Shimadzu) spectrometer while the photoluminescent (PL) spectra were measured on a Nanolog (Horiba) spectrometer. Transmission electron microscope (TEM) images of CdTe QDs were obtained on a JEM 2100 microscope while the X-ray diffraction pattern (XRD) of QDs was carried out on a D8 advance X-ray diffractometer. FTIR spectrum of CdTe QDs was measured using an FT/IR-4600-type A FT-IR spectrometer.

## 3. Results and discussion

The X-ray diffraction pattern of CdTe QDs is shown in Figure 1.a. The pattern exhibits two broad diffraction peaks near  $27^\circ$  and  $46.3^\circ$ , and a shoulder at about  $50.7^\circ$ . The first broad peak near  $27^\circ$  is close to the CdTe, ZnTe, CdS, and ZnS (111) plans. Among those possible crystal phases, CdS and ZnS were taken into account because under hydrothermal conditions  $S^{2-}$  ions could be *in situ* formed via the decomposition of GSH molecules. The peak at  $46.3^\circ$  could be assigned to the (311) plan CdTe or the (200) plan of ZnS while the shoulder at  $50.7^\circ$  was attributed to either the (311) of ZnTe or CdS. The fact that the diffraction peaks could not be assigned to a single crystal phase indicates the QDs were in a mixed phase such as a CdZnTeS alloyed structure that is obtained by heating a mixture of Cd and Zn salts, tellurite, mercapto ligands, and hydride [14], [15], [26]. On the other hand, the broadness of the diffraction peaks was partial due to the small size of QDs. TEM images shown in Figure.1b indicate that CdTe QDs were fairly spherical with a diameter varying from 4.5 to 10 nm. In addition, QDs were individual in TEM images indicating that QDs were stable colloids. XRD and TEM results show that crystalline CdTe QDs were successfully synthesized. The FTIR spectrum of CdTe QDs shown in Figure 1.c exhibited a broad band at  $3250\text{ cm}^{-1}$ , a peak at  $2972\text{ cm}^{-1}$ , a double peak at  $1568$  and  $1628\text{ cm}^{-1}$ , and a sharp peak at  $1390\text{ cm}^{-1}$ . The broadband was attributed to vibrations of polar bonds such as N-H and O-H in GSH molecules. The peak at  $2972\text{ cm}^{-1}$  was assigned to the C-H stretching in methylene groups of GSH while the double peak was due to the amide group in GSH [16]. The sharp peak at  $1390\text{ cm}^{-1}$  could be attributed to the bending vibration of C-NH bonds in GSH [27]. Those features show that the QDs were capped with GSH molecules. Additionally, the characteristic peak of the S-H bond at about  $2526\text{ cm}^{-1}$  [19] was absent in the FTIR spectrum indicating that GSH molecules bond to CdTe QDs via the thiol group.



**Figure 1.** a) XRD spectrum, b) TEM image, and c) FTIR spectrum of CdTe quantum dots obtained after 90 minutes of hydrothermal treatment. In a) the standard XRD peaks of CdTe (JCPDS#: 15-0770), CdS (JCPDS#: 47-1179), ZnTe (JCPDS#: 15-0746), and ZnS (JCPDS#: 05-0566) are indicated for comparison.



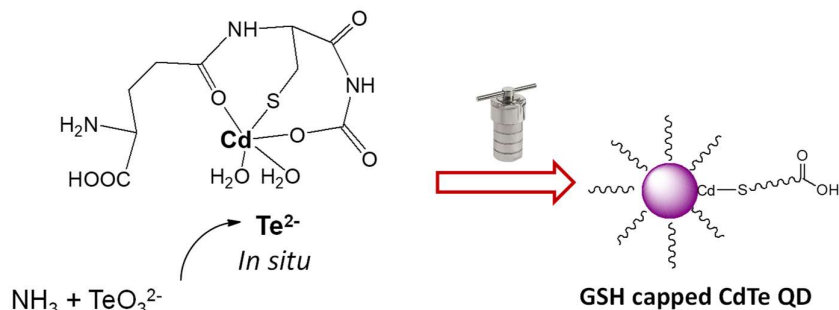
**Figure 2.** a) Pictures of CdTe quantum dots solutions under daylight and b) under UV light (365 nm). c) The absorption and d) photoluminescent spectra of CdTe quantum dots obtained after 60 minutes (60 m) or 90 minutes (90 m) of hydrothermal treatment.

The optical properties of CdTe QDs are summarized in Figure 2. The resultant QDs were soluble in water and exhibited strong emission in the yellow region (Figure 2.a-b). The absorption spectra (Figure 2.c) of QDs obtained at different periods of hydrothermal treatment show an adsorption shoulder at about 400 nm but the excitonic peak was not observed. In fact, when the hydrothermal temperature increased to 160°C, the shoulder red-shifted to about 410 nm (results are not shown). In the conventional synthesis in which  $\text{NaBH}_4$  is used as a reducing agent, we could obtain CdZnTeS alloyed QDs with a well-resolved excitonic peak which gradually red-shifts as increasing reaction time [16]. Because the sharpness of the excitonic peak is governed by the narrowness of QDs size distribution the absence of excitonic peaks in Figure 2.b coincided with the broadness in the diameter of QDs seen in Figure 1.b and infers that the current method provides QDs with a lower size-distribution as compared with conventional method [16]. PL spectra excited at 400 nm are shown in Figure 2.d. The emission spectra were broad, maximized at about 570-576 nm which was about the onset point of the absorption spectrum seen in Figure 2.c. This was due to the energy transfer among QDs of different sizes in the QDs solution. Although the size distribution of CdTe QDs was not very narrow, highly photoluminescent CdTe QDs were obtained probably due to the inclusion of Zn on the surfaces [16], [19]. In the next part, we will discuss the formation mechanism of CdTe QDs upon hydrothermal treatment.

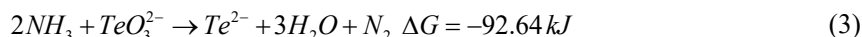
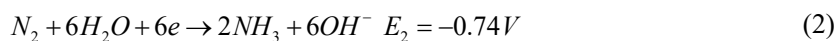
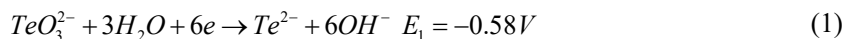
In conventional synthesis, a mixture solution containing  $\text{Cd}^{2+}$ ,  $\text{TeO}_3^{2-}$ , mercapto ligands, and  $\text{NaBH}_4$  is heated either by refluxing or hydrothermal treatments [16], [19], [28], [29]. The formation of CdTe QDs relies on the reaction between Cd-mercapto complexes and  $\text{Te}^{2-}$  ions, which are formed *in situ* by reducing  $\text{TeO}_3^{2-}$  with  $\text{NaBH}_4$ . There have been some reports about replacing hydride salts by other reducing agents. In an early study, S. R. Stürzenbaum and co-workers could obtain green-emitting CdTe QDs by exposing earthworms to soils spiked with  $\text{CdCl}_2$  and  $\text{Na}_2\text{TeO}_3$  and the authors reasoned the formation of QDs to be due to the reduction of  $\text{Na}_2\text{TeO}_3$  by GSH [30]. Similarly, M. Shen attributed the formation of CdTe QDs by refluxing a mixture of  $\text{CdCl}_2$ ,  $\text{Na}_2\text{TeO}_3$ , 3-mercaptopropionic acid (MPA), and NaOH to the reduction of  $\text{Na}_2\text{TeO}_3$  by MPA [25].

In this study, we heated a mixture of  $\text{CdCl}_2$ ,  $\text{Na}_2\text{TeO}_3$ , GSH, and ammonia. GSH and ammonia were possible reduction agents in this system. Based on the standard redox potential of chalcogenides the potential of  $\text{TeO}_3^{2-}$  is calculated to be about -0.58V versus the standard hydrogen electrode (vs. SHE) [31], see the half-reaction (1). The redox potential of mercapto ligands was reported to be about

-0.4V vs. SHE [32] in alkaline solution which is more positive than the potential of  $\text{TeO}_3^{2-}$ . Therefore, GSH cannot reduce  $\text{TeO}_3^{2-}$  to  $\text{Te}^{2-}$  at the standard conditions. However, the works reported by S. R. Stürzenbaum [30] and M. Shen [25] mentioned above indicate that GSH can reduce  $\text{TeO}_3^{2-}$  in biological systems or at higher temperatures. Under hydrothermal conditions as in our synthetic method, GSH could reduce  $\text{TeO}_3^{2-}$  forming *in situ*  $\text{Te}^{2-}$  ions. Considering half-reaction (2) shown below, the redox potential of  $\text{N}_2/\text{NH}_3$  is -0.74V vs. SHE [33] which is more negative than GSH and makes the reduction of tellurite (reaction 3) to be thermodynamically favorable. Likely, in our study,  $\text{NH}_3$  was the main agent to reduce tellurite resulting *in situ*  $\text{Te}^{2-}$  ions for the formation of CdTe QDs as illustrated in Figure 3.



**Figure 3.** A tentative formation mechanism of CdTe quantum dots by the reaction between Cd-GSH complex and *in situ* formed telluride ions.



#### 4. Conclusions

Water-soluble, highly photoluminescent CdTe quantum dots were successfully prepared by hydrothermal treatment mixtures of  $\text{CdCl}_2$ ,  $\text{ZnCl}_2$ , and  $\text{Na}_2\text{TeO}_3$  in ammonia solutions. The QDs were crystalline in a CdZnTeS alloyed structure. The QDs were spherical colloids with a diameter ranging from 4.5 to 10 nm. Based on thermodynamic calculations we deduced that ammonia can reduce tellurite ( $\text{TeO}_3^{2-}$ ) ions to telluride ( $\text{Te}^{2-}$ ) ions for the formation of QDs. The results demonstrated herein suggest new methods to prepare cost-effective telluride-based quantum dots.

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