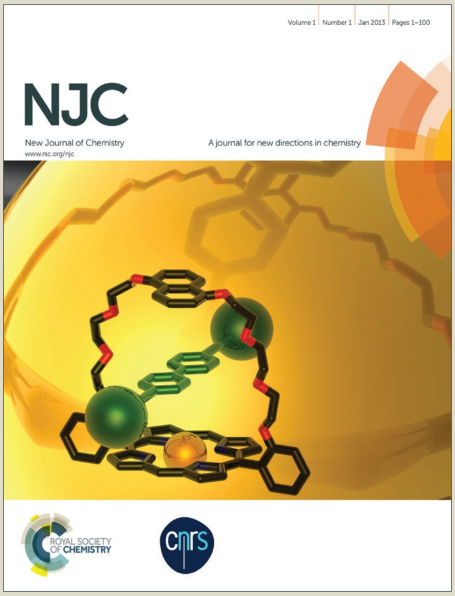

Facile synthesis of CdTe-based quantum dots promoted by mercaptosuccinic acid and hydrazine

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ABSTRACT

CdTe-based quantum dots (QDs) with high photoluminescence quantum yield (PL QY) were synthesized in a short time (less than 45 minutes). Mercaptosuccinic acid (MSA) was employed as a stabilizer and N_2H_4 as a growth promoter to accelerate the growth of CdTe and $CdSe_xTe_{1-x}$ QDs. Red-emitting CdTe QDs with PL QY of 25% were obtained and the highest PL QY reached 55%. $CdSe_xTe_{1-x}$ QDs with emission peak positions of 518 nm to 750 nm were obtained. The rapid growth of the QDs depends on the interaction between MSA and Cd^{2+}, and N_2H_4 plays a key role in accelerating the growth to a certain level. Thus, the QD particle size can be controlled by manipulating N_2H_4 concentration in the solution. A low N_2H_4 concentration seems feasible to obtain high-quality QDs.

Key words: mercaptosuccinic acid; hydrazine; CdTe quantum dots; growth mechanism; photoluminescence quantum yield

1. Introduction

Quantum dots (QDs) exhibit special physical and chemical properties and are significantly different from the corresponding bulk materials.[1-5] In the past two decades QDs demonstrated application potentials in the fields of light emitting diodes (LEDs), solar cells, biomedical labels and so on.[6-15] Among various types of QDs, CdTe QDs have received wide attention due to their tunable optical properties and simple synthesis process.

High quality CdTe QDs are generally synthesized in organic phases, while aqueous route has attracted increasing interest since the appearance of the aqueous synthesis of mercaptoethanol- or thioglycerol-capped CdTe QDs.[16] Nevertheless, in most of the aqueous routes, a relatively high temperature (≥100 ° C) and/or the degassing by N_2 are usually indispensable in order to provide suitable conditions for QDs to grow. It is reported that N_2H_4 can create oxygen-free surroundings at high temperature in preparing aqueous CdTe QDs.[17-22] Though N_2H_4 shows good performance, large dosage of N_2H_4 was employed in the above work ($N_2H_4:Cd^{2+} \geq 800$), leading to a waste of N_2H_4 and low stability of CdTe QDs in N_2H_4-rich solutions. Mercaptosuccinic acid (MSA) is a new stabilizer that shows good performance in synthesizing aqueous QDs,[23-28] but not used so widely as the classical stabilizers like thioglycollic acid (TGA) and mercaptopropionic acid (MPA).

Herein we present a green and facile method to synthesize high-quality aqueous CdTe QDs with tunable sizes by employing MSA as stabilizer and N_2H_4 as growth promoter. There are reports on the application of MSA or N_2H_4 (or some alkyl chain diamines) in aqueous synthesis of QDs,[17-19, 24-28] but to the best of our knowledge no literature has referred to the combined use of the two reagents. There was no need to prepare precursors and use N_2 to protect the reaction systems in this work. Furthermore, alloyed $CdSe_xTe_{1-x}$ QDs were prepared in a similar way to broaden the emission range.

It is difficult to obtain CdTe QDs with red emission. Such QDs are usually prepared by heating for a long time (over 4 hrs), leading to a decrease in photoluminescence quantum yield (PL QY).[29] We successfully synthesized CdTe QDs with blue-green to near-infrared emission within only 30 - 45 minutes, and the PL QY reached 55%.

2. Experimental section

2.1 Materials

Cadmium chloride hemipentahydrate ($CdCl_2 \cdot 2.5H_2O$, 99.0%) was purchased from Sinopharm Chemical Reagent Co., Ltd, China. Mercaptosuccinic acid (MSA, 98%), Rodamine 6G and sodium tellurite (Na_2TeO_3, 97%) were purchased from Aladdin Chemistry Co., Ltd, China. Sodium borohydride ($NaBH_4$, 96.0%) was purchased from Shanghai Hengsheng Fine Chemical Technology Co., Ltd, China. Hydrazine hydrate ($N_2H_4 \cdot H_2O$, 80%) was purchased from Tianjin Fuchen Chemical

Reagents Factory, China. Sodium selenite (Na_2SeO_3, 98%) and sodium hydroxide (NaOH, 96%) were purchased from Beijing Chemical Works, China. All the chemicals were of analytical grade and used as received without any further purification.

2.2 Preparation of MSA-stabilized CdTe QDs

Typical synthesis procedure of MSA-capped CdTe QDs was as follows (some process referred to the work of Zhou et al.):[18] 1.5mmol of $CdCl_2{\cdot}2.5H_2O$ and 1.65 mmol of MSA were dissolved in 40 ml of deionized water, which was degassed with N_2 for 25 minutes. The pH of the solution was adjusted to 8 with 5 M NaOH to obtain solution A, while solution B was prepared by dissolving 0.25 mmol of Na_2TeO_3 into 10 ml of deionized water. Solution A, solution B and 1.75 mmol of $NaBH_4$ were mixed together under stirring for 3 minutes to obtain a deep orange solution C containing CdTe monomer. The molar ratio of MSA/Cd/Te was 6.6/6/1. The CdTe monomer concentration in solution C was 5 mM and the pH of solution C reached about 10 due to the alkalinity of $NaBH_4$. A series of samples (10 ml for each) were taken from solution C and added with different amount of N_2H_4 to tune the size of CdTe QDs. Then the samples were placed in a water bath of 95°C in open air. After 30 minutes, the solution was cooled down slowly to room temperature. This process is more facile and efficient than that in our previous work,[30] which also employed some assistant reagents to improve the quality of CdTe QDs. The PL QYs of CdTe QDs samples were calculated using rhodamine 6G (with ethanol as solvent) as a reference (QY considered as 95%, excited at 488 nm).[19, 31] All the samples were tested by a UV lamp (ZF-I, 254 nm, 365 nm, 15W*2), UV-visible spectroscopy (UV-2600, Shimadzu) and PL spectrophotometer (Cary Eclipse, Varian). The morphologies and sizes of the samples were characterized by a high resolution transmission electron microscope (HRTEM, JEOL JEM-3010). The crystal structures of the samples were analyzed by X-ray diffraction (D8 FOCUS, Bruker AXS).

2.3 Preparation of MSA-stabilized $CdSe_xTe_{1-x}$ QDs

The synthesis of $CdSe_xTe_{1-x}$ QDs was similar to that of CdTe QDs mentioned above. Solution A contained the very same materials as in the synthesis of CdTe, while solution B2 was prepared by dissolving a certain mole of Na_2TeO_3 and Na_2SeO_3 into 10 ml of deionized water. The total mole number of Se+Te was 0.25 mmol in solution B2. Five different molar ratios of Se/(Se+Te) (Se composition), i.e. 0, 0.1, 0.25, 0.5, 0.6, were employed to prepare the QDs. Solution A, solution B2 and 1.75 mmol of $NaBH_4$ were mixed together to obtain solution C2 with an orange colour. Se composition influenced the final colour of solution C2, and the higher the Se/(Se+Te), the lighter the colour of solution C2 was. The molar ratio of MSA/Cd/(Se+Te) was 6.6/6/1. After adding a certain dosage of N_2H_4, solution C2 was heated in a water bath of 95°C in open air for 45 minutes and then cooled down slowly to room temperature. The samples were characterized by the above-mentioned instruments and methods for CdTe QDs.

3. Results and Discussion

Basically, CdTe QDs were formed in aqueous phase according to the following reactions:

CdTe QDs by MSA and hydrazine

$$3\,BH_4^- + 4\,TeO_3^{2-} \rightarrow 4\,Te^{2-} + 3\,BO_2^- + 6\,H_2O \qquad \text{(equation 1)}$$

$$Cd\text{-}MSA + Te^{2-} \rightarrow CdTe\text{-}MSA \qquad \text{(equation 2)}$$

As can be seen in equation 1 and equation 2, $NaBH_4$ reduces Na_2TeO_3 to Te^{2-}, then the freshly-generated Te^{2-} reacts with Cd^{2+} to give CdTe. It is known that the stabilizer, precursor, assistant reagent have strong influences on CdTe QDs growth.[32,33] MSA was employed as a stabilizer in this work because MSA can result in a fast growth of the CdTe QDs and obtain high quality products.[24, 25] N_2H_4 is another key reagent in this work because it can facilitate the growth of CdTe QDs and protect the QDs from oxidation.

3.1 Optical properties of the as-prepared CdTe QDs

Zero, 20, 95, 295, 605, 1090 µL of N_2H_4 were added into six solution C samples respectively to reach N_2H_4 concentrations of 0, 30, 150, 450, 950, 1600 mM. Fig. 1 (a) (b) show the images of CdTe QDs with various sizes under natural light and UV light at 365 nm. The emission peak positions (from left to right) were 512 nm (blue-green), 540 nm (bright green), 564 nm (bright yellow), 590 nm (amber), 606 nm (light red) and 650 nm (dark red). Fig. 1 (c) (d) indicate that the peak position of UV-visible spectra shifted from 496 nm to 636 nm while the peak position of PL spectra shifted from 512 nm (blue-green) to 650 nm (dark red). The full widths at half maximum (FWHMs) were 45 nm, 47 nm, 51 nm, 53 nm, 56 nm, 60 nm. It can be found that the dosage of N_2H_4 had significant effect on CdTe QDs growth. Also, different concentrations of NaOH were used instead of N_2H_4 to prepare CdTe QDs, but it was observed that CdTe QDs with various sizes were not obtained, which means that the effect of N_2H_4 in CdTe QDs synthesis is not due to the alkalinity but for its reductive and growth-promoting properties.

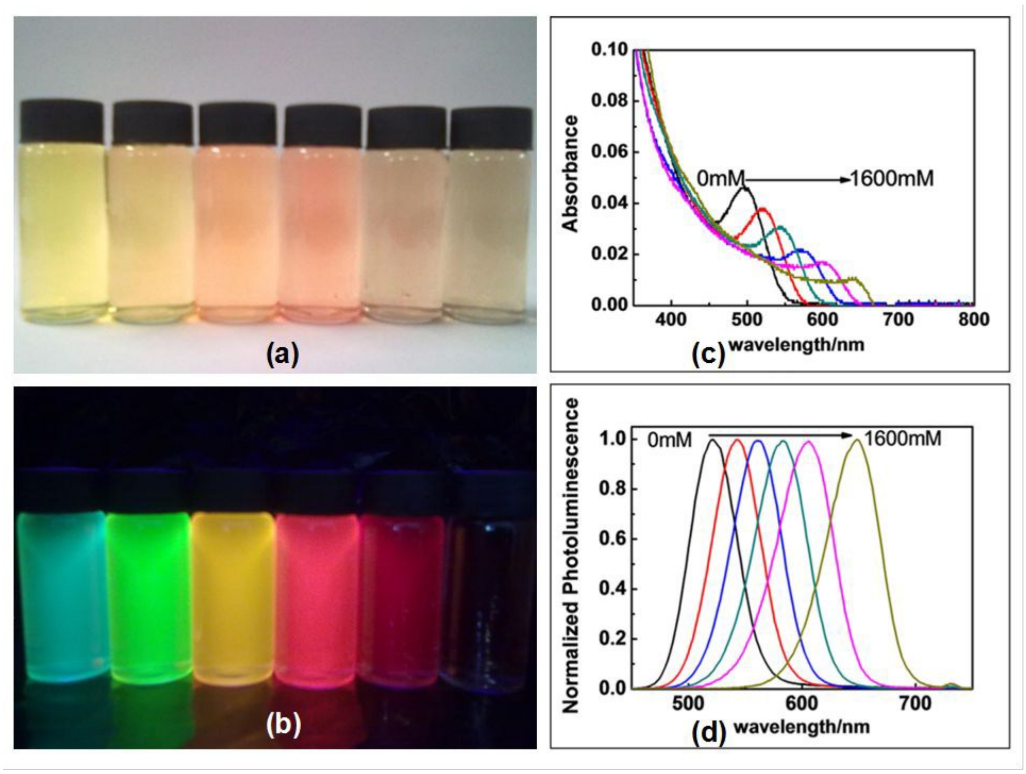


Fig. 1 Optical properties of CdTe QDs synthesized with different concentrations of N_2H_4.Images of CdTe QDs irradiated with (a) natural light and (b) UV light (365 nm, 15W). The emission peak positions of the samples (from left to right) were: 512 nm (blue-green), 540 nm (bright green), 564 nm (bright yellow), 590 nm (amber), 606 nm (light red) and 650 nm (dark red); (c) UV-visible spectra and (d) normalized PL intensity of CdTe QDs (N_2H_4 concentration from left to right: 0, 30, 150, 450, 950, 1600 mM; molar ratio of MSA/Cd/Te=6.6/6/1).

Molecular structures of stabilizers strongly influence the growth of QDs.[34] In aqueous phase, the main effect on the growth of CdTe QDs should be attributed to the different terminal groups on different thio-ligands.[35] The nucleation is followed by crystal growth via coalescence and fusion of smaller clusters, according to a classical theory.[36, 37] The terminal groups on the thio-ligands may facilitate or prevent the coalescence and fusion into larger clusters. For example, the amine terminal groups on mercaptoethylamine (MA) have strong hydrogen bonds with the amine terminal groups of the neighboring monomers and/or the smaller clusters at pH 9 since the amine terminal groups are uncharged. Thus it may facilitate the coalescence and fusion, leading to a fast growth of QDs.

Thio-ligands, such as TGA and MPA with the carboxylic groups, can prevent the coalescence and fusion like hydroxyl groups do.[35] However, the carbonyl-oxygen of TGA or MPA may coordinate with Cd sites of monomers and/or clusters, called secondary coordination,[38] which favors the coalescence of the monomers and/or clusters. But TGA and MPA are somewhat different. The carbonyl-oxygen of TGA prefers to couple the Cd sites not capped with the thio-sulfur, thus favoring the addition of monomers or clusters to QDs, while the carbonyl-oxygen of MPA may link to the same Cd site as the thio-sulfur does.[39]

The growth of QDs may be divided into the early fast growth stage and the later slow growth stage. In the early fast growth stage, QDs growth is dominated by adding monomers and/or clusters to nanocrystals so that TGA-stabilized QDs have a faster growth. In the later stage, MPA-stabilized QDs benefit from the diffusion-controlled QDs growth via Ostwald Ripening (OR), making a faster growth than TGA-stabilized QDs. MSA consist of both TGA-like and MPA-like moieties. Thus the TGA-like part is dominant in the early fast growth stage and the MPA-like part is dominant in the later slow growth stage, thereby leading to a fast growth of QDs.

CdTe QDs by MSA and hydrazine

In our work, the addition of N_2H_4 led to NH_2-rich surroundings. The amine terminal groups on N_2H_4 are similar to those on MA, which form strong hydrogen bonds with the amine terminal groups of the neighboring monomers and/or the smaller clusters at pH 10. In the early fast growth stage, Cd sites may couple with both carbonyl-oxygen on TGA-like part of MSA and hydrazine-nitrogen of N_2H_4, making a combined effect on facilitating coalescence of the monomers and/or clusters. In the later slow growth stage, the MPA-like part of MSA facilitates the diffusion-controlled QDs growth. The rate of CdTe QDs growth in our work was manipulated by changing N_2H_4 concentration, which can at least accelerate the early fast growth stage to a certain level. A higher N_2H_4 concentration led to a faster growth of the CdTe QDs. When N_2H_4 concentration was equal to or higher than 950 mM, red-emitting CdTe QDs were obtained in only 30 minutes (see Fig. 1). It can be inferred that the growth of red-emitting QDs in this work may experience the early fast growth stage and the later slow growth stage in a short time.

3.2 XRD patterns and HR-TEM images of the as-prepared CdTe QDs

XRD patterns and HR-TEM images of the as-prepared CdTe are showed in Fig. 2 and Fig. 3, respectively. The samples were obtained under typical conditions as mentioned in the experimental section. The XRD patterns indicate that the lattice parameters of the as-prepared CdTe QDs fitted the zinc blende structure of bulk CdTe crystal.[16] The diffraction peaks at about $2\theta=24^{\circ}$, 39° and 47° belong to the zinc blende CdTe crystal faces of (111), (220) and (311), respectively (zinc blende pattern of bulk CdTe also shown in the background of Fig. 2). Particle sizes displayed in Fig. 2 were measured directly from the HR-TEM images. When the particle size decreased, the width of the diffraction peaks broadened considerably. Fig. 3 shows the HR-TEM images of CdTe QDs samples prepared with N_2H_4 concentration of 0, 150, 450 and 1600 mM. It can be seen that the particles had a good dispersion and the particle size was about 3-6 nm.

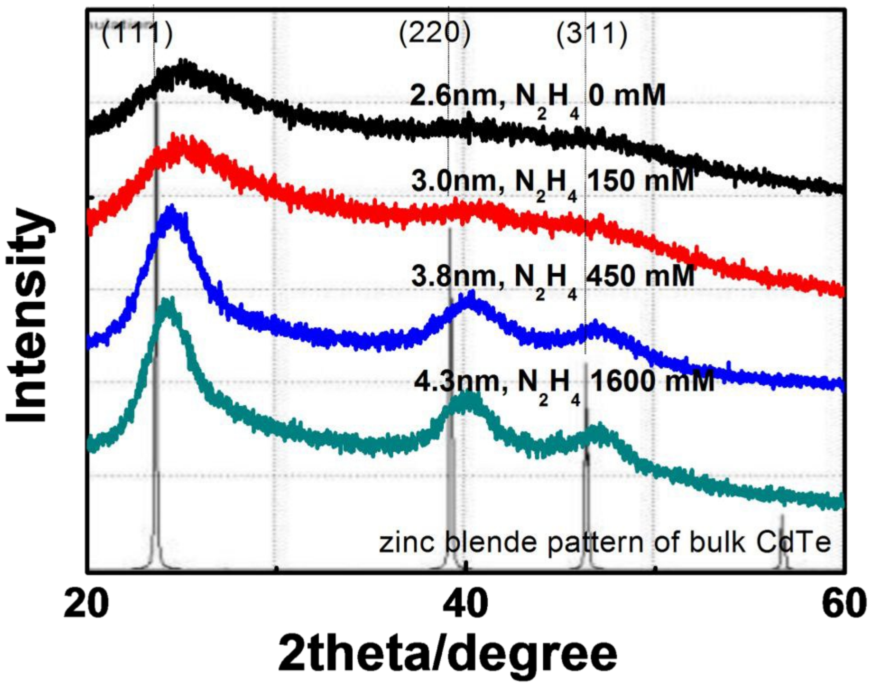


Fig. 2 XRD patterns of CdTe QDs synthesized with different N_2H_4 concentrations.

CdTe QDs by MSA and hydrazine

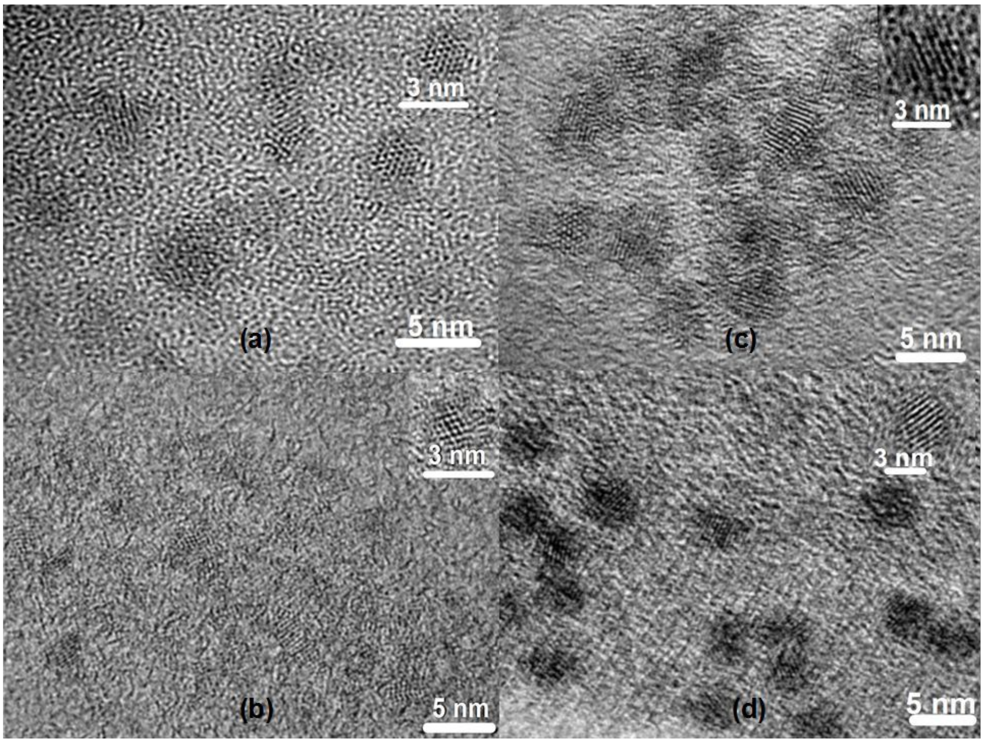


Fig. 3 HR-TEM images of CdTe QDs prepared with N_2H_4 concentrations of (a) 0, (b) 150, (c) 450 and (d) 1600 mM.

3.3 Stability of the as-prepared CdTe QDs

Table 1 listed the emission peak positions and PL QYs of the as-prepared CdTe QDs measured the very same day and after two months. The samples were synthesized under typical conditions as mentioned in the experimental section without any further treatment since they were cooled down to room temperature. As can be seen, N_2H_4 concentration had a significant influence on the red-shift of the CdTe QDs. Even without N_2H_4, the QDs had a small red-shift of 6 nm, while a N_2H_4 concentration of 30 mM led to a big red-shift of 20 nm. A higher N_2H_4 concentration led to a greater red-shift of the QDs, indicating that excessive N_2H_4 led to a significant growth of CdTe even at room temperature.[19] The PL QYs did not decrease much (some increased indeed) after two months' storage in untreated solution. Generally, the stability of CdTe QDs in this work was superior to that promoted by N_2H_4 at room temperature in reference,[18] because our products formed at a low concentration of N_2H_4.

One as-prepared CdTe QDs sample was precipitated with ethanol to wash the excessive N_2H_4 and MSA, and the precipitate was separated by centrifugation and re-dissolved in deionized water. This process was repeated for 4~6 times to purify the CdTe QDs. The purified QDs powder was stored in a dry, shady place at room temperature for more than 8 months, and it was still very stable whether kept at solid state or re-dissolved into water, without any appreciable change on observation and still emitting bright light under UV irradiation.

Table 1 Emission peak positions and PL QYs of CdTe QDs measured the very same day compared with those stored at room temperature for two months.

N_2H_4 concentration/mM	0	30	150	450	950
Emission peak/nm	512	540	564	590	606
PL QY/%	20.3	39.3	32.4	17.7	10.3
Emission peak- two months later/nm	518	560	590	623	660
PL QY- two months later/%	23.0	41.1	27.3	14.2	7.7
Red shift/nm	6	20	26	33	54

3.4 Emission peak positions and PL QYs of CdTe QDs synthesized at different material ratios

Different molar ratios of materials were investigated in order to determine suitable operating conditions for CdTe QDs synthesis. Instead of MSA/Cd/Te ratio of 6.6/6/1 in a typical synthesis, ratios of 12/10/1, 24/20/1 and 30/25/1 were investigated while other conditions remained unchanged. Zero, 50, 580, 1200, 2500, 3850 mM of N_2H_4 was added respectively for the ratio of 12/10/1. Zero, 60, 500, 1200, 2500, 4500 mM of N_2H_4 was added respectively for the ratio of 24/20/1, and 0, 50, 400, 950, 2000, 3200 mM of N_2H_4 was added respectively for the ratio of 30/25/1. The final concentration of CdTe QDs was 5 mM (calculated by monomer CdTe concentration) in all the experiments.

Fig. 4 (a) shows the effect of N_2H_4 concentrations on the emission peak positions. The four curves almost overlapped, except that the curve obtained at the ratio of 6.6/6/1 deviated slightly from the other three. This is because the ratio of MSA/Cd was 1.1 in samples of this curve but the ratio was 1.2 in other samples. A lower ratio of MSA/Cd led to a looser structure of Cd^{2+}-thiol complex and facilitated the coalescence and fusion into larger clusters, resulting in a faster growth of CdTe QDs, at least in the early fast growth stage. Even so, the difference between the curve with the ratio of 6.6/6/1 and the other three is unobvious. These results suggest that at a certain ratio of MSA/Cd, the emission peak position can be calculated by the N_2H_4 concentration even if the Cd/Te value changes much.

CdTe QDs by MSA and hydrazine

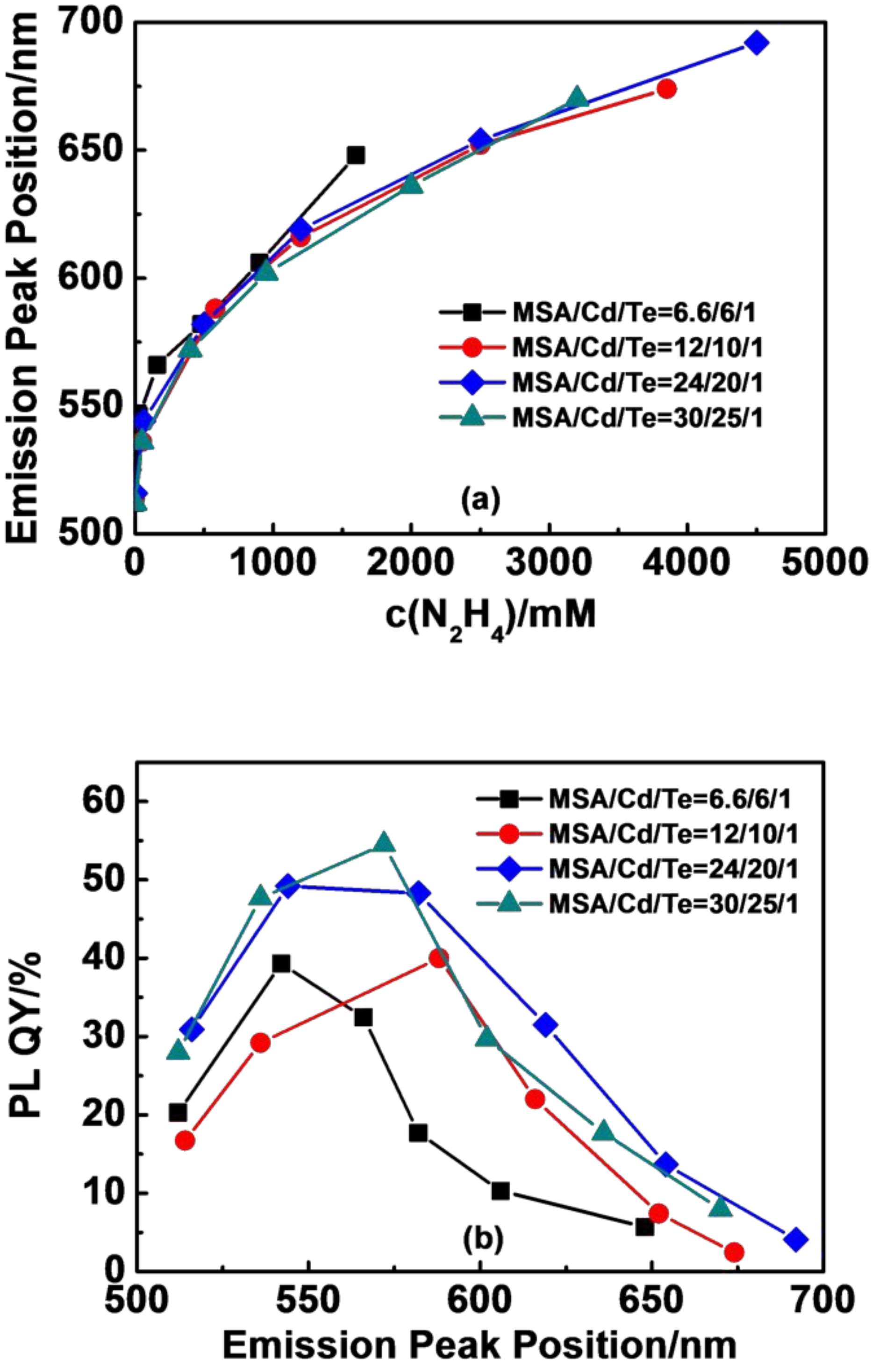


Fig. 4 Emission peak positions and PL QYs of CdTe QDs. (a) Emission peak positions of CdTe QDs synthesized with different N_2H_4 concentrations; (b) Relationship of PL QYs of CdTe QDs with emission peak positions. The CdTe QDs

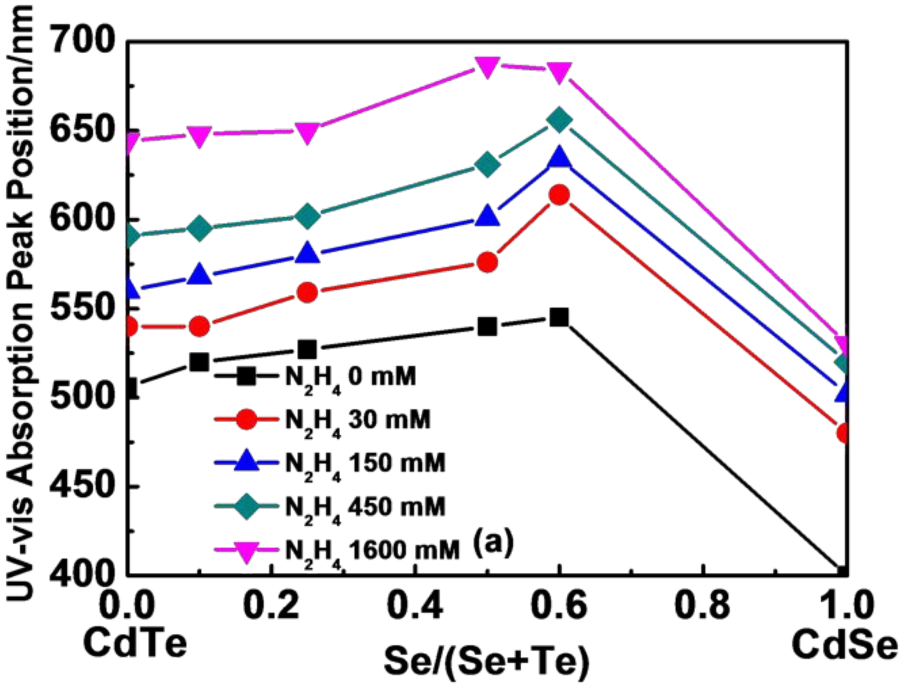
concentration was 5 mM.

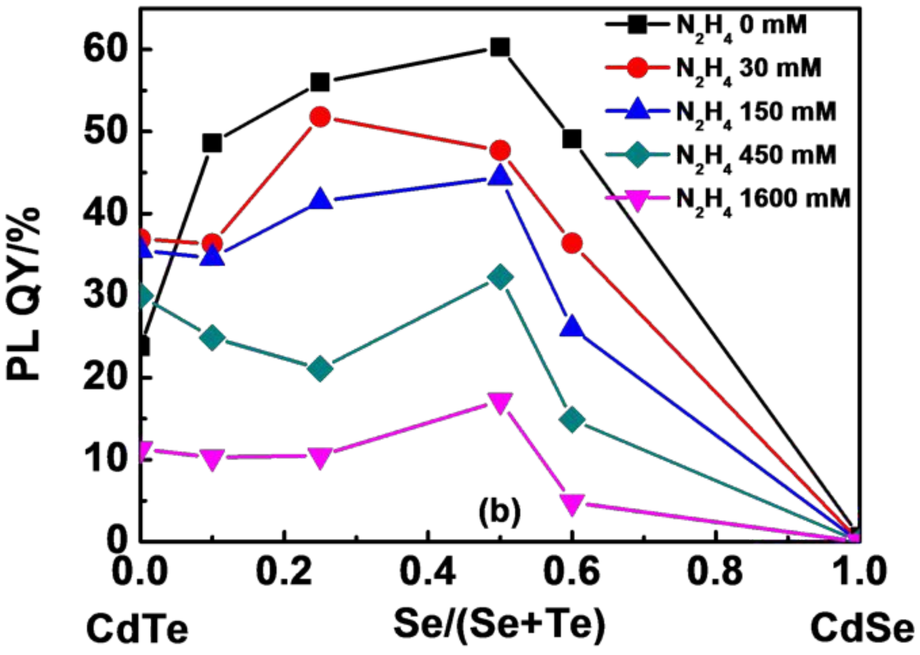
Fig. 4 (b) demonstrates the PL QYs at different emission peak positions. Generally, a higher Cd/Te ratio resulted in a higher QY. It can be seen that the highest QYs of 40%, 41%, 49% and 55% were obtained with MSA/Cd/Te ratio of 6.6/6/1, 12/10/1, 25/20/1 and 30/25/1 at the emission peaks of 540, 590, 544 and 573 nm respectively. The corresponding N_2H_4 concentrations were 30, 580, 60 and 400 mM as can be seen in Fig. 4 (a). These results indicate that a low N_2H_4 concentration (several dozen to several hundred mM) is favorable for the production of CdTe QDs with high QYs.

Some QDs require specific preparation methods to achieve special performance.[40, 41] It is not easy to obtain red-emitting CdTe QDs. A long reaction time is required to synthesize such QDs and usually hydrothermal, microwave or other means are needed.[25,42] These red-emitting CdTe QDs cannot meet the requirements for biomedicine applications due to low PL QYs.[24] To address this issue, transition metal like Mn was doped into CdTe QDs or alloyed QDs instead of bare CdTe QDs were synthesized to obtain high quality red-emitting QDs.[43-46] In this work, red-emitting CdTe QDs with the PL QYs of about 25% were obtained at the MSA/Cd/Te ratios of 30/25/1 and 24/20/1 and N_2H_4 concentration of around 1700 mM.

3.5 Spectra properties and PL QYs of the as-prepared $CdSe_xTe_{1-x}$ QDs

Fig. 5 shows UV-vis peak positions, PL QYs and PL peak positions of bare CdTe, CdSe QDs and alloyed $CdSe_xTe_{1-x}$ QDs obtained at an MSA/Cd/(Se+Te) ratio of 6.6/6/1. It is known that alloyed $CdSe_xTe_{1-x}$ QDs have a lower bandgap and the relationship between Se molar ratio *x* (or Se composition, or Se/(Se+Te)) and energy change is nonlinear,[46] which suggests that Se doped CdTe QDs can absorb light with a longer wavelength compared with bare CdSe and CdTe QDs.





CdTe QDs by MSA and hydrazine

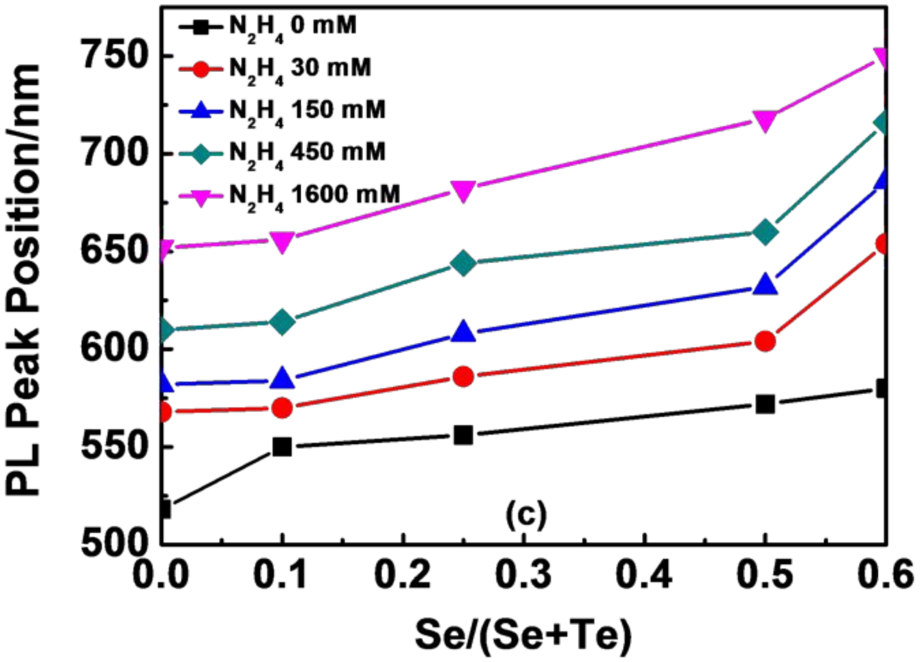


Fig. 5 Optical properties of $CdSe_xTe_{1-x}$ QDs.(a) UV-vis absorption peak positions vs. Se/(Se+Te); (b) PL QYs vs. Se/(Se+Te); (c) PL peak positions vs. Se/(Se+Te).

Fig. 5 (a) demonstrates the UV-vis absorption peak positions of $CdSe_xTe_{1-x}$ QDs at different concentrations of N_2H_4, compared with bare CdSe and CdTe QDs. When the molar ratio of Se/(Se+Te) increased, the UV-vis absorption peaks in the curves showed a red shift and reached the longest wavelength at the molar ratio of 0.6. The wavelengths of UV-vis absorption (UV-vis peak position) of bare CdSe and CdTe QDs were shorter than any of the alloys, implying that alloyed $CdSe_xTe_{1-x}$ QDs with lower bandgap transition energy were synthesized successfully. The addition of N_2H_4 led to a faster shift of the UV-vis absorption position to longer wavelength.

Fig. 5 (b) exhibits the PL QYs of the $CdSe_xTe_{1-x}$ QDs at different concentrations of N_2H_4. It can be found that the highest QYs were generally reached when the Se/(Se+Te) ratio was 0.5 in the $CdSe_xTe_{1-x}$ QDs. The bare CdSe was dominated by deep trap emission with a negligible QY of about 0.5%.[12] The increase of N_2H_4 concentration generally reduced the QY of the $CdSe_xTe_{1-x}$ QDs at the same Se composition. Simple amines like N_2H_4 can help improve the QY of the bare CdTe QDs,[47] but they may not play a similar role in synthesizing $CdSe_xTe_{1-x}$ QDs.

Fig. 5 (c) shows the PL emission peak positions of the $CdSe_xTe_{1-x}$ QDs with Se composition from 0 to 0.6. Bare CdSe QDs are not presented because no emission peak was detected. PL peaks red-shifted with the increase of N_2H_4 concentration. In this work, the emission peak positions varied from 518 nm (blue-green) to 750 nm (near-infrared) only by changing Se ratio and N_2H_4 concentration in the synthesis of $CdSe_xTe_{1-x}$ QDs.

4. Conclusions

Various sized CdTe and $CdSe_xTe_{1-x}$ alloyed quantum dots (QDs) with good luminescent properties were synthesized. Without precursor preparation and N_2 protection, CdTe and $CdSe_xTe_{1-x}$ QDs were synthesized by adding mercaptosuccinic acid (MSA), $CdCl_2$, Na_2TeO_3, Na_2SeO_3 and N_2H_4 into one pot in the open air and heating for a short time. The highest PL QY of CdTe QDs was 55% at wavelength of 573 nm and the photoluminescence quantum yield (PL QY) of the red-emitting CdTe QDs reached 25%. $CdSe_xTe_{1-x}$ QDs with emission peak position of 518 nm to 750 nm (blue-green to near-infrared) was obtained by manipulating Se ratio and N_2H_4 concentration.

It is found that a suitable N_2H_4 concentration was important to accelerate the growth of QDs to a certain level, thus improving the PL QYs of the QDs. The method reported herein is economical and facile, so it may find application in commercial synthesis of highly luminescent water-soluble QDs.

Acknowledgments

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Table of Content

High-quality CdTe and $CdSe_xTe_{1-x}$ QDs were prepared at 95 ˚C within 45 minutes with the sizes controlled by N_2H_4 concentration.