

Research Article

Time-Resolved Photoluminescence Spectroscopy Evaluation of CdTe and CdTe/CdS Quantum Dots

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CdTe and CdTe/CdS quantum dots (QDs) were prepared in aqueous solutions using thioglycolic acid as a stabilizing agent. The photoluminescence (PL) wavelength of the QDs depended strongly on the size of CdTe cores and the thickness of CdS shells. Being kept at room temperature for 130 days, the PL wavelength of CdTe and CdTe/CdS QDs was red-shifted. However the red-shifted degree of CdTe QDs is larger than that of CdTe/CdS QDs. The size of CdTe QDs and the thickness of CdS play important roles for the red-shift of PL spectra of CdTe/CdS QDs. In contrast, the full width at half maximum of PL spectra of CdTe and CdTe/CdS QDs almost remained unchanged. This is ascribed to the effects of Cd²⁺ and TGA in solutions on the growth of CdTe and CdTe/CdS QDs. This associated with the variation of surface state of the QDs during store. The results demonstrate that CdTe/CdS core/shell QDs have high stability compared with CdTe QDs due to a CdS shell.

1. Introduction

Quantum dots (QDs) have attracted considerable interest in the past two decades because of their excellent properties such as narrower and symmetric emission spectra, broad absorption spectra, and better photostability than traditional fluorescent labels [1–5]. An organic synthetic approach in trioctylphosphine or trioctylphosphine oxide at high temperature has been well developed to prepare highly luminescent II–VI QDs [6]. However, the QDs are insoluble in an aqueous solution and not compatible for biological applications. Compared with organic strategies, an alternative approach to produce water soluble QDs is to directly synthesize QDs in an aqueous solvent. Such approach is less expensive, highly reproducible, less toxic, and more biocompatible [7]. It is easy to control QD size in an aqueous approach because of a low nucleation and growth rate.

Early aqueous CdTe QDs with a low stability were prepared in the presence of thioglycolic acid (TGA) as the capping agent by using a reaction between Cd²⁺ and NaHTe solution [8]. These QDs were easily subjected to the photooxidation and photobleaching when used to labeling in living cells, resulting in significant PL quenching [9]. To

improve the photostability, epitaxial growth of an inorganic shell on the surface of CdTe QDs in aqueous solution was explored in a few research groups, similar to growing a shell of ZnS, CdS, or ZnSe on CdSe cores in organic solutions [6, 10–13]. It has been illustrated that epitaxial growth of an inorganic shell with a broader band gap on the surface of luminescent QDs is an ideal approach to improve the properties of QDs through the elimination of both the anionic and the cationic surface dangling bonds on the QDs, which leads to an enhancement of stability [14]. CdS is in principle a good candidate as the shell material for CdTe cores not only because its band gap (2.5 eV) is wider than that of CdTe (1.5 eV) but also because its lattice parameter mismatch (3.6%) to CdTe is relatively small compared with ZnSe (12.5%) and ZnS (16.5%) [10, 15, 16]. Wang and coworkers reported on the preparation of 1-thioglycerol-capped CdTe/CdS QDs by the ultrasonic irradiation [17]. Peng's group prepared TGA-capped CdTe/CdS QDs with enhanced photostability [18]. Overcoating CdS layer outside of the CdTe core has become an important stratagem to enhance the photostability of QDs.

In this work, TGA-capped CdTe and CdTe/CdS QDs have been synthesized in an aqueous solution. The structural and optical properties of CdTe and CdTe/CdS QDs were characterized and studied by transmission electron microscopy (TEM), X-ray diffraction (XRD), and optical spectroscopy. The thickness of the CdS shell was accurately controlled by changing the stoichiometry of the injection solutions and the growth condition. In particular we have performed the experiments on the thickness effect of CdS shell on the PL dynamics of CdTe/CdS QDs. Finally the storage experiment was carried out to examine the high photostability of as-prepared CdTe/CdS QDs. Systematic investigations demonstrated that the CdTe/CdS core/shell QDs revealed a prolonged average PL lifetime and good PL stability properties than CdTe core QDs. We expect that the unique optical properties of the CdTe/CdS core/shell QDs reported herein will advance understanding of CdTe QDs and will suggest new applications of QDs.

2. Material and Methods

2.1. Materials. TGA (99%), tellurium powder (Te, 99%), $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$ (98%), and sodium borohydride (NaBH_4) (96%) were purchased from Shanghai Chemical Reagents Company. Sodium sulfide ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) was supplied from Tianjin Damao Chemical Reagents Institute. The pure water was obtained from a Milli-Q synthesis system. All chemicals mentioned in the current investigations were used as received.

2.2. Synthesis of Water-Soluble TGA-Capped CdTe QDs. Aqueous TGA-capped CdTe QDs were prepared by a mild method according to the procedure reported in literature [19]. Firstly synthesize sodium hydrogen telluride (NaHTe) solution by reacting NaBH_4 with Te (5/1 molar ratio). Briefly, 1 mmol of NaBH_4 and 0.2 mmol of Te were added to a small two-neck flask and protected by N_2 for 30 min. Secondly, 0.4 mmol $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$ was dissolved in 25 mL water and 0.6 mmol TGA was added followed by adjusting pH to 11 with 1 M NaOH solution under vigorous stirring. When the mixture had been degassed by high-purity N_2 for 30 min, a certain amount of oxygen-free NaHTe solution was injected. Finally the complex solution with a faint yellow color was refluxed at 100°C for a certain time. Different-sized CdTe QDs were prepared by varying the reflux time.

2.3. Synthesis of TGA-Stabilized CdTe/CdS QDs. Synthesis of CdTe/CdS core/shell QDs is described as follows. 10 mL as-prepared CdTe colloidal solution was precipitated using 2-propanol mixed with ethanol and then redispersed in 20 mL of H_2O including 0.2 mmol of $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$ and 0.3 mmol of TGA to get solution A (PH = 11, adjust by 1 M NaOH). 0.4 mmol of $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ was dissolved in 40 mL of pure water to get solution B. Both solutions A and B were bubbled by N_2 for 1 h. When the solution A was heated to 100°C , solution B was added using an injection rate of 1 mL/min with stirring. For controlling the thickness of CdS shell, 1.5 mL and 3.5 mL of solution B were added to solution A to get different size CdTe/CdS core/shell QDs.

2.4. Characterization. All optical measurements were performed at room temperature under ambient conditions. The absorption spectra were recorded with a HITACHI U-4100 UV-vis-NIR Spectrophotometer. Integrated PL measurements were performed using a HITACHI F-4600 Fluorescence Spectrophotometer. The sizes of QDs were determined by a TEM (Hitachi H9000NA). Samples were precipitated by 2-propanol and dried in a vacuum oven for X-ray diffractometer (XRD) characterization with a Bruker D8 diffractometer. The PL lifetimes were measured using a time-correlated single-photon-counting spectrofluorometer system (Fluorocube-01, JY-IBH, Horiba). The recorded decay curves were fitted with a biexponential function by a least squares fitting method.

3. Results and Discussion

3.1. PL Properties and Crystal Structure of CdTe/CdS QDs. Figure 1 shows the PL and absorption spectra of TGA-capped CdTe cores and CdTe/CdS core/shell QDs list in Table 1 (as-prepared). By comparison, Figure 2 shows the PL and absorption spectra of TGA-capped CdTe cores and CdTe/CdS core/shell QDs list in Table 1 (130 days). Table 1 summarizes the characteristics of the optical properties of the CdTe cores and CdTe/CdS core/shell QDs. In Figures 1 and 2, the PL and absorption spectra of TGA-capped CdTe/CdS core/shell QDs have a significant red shift and the full width at half maximum (fwhm) decreased compared with CdTe cores. For example, the emission peaks (λ_{em}) of CdTe/CdS core/shell QDs shift from 541 nm for CdTe core QDs (sample A0) to 578 nm for CdTe/CdS core/shell QDs (sample A3), when the sample is as-prepared. The λ_{em} of CdTe/CdS core/shell QDs shift from 556 nm for CdTe core QDs (sample A0) to 585 nm for CdTe/CdS core/shell QDs (sample A3), when the storage time is 130 days. In addition, another important requirement for QDs is their fwhm. Increased fwhm indicates a broader particle size distribution. It is noteworthy that our CdTe/CdS emitting QDs possess an extremely narrow fwhm of the PL band ranging from 39 to 46 nm (as-prepared) and from 38 to 47 nm (130 days), which can be used directly in a chemical or biological system without any extra purification.

From the data list in Table 1, we know that the λ_{em} of sample A0 shifted from 541 (as-prepared) to 556 nm (130 days) and simultaneously the fwhm of sample A0 decreased from 40 nm to 38 nm. For the phenomena, the main reason is that in the first stage of CdTe nucleating and growing, the surface of CdTe QDs has many defects. Due to Ostwald ripening mechanism little particles dissolved and big particles grew; consequently the λ_{em} of sample A0 have a red shift and the fwhm of sample A0 has a decrease. The λ_{em} of sample A1 shift from 561 (as-prepared) to 572 nm (130 days), while the fwhm of sample A1 increases from 39 nm to 40 nm. Compared with sample A0, sample A1 has a CdS shell and better stability, which is shown in Figures 1 and 2. The red shift of λ_{em} from as-prepared to 130 days for samples A0, A1, and A2 were 15, 11, and 7 nm, respectively. The shorter red shift of CdTe/CdS core/shell QDs demonstrates better stability compared with core CdTe QDs. Groups B, C, and

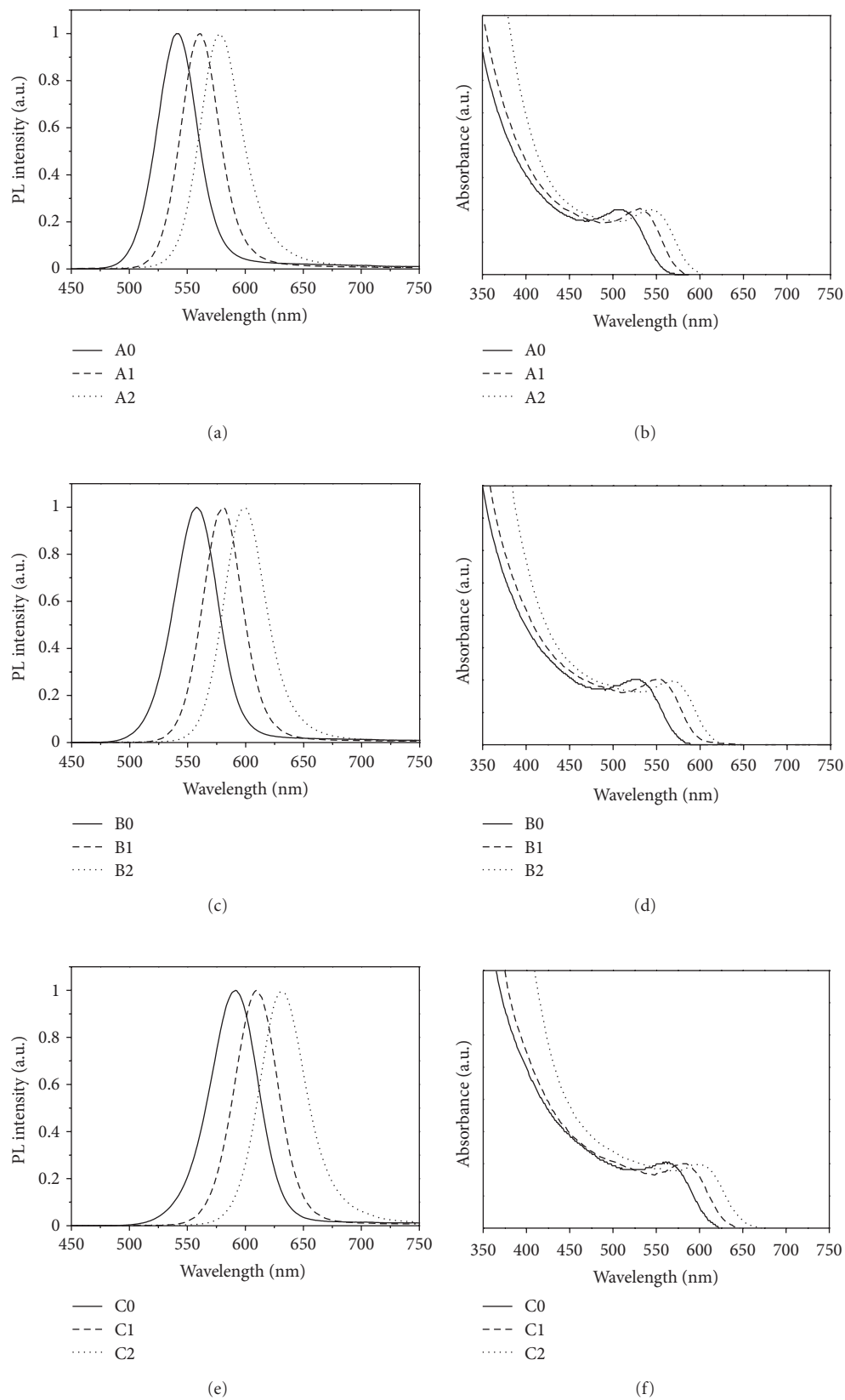


FIGURE 1: PL and absorption spectra of TGA-capped CdTe cores and CdTe/CdS core/shell QDs as shown in Table 1 (as-prepared). (a) PL spectra of samples A0, A1, and A2; (b) absorption spectra of samples A0, A1, and A2; (c) PL spectra of samples B0, B1, and B2; (d) absorption spectra of samples B0, B1, and B2; (e) PL spectra of samples C0, C1, and C2; (f) absorption spectra of samples C0, C1, and C2.

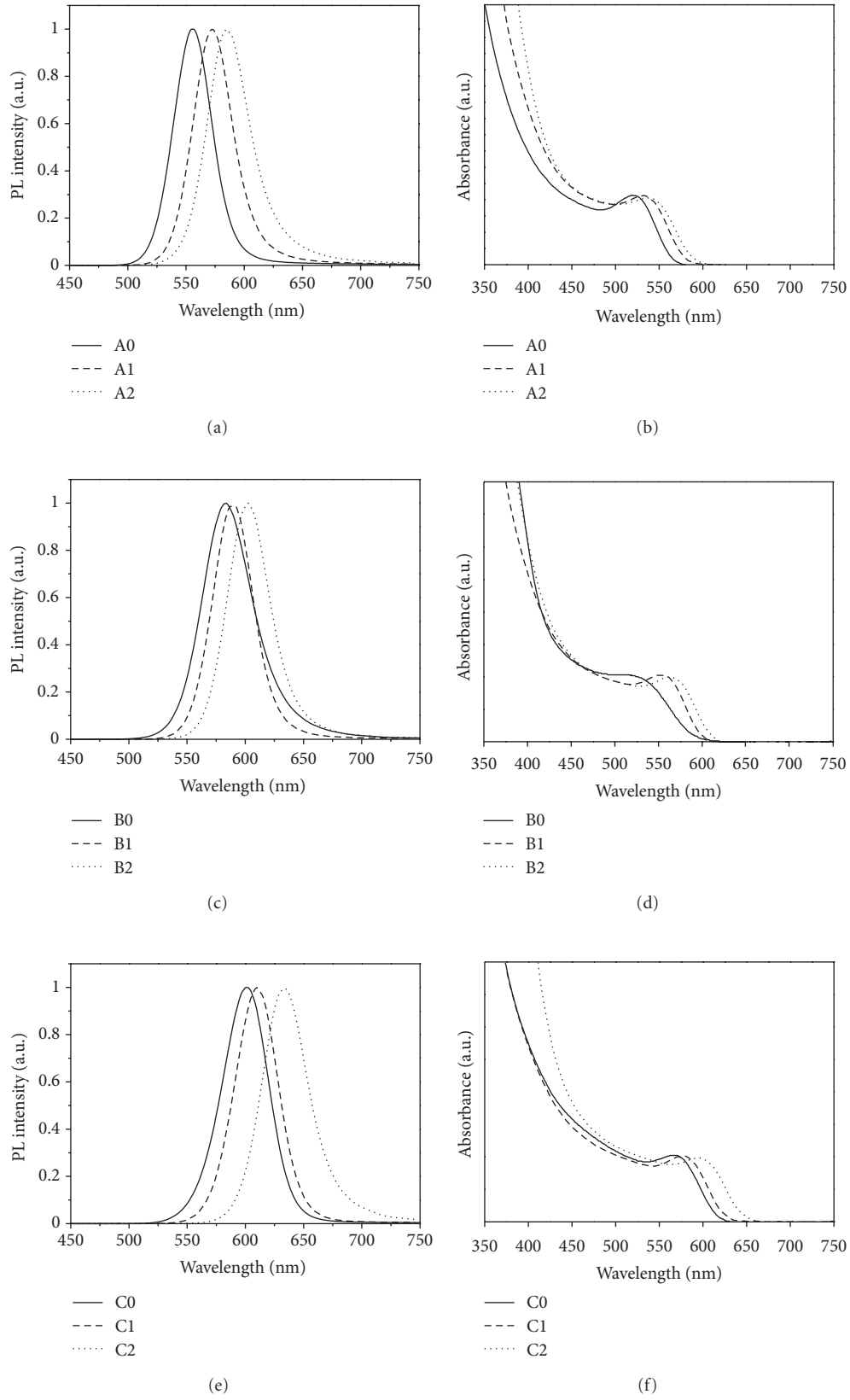


FIGURE 2: PL and absorption spectra of TGA-capped CdTe cores and CdTe/CdS core/shell QDs as shown in Table 1 (130 days). (a) PL spectra of samples A0, A1, and A2; (b) absorption spectra of samples A0, A1, and A2; (c) PL spectra of samples B0, B1, and B2; (d) absorption spectra of samples B0, B1, and B2; (e) PL spectra of samples C0, C1, and C2; (f) absorption spectra of samples C0, C1, and C2.

TABLE 1: PL properties of TGA-capped CdTe and CdTe/CdS core/shell QDs.

Sample	QD kinds	λ_{em}/nm as-prepared	fwhm/nm as-prepared	λ_{em}/nm 130 days	fwhm/nm 130 days	Red shift/nm
A0	CdTe	541	40	556	38	15
A1	CdTe/CdS	561	39	572	40	11
A2	CdTe/CdS	578	43	585	43	7
B0	CdTe	557	44	582	50	25
B1	CdTe/CdS	580	40	589	41	9
B2	CdTe/CdS	598	41	602	43	4
C0	CdTe	591	48	601	47	10
C1	CdTe/CdS	610	44	610	43	0
C2	CdTe/CdS	631	46	633	47	2

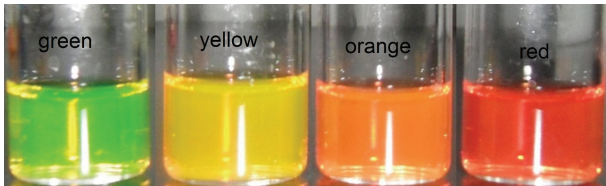


FIGURE 3: The photographs are aqueous solution of CdTe and CdTe/CdS core/shell QDs. They were taken under normal room light without using additional excitation light source. The green to red colors are from the fluorescence of CdTe and CdTe/CdS core/shell QDs.

D have analogical phenomena with group A. Sample B0 may be a special case; the red shift of λ_{em} from as-prepared to 130 days is 25 nm. The size of sample B0 has a great increase due to the slight aggregation and instability of CdTe core QDs. From the datas list in Table 1, a conclusion can be drawn that longer λ_{em} of CdTe/CdS core/shell QDs have better stability than shorter ones. For example, sample C1 has 0 nm red shift and sample C2 has 2 nm red shift, while sample B1 has 9 nm red shift and sample B2 has 4 nm red shift.

Figure 3 shows the photographs of aqueous solution CdTe and CdTe/CdS core/shell QDs. They were taken under normal room light without using additional excitation light source. The luminescent color varied from green to red. In Figure 3 the sample shows well photoluminescent properties. To confirm the CdS shell formation on the CdTe cores, Figure 4 shows the XRD patterns of CdTe cores and CdTe/CdS core/shell QD. The CdTe cores (sample C0) exhibited a diffraction pattern quite close to that of bulk cubic CdTe. The dominant structure of the core is the CdTe crystal phase. In contrast, the diffraction pattern of the CdTe/CdS QDs (sample C2) slightly moves toward higher angles with peak widths and shapes being nearly unchanged, which is close to the diffraction peak of cubic CdS. Our results are similar to the changes observed in the diffraction pattern of CdTe cores when a CdS shell grows on them [17, 20].

To characterize the quality of the samples, typical TEM images of CdTe cores and corresponding CdTe/CdS core/shell QDs with different CdS shell thickness are shown in Figure 5. Figure 5 shows the TEM images of CdTe core

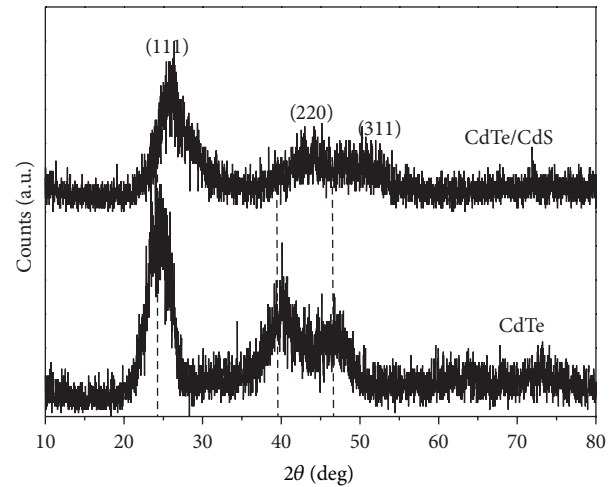


FIGURE 4: XRD patterns of TGA-capped CdTe QDs and CdTe/CdS core/shell QDs shown in Table 1. CdTe: sample C0; CdTe/CdS: sample C2. The dotted lines in figures show the cubic CdTe reflections with relative intensities.

QDs (sample C0) with an average size of 3.6 nm, CdTe/CdS core/shell QDs (sample C1) with an average size of 4.2 nm, and CdTe/CdS core/shell QDs (sample C2) with an average size of 4.7 nm. Figure 5 approximately match the theoretical thickness calculated from the amount of injected stock solution, which gives strong evidence for the epitaxial growth of CdS onto the CdTe cores and excludes the independent nucleation of CdS. It is quite evident that these QDs are close to spherical particles with excellent monodispersity.

3.2. PL Lifetime of CdTe/CdS QDs. Photoluminescence lifetime measurement is another useful approach to confirm a change in the surface state of QDs. Figure 6 shows the time-resolved luminescence decay curves of TGA-capped CdTe cores and CdTe/CdS QDs (as-prepared), and Figure 7 shows the time-resolved luminescence decay curves of TGA-capped CdTe cores and CdTe/CdS QDs (130 days): (a) luminescence decay curves of samples A0, A1, and A2; (b) luminescence decay curves of samples B0, B1, and B2; (c) luminescence decay curves of samples C0, C1, and C2. For all samples, time-resolved emission measurements have been done at

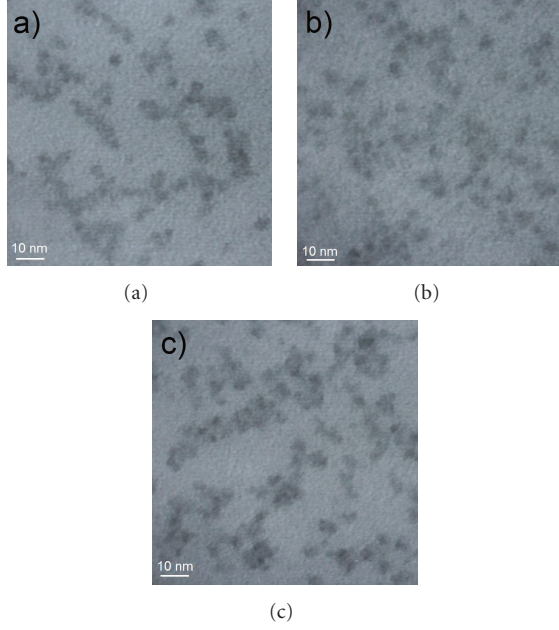


FIGURE 5: TEM images of TGA-capped CdTe, CdTe/CdS core/shell QDs: (a) sample C0 (3.6 nm); (b) sample C1 (4.2 nm); and (c) sample C2 (4.7 nm).

their peak wavelengths in steady state PL spectra. The decay curves can be well fitted to a biexponential model described by the equation as follows:

$$F(t) = A + B_1 \exp\left(-\frac{t}{\tau_1}\right) + B_2 \exp\left(-\frac{t}{\tau_2}\right), \quad (1)$$

where τ_1 and τ_2 ($\tau_1 < \tau_2$) represent the time constants, and B_1 and B_2 represent the amplitudes of the fast and slow components, respectively. Average lifetime τ was calculated using the equation below [21]:

$$\tau = \frac{(B_1 \tau_1^2 + B_2 \tau_2^2)}{(B_1 \tau_1 + B_2 \tau_2)}. \quad (2)$$

The fitted values of the parameters B_1 , B_2 , τ_1 , τ_2 , and τ are summarized in Table 2 (as-prepared) and Table 3 (130 days).

In Figures 6 and 7, Tables 2 and 3, a CdS shell coating makes CdTe/CdS QDs a long lifetime compared to the CdTe cores due to drastic increase in the charge carrier in the core/shell structure. With increasing the shell thickness, the average lifetime of the core/shell QDs was also prolonged. The spatial separation of electron and hole can result in a decrease of the wave function overlap and thus a longer lifetime of the charge recombination is expected in type II heterostructures. For epitaxial growth of the shell over the core, it is very important to have less lattice mismatch between core and shell material.

In Table 2, the lifetime of sample B0 has a significant increase from as-prepared to 130 days, this phenomenon is ascribed to the increased surface defects, in which the defects led the lifetime to be prolonged. To the contrary, the lifetimes of samples A0 and C0 have a slight decrease, from

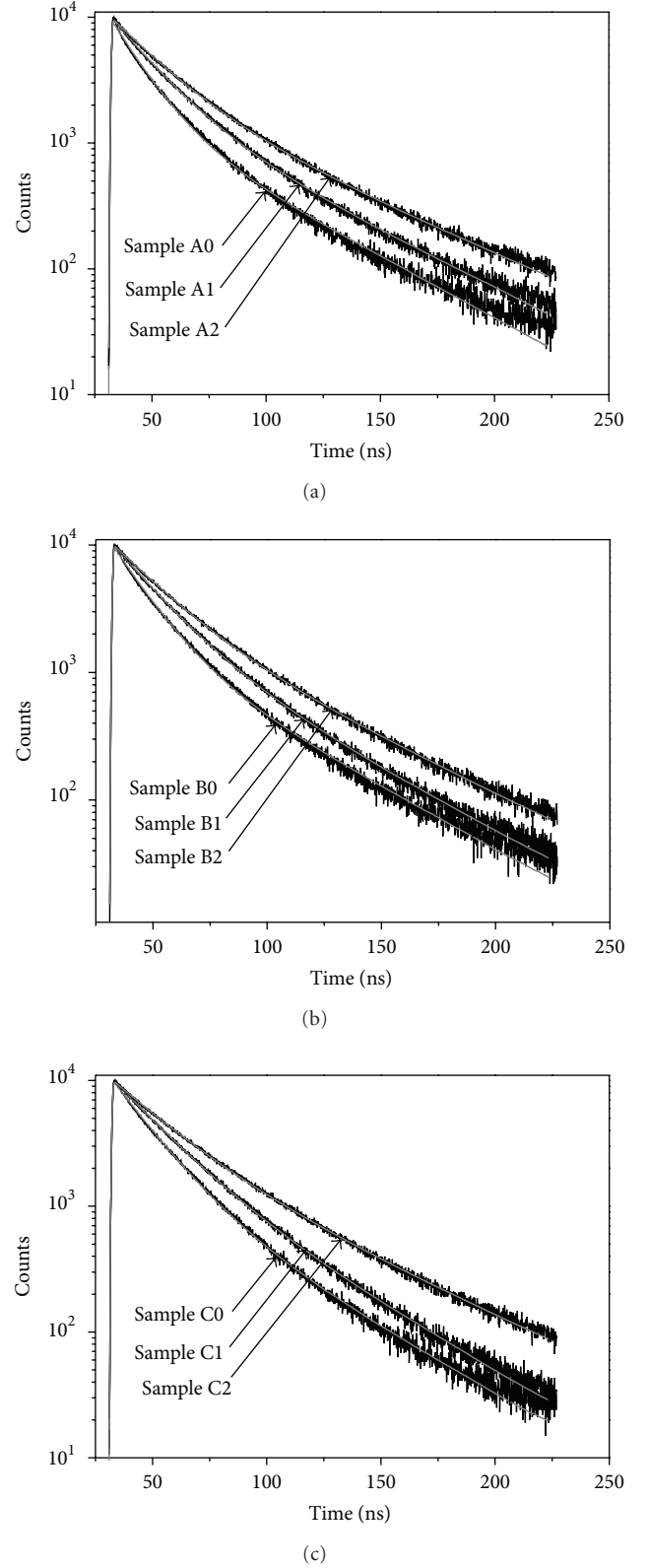


FIGURE 6: Time-resolved luminescence decay curves (measured at maximum emission peak, $\lambda_{ex} = 370$ nm) of TGA-capped CdTe, and CdTe/CdS core/shell QDs (as-prepared). (a) samples A0, A1, and A2; (b) samples B0, B1, and B2; (c) samples C0, C1, and C2. Reproduced curves for data shown in Table 2 are plotted as thin gray lines.

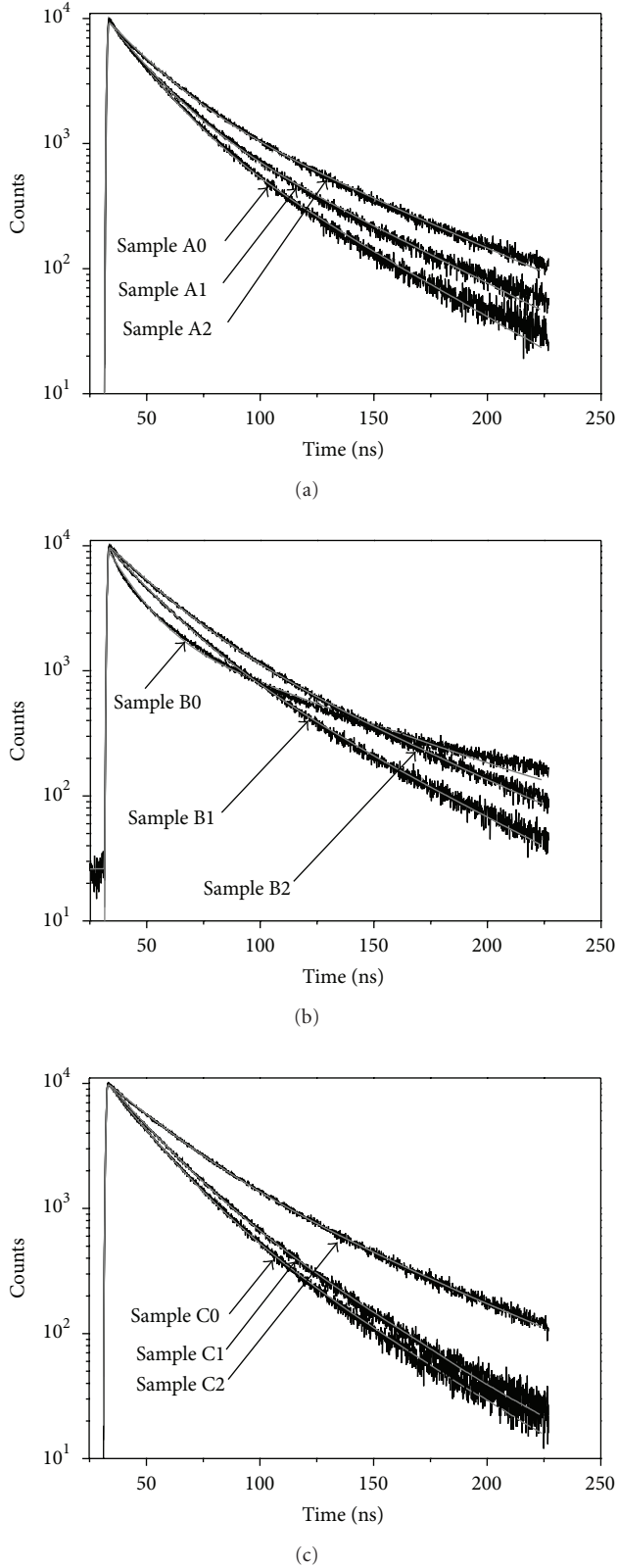


FIGURE 7: Time-resolved luminescence decay curves (measured at maximum emission peak, $\lambda_{\text{ex}} = 370$ nm) of TGA-capped CdTe, and CdTe/CdS core/shell QDs (130 days). (a) samples A0, A1, and A2; (b) samples B0, B1, and B2; (c) samples C0, C1, and C2. Reproduced curves for data shown in Table 3 are plotted as thin gray lines.

TABLE 2: Time constants τ_1 and τ_2 , components B_1 and B_2 , and average lifetime τ of as-prepared TGA-capped CdTe, and CdTe/CdS core/shell QDs (samples shown in Table 1 (as-prepared)).

Sample	B_1	τ_1/ns	B_2	τ_2/ns	τ/ns
A0	56.73	12.99	43.27	43.26	34.7
A1	56.21	17.37	43.79	47.58	37.9
A2	50.33	20.09	49.67	54.77	45.4
B0	59.65	14.26	40.35	42.80	33.4
B1	61.38	18.54	38.62	45.35	34.8
B2	52.90	21.01	47.10	51.06	41.6
C0	66.91	16.44	33.09	41.09	30.1
C1	63.15	20.24	36.85	42.14	32.3
C2	51.92	23.36	48.08	52.87	43.3

TABLE 3: Time constants τ_1 and τ_2 , components B_1 and B_2 , and average lifetime τ of TGA-capped CdTe, and CdTe/CdS core/shell QDs (samples shown in Table 1, stored at room temperature for 130 days).

Sample	B_1	τ_1/ns	B_2	τ_2/ns	τ/ns
A0	57.12	15.32	42.88	40.53	32.1
A1	49.93	15.86	50.07	47.01	39.2
A2	44.89	18.46	55.11	55.05	47.2
B0	38.52	11.91	61.48	62.93	57.5
B1	55.48	18.60	44.52	45.24	36.2
B2	51.12	21.30	48.88	53.58	44.1
C0	62.62	17.01	37.38	37.16	28.4
C1	55.99	17.97	44.01	37.68	30.2
C2	51.42	24.13	48.58	57.39	47.2

34.7, 31.0, 30.1 ns (0 day) to 32.1, 28.4, 28.4 (130 days); this phenomenon is due to the decreased surface defects caused by light etching. The lifetime of most CdTe/CdS core/shell QDs (sample A1, A2, B1, B2, and C2) increased from as-prepared to 130 days, which is corresponding with the red shift of λ_{em} from as-prepared to 130 days. With increasing the time of storage time, the average lifetime of the core/shell QDs was also prolonged. For Ostwald ripening mechanism, the sizes of CdTe/CdS core/shell QDs increased, which give rise to the increase of the charge separation in the CdTe/CdS core/shell QDs. It is evident that the average PL decay times increase steadily with increasing size of the QDs, a tendency that is generally observed for II–VI QDs [22].

4. Conclusion

TGA-capped CdTe/CdS core/shell QDs in an aqueous solution were synthesized by depositing a CdS shell on CdTe cores in an aqueous solution. The epitaxial growth of the CdS shell was confirmed by the red-shifted absorption and emission spectra of the CdTe/CdS core/shell QDs and the XRD patterns of the QDs. The CdTe cores and CdTe/CdS core/shell QDs exhibited tunable PL peak wavelengths from 541 to 631 nm for as-prepared synthesized samples (as-prepared) and from 556 to 633 nm for a storage time of 130 days, respectively. The red-shifted degree of the CdTe cores is larger

than that of the CdTe/CdS core/shell QDs. It proved that CdTe/CdS core/shell QDs have better PL properties than CdTe cores. Systematic investigations of PL lifetime demonstrated that the CdTe/CdS core/shell QDs revealed a prolonged average PL lifetime and PL stability compared with CdTe cores. The research on the PL stability of stored CdTe and CdTe/CdS core/shell QDs has a significant meaning for the application of QDs.

Acknowledgments

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