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Effect of Mercaptocarboxylic Acids on Luminescent Properties of CdTe Quantum Dots

Zhimin Yuan · Aiyu Zhang · Yongqiang Cao · Jie Yang · Yuanna Zhu · Ping Yang

Abstract CdTe quantum dots (QDs) were prepared in an aqueous solution using various mercaptocarboxylic acids, such as 3-mercaptopropionic acid (MPA) and thioglycolic acid (TGA), as stabilizing agents. The experimental result indicated that these stabilizing agents played an important role for the properties of the QDs. Although both TGA and MPA-capped CdTe QDs exhibited the tunable photoluminescence (PL) from green to red color, the TGA-capped QDs revealed a higher PL quantum yield (QY) up to 60% than that of MPA-capped QDs (up to 50%) by using the optimum preparation conditions, such as a pH value of ~11.2 and a TGA/Cd molar ratio of 1.5. PL lifetime measurements indicate that the TGA-capped QDs exhibited a short average lifetime while the MPA-capped QDs revealed a long one. Furthermore, the average lifetime of the TGA-capped QDs increased with the increase of the QDs size, while a decreased lifetime for the MPA-capped QDs was obtained. This means that the PL lifetime depended strongly on the surface state of the CdTe QDs. These results should be utilized for the preparation and applications of QDs.

Keywords CdTe · Quantum dots · Photoluminescence · Lifetime · Capping agent

Abbreviations

QDs	Quantum dots
MPA	3-mercaptopropionic acid
TGA	Thioglycolic acid
PL	Photoluminescence
QY	Quantum yield
FWHM	Full width at half maximum

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Introductions

Quantum dots (QDs), as an important material both in academic researches and in technical applications, was studied extensively in the past few decades, due to their unique optical properties, such as size-tunable photoluminescence (PL), high PL quantum yield (PLQY), narrow emission spectra, and high photostability [1]. As the initial fabrication method for QDs, the synthetic routes in highly boiling organic solvents were used extensively, and the QDs with the superior PL properties, such as the higher PLQY and narrower emission peak, were obtained [2]. However, the insolubleness in water for oil-phase QDs greatly impedes their application in the areas, such as biodetection, biosensing, and so on. Therefore, the direct aqueous synthesis of QDs by employing the different stabilizing agents are exploited and provides a useful alternative to widely used synthetic routes in oil phase [3].

Many researches upon the QDs synthesis indicated that the surface chemistry of QDs, especially the capping molecules, is a key factor for the synthesis of QDs with the excellent properties for promoting further progresses of nanoscience and nanotechnology [4–8]. First, the capping molecules can function as passivating agents for surface states. Second, the growth kinetics of the QDs is determined by the affinity of the precursors for the capping molecules and the stability of the capping ligand precursor intermediate complex. Third, the capping molecules prevent the QDs from aggregation via sterical hindrance or by charge stabilization [2, 8, 9]. Thus, the effect of capping agents on the properties of QDs has become a significant research field. Recently, zinc-blende CdTe QDs, a typical emissive II-VI group semiconductor QDs, has been synthesized by various aqueous synthetic approaches, using the thioglycolic acid (TGA), 1-thioglycerol, mercaptoethylamine,

or L-cysteine as the stabilizing agents[10, 11]. These studies suggested that the capping agents had an important effect on the properties of CdTe QDs. For example, Wuister and co-workers prepared water-soluble, thiol-capped CdTe QDs with a high PL efficiency of 60% and a monoexponential PL decay, demonstrating that the capping exchange with thiols has a beneficial effect on the PLQY for CdTe QDs [12]. They also further reported the remarkable difference between the luminescence behavior of CdTe and other QDs upon binding to different thiol molecules [13]. Rogach et al. found that, in contrast to CdTe QDs capped by TGA, mercapto-propionic acid (MPA)-capped CdTe QDs showed up to 1 order of magnitude longer (up to 145 ns) emission decay times [3]. But up to now, some effect mechanisms of different thiol capping agents are not very clear, and more experiments are still required in order to understand the influence of the capping agent on the PL properties of the CdTe QDs.

In order to further uncover the effect of different thiol molecules as the capping agent on the properties of CdTe QDs, in the present work, we prepared CdTe QDs capped with the TGA or MPA respectively through a similar aqueous synthesis, and investigated their optical properties by the UV–vis absorption, PL emission, and time-resolved PL emission measurments. The results revealed that, besides a common property of the tunable PL peak wavelength with the increasing sizes for both two CdTe QDs, the PL properties were strongly depended on the capping agents. Compared with MPA-capped CdTe QDs, TGA-capped ones exhibited the higher PLQY, narrower emission spectra, but a short average PL lifetime and a slow growth rate. The capping agent dependence of the emission profile enables us to propose a fine structure of the exciton states and a complicated trap-related state in CdTe QDs.

Materials and Method

Chemicals

All chemicals were used without additional purification. TGA (99%), Tellurium powder (99%), $CdCl_2 \cdot 2.5H_2O$ (98%), and $NaBH_4$ (96%) were all purchased from Shanghai Chemical Reagents Company (China). MPA (99%) was supplied by Alfa Aesar. Rhodamine 6G was purchased from Aldrich. The pure water ($\rho > 18$ $M\Omega \cdot cm^{-1}$) was obtained from a Milli-Q synthesis system.

Synthesis of CdTe QDs

Water-soluble TGA- and MPA-capped CdTe QDs were prepared by a mild method. Briefly, by a molar ratio of 5:1 for $NaHB_4$/Te, sodium borohydride was used to react with

tellurium in water at 60 °C to facilely prepare a sodium hydrogen telluride (NaHTe) soultion. Fresh solutions of NaHTe were prepared for further use. For the preparation of TGA-capped CdTe QDs, $CdCl_2 \cdot 2.5H_2O$ (0.4 mmol) and TGA (0.6 mmol) were dissolved in 25 mL water to get a precursor solution followed by adjustment to a pH value of 11.2 by the 1 M NaOH solution. This pH value is crucial for getting a high PLQY. After the precursor solution was bubbled by N_2 for 30 min, a fresh deoxygenated NaHTe solution was added in the above precursor solution with stirring. The typical molar ratio of Cd:Te:TGA was 1:0.5:1.5. The mixture was subsequently refluxed for different time to synthesize the TGA-capped CdTe QDs with the different sizes. The MPA-capped CdTe QDs was prepared using a similar preparation procedure as the TGA-capped QDs. The molar ratios of Cd/MPA are same as those of Cd/TGA. It was worth noted that the preparation of precursor solution of Cd^{2+} and MPA need a long stirring time (1 h) compared with that of Cd^{2+} and TGA precursor solution (0.5 h).

Apparatus

The absorption and PL spectra were recorded using conventional spectrometers (Hitachi U-4100 and F-4600, respectively). The PLQY of CdTe QDs at room temperature was estimated by comparison with Rhodamine 6G in an ethanol solution assuming its PLQY as 95% [14, 15]. 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 multiexponential function by a least squares fitting method.

Results and Discussion

It is well known that Cd^{2+} ions in an aqueous solution can combine with TGA to form complex. Eychmüller's group reported that the composition of the Cd-TGA complex depends on the initial concentrations of Cd^{2+} and TGA in the water and that the concentrations of three types of Cd-TGA complexes (Cd^{2+} connected with 1, 2, and 3 ligands, respectively) depend on the pH of the solution [16].To investigate the effect of TGA/Cd molar ratio on the properties of CdTe QDs, the molar ratio of TGA/Cd was adjusted in a range of 2.4 to 1.1. The result indicated that CdTe QDs exhibited a maximum PLQY when the TGA/Cd molar ratio of 1.5 was used. In the case of a TGA/Cd molar ratio of 1.5, the Cd-TGA complex has one or two ligands. The amount of monothio complex was dominant (We note that the solution of Cd precursors at low TGA/Cd ratios (less than 1.3) can be slightly turbid, which is an additional

indirect evidence of the domination of the uncharged and less-soluble Cd-SR complex). A large amount of monothio complexes existing in precursors directly lead to increasing concentration of free Cd^{2+} ions, which would play an important role in the nucleation and growth of CdTe QDs. Gradual dissolution of the Cd-SR complex during QDs growth could provide a constant transport rate of Cd ions to the particles. A slow flux of the cadmium precursor provides the possibility of growing the QDs under diffusion control, which, as has been predicted theoretically, is preferable for narrowing the size distribution and may be a key factor for the dynamic improvement of the surface quality of the growing QDs [16]. To the best of our knowledge, the QDs with high PLQY possessed fewer surface defects, suggesting that it was a favorable structure from a thermodynamic point of view. If the QDs grew under conditions of the equilibrium of growth and dissolution, the favorable structure was more likely to be obtained.

Generally, the growth procedure of CdTe QDs could be observed by their absorption and PL spectra. Since the absorption and PL peak wavelength was related to the size of semiconductor QDs, the red-shift of the adsorption and PL peak with the prolonged reflux time implied that the bigger sizes of the CdTe QDs were formed as the reflux was proceeding. Figure 1 shows the absorption and PL spectra of TGA-capped CdTe QDs with tunable PL peak wavelength. A significant red-shift of emission was created with increasing reflux time, e.g. PL peak wavelength was at 525 nm for green-emitting CdTe QDs while for red-emitting ones was at 630 nm, and at the same time their first absorption peak was shifted from 494 to 600 nm. The similar stokes shift indicated there are the same electron–phonon coupling in the interior of the green and red-
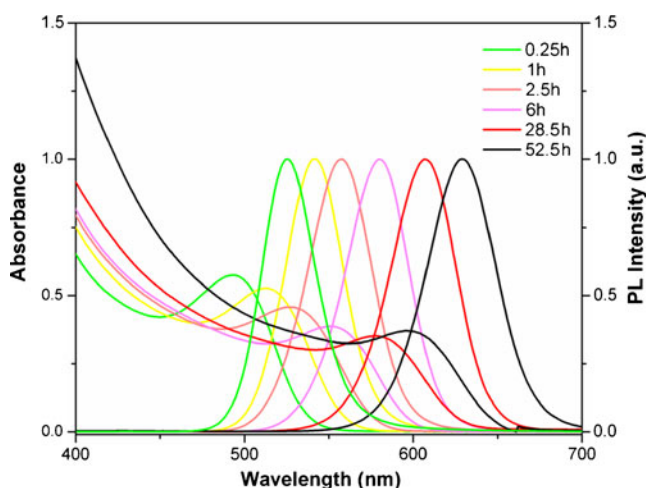
emitting QDs [17]. As a result, a strong luminescence band is observed near the band edge and no deep trap luminescence is detected, indicating the decent emissive properties of the QDs [18–20].

For biolabeling and imaging applications, narrow PL spectra together with high PLQY make the QDs a special interest. Figure 2 shows the full width at half maximum (FWHM) of PL spectra and PLQY of TGA-capped CdTe QDs versus reflux time. It can be seen that the FWHM of the PL spectra showed a significant increase in the first stage (reflux time ≤6 h) and then it entered to plateau phase within the succent reflux of 20 h, and subsequently a gentle increase was observed. The PLQY of the QDs similarly showed a rapid increase in the first 6 h of reflux and reached a maximum value up to 60%, and then dramatically decreased with the reflux. The detailed results were illustrated in Table 1, including the QDs diameters which were estimated based on the reported extinction coefficient of the first absorption peak and its wavelength dependences [21]. The increased QDs diameters were consistent with the successive red-shift of PL peak.

Due to the larger amount of dangling bonds and lattice defect existing in the QDs surface caused by the immense surface-to-volume ratio of QDs, most of luminescence quenching processes occur at the nanocrystal surface. However, the capping molecules dynamically absorbed on the QD surface could passivate these defections and prevent this nonradiative recombination. For the purpose of uncovering the influence of different capping molecules on QDs properties, MPA was used as a capping agent to prepare CdTe QDs with a MPA/Cd molar ratio of 1.5 for comparison. Figure 3 shows the absorption and PL spectra of MPA-capped CdTe QDs with tunable absorption and PL peak wave-
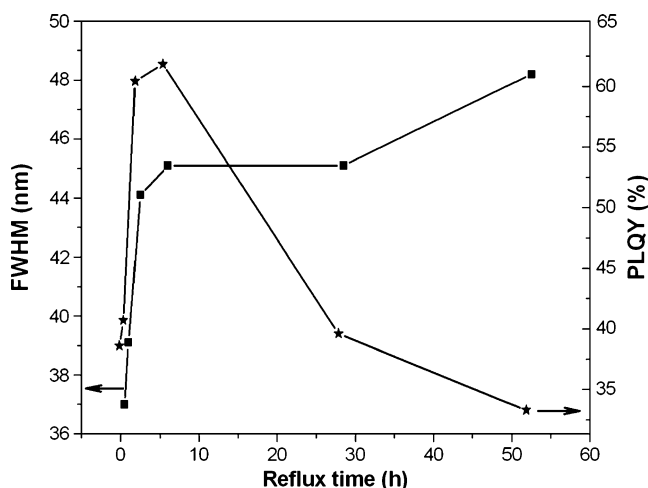

Fig. 1 Absorption and PL spectra of TGA-capped CdTe QDs. Reflux time of the QDs is shown in figure. The first absorption peak and PL peak wavelength of the QDs revealed a red shift with reflux time

Fig. 2 Full width at half maximum of photoluminescence spectra and photoluminescence quantum yield of TGA-capped CdTe QDs versus reflux time

Table 1 Preparation condition and properties of TGA and MPA-capped CdTe QDs

Reflux time (h)	Capping agent	PL peak wavelength (nm)	FWHM (nm)	PLQY (%)	Diameter (nm)
0.25	TGA	524	37	38.6	2.20
1.0	TGA	541	39.5	40.7	2.70
2.5	TGA	553	44.1	60.4	2.90
6.0	TGA	580	45.1	34.2	3.30
28.5	TGA	606	45.1	39.6	3.48
52.5	TGA	630	48.2	33.3	3.59
0.5	MPA	512	45.6	31.2	1.70
1.0	MPA	533	47.9	38.4	2.40
2.0	MPA	553	52.8	41.5	2.68
4.0	MPA	578	63.5	50.1	3.10
5.5	MPA	594	65.9	48.8	3.21
7.0	MPA	605	64.7	35.3	3.32
17.5	MPA	650	78.9	26.3	3.63
21.5	MPA	664	83.9	23.9	3.70

FWHM is the full width at half maximum of photoluminescence spectra. PLQY is photoluminescence quantum yield of CdTe QDs

length. Figure 4 shows the FWHM of PL spectra and PLQY of MPA-capped CdTe QDs against reflux time. As we can see that, it is different from the case of TGA-capped CdTe the FWHM of MPA-capped keep a relatively high speed of increase at all times except for a gentle slope in the period of 4–7 h; however, the PLQY of QDs significantly increased in the first 4 h and then dropped rapidly with the continuously refluxing, which is similar with the TGA-capped case.

Table 1 summarizes the detailed PL parameters and diameters of TGA- and MPA-capped CdTe QDs. Compared with TGA-capped case, MPA-capped ones exhibited a larger red-shifted PL peak (i.e. a larger diameter of QDs) with the increase of reflux time. The PL peak wavelength was from 512 nm to 664 nm when the reflux time became

into 21.5 h from 0.5 h. Moreover, the MPA-capped CdTe revealed a broader PL emission band (FWHM of 45.6 to 83.9 nm) and a lower PLQY (up to 50%). Because of carboxyl groups on the surface of TGA- and MPA-capped CdTe QDs, they are utilizable for biolabeling. For example, the carboxyl groups on the surface can be conjugated with amino group in biomolecules. Compared with the MPA-capped QDs, the TGA-capped QDs are much useful because of their high PLQY.

The high growth speed of MPA-capped CdTe could be ascribed to the poor complex stability of MPA and Cd^{2+} ions to that of TGA and Cd^{2+} ions. This poor stability led the complex of MPA and Cd^{2+} ions could release more monomers for the fast growth of CdTe QDs. And often, the faster the QDs grow, the more defects and
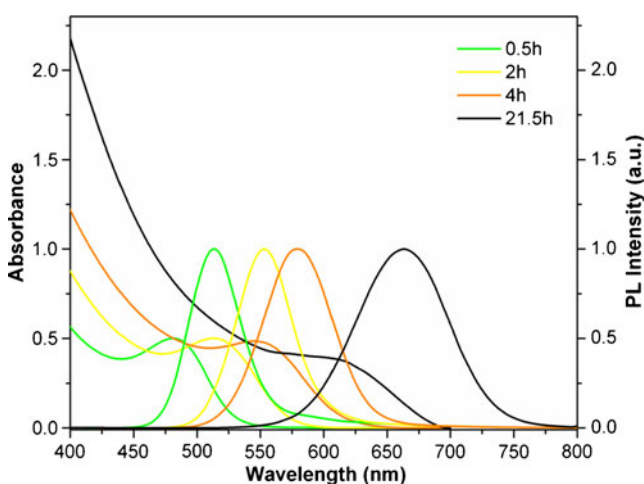

Fig. 3 Absorption and PL spectra of MPA-capped CdTe QDs. Reflux time of the QDs is shown in figure. The first absorption peak and PL peak wavelength of the QDs revealed a red shift with reflux time
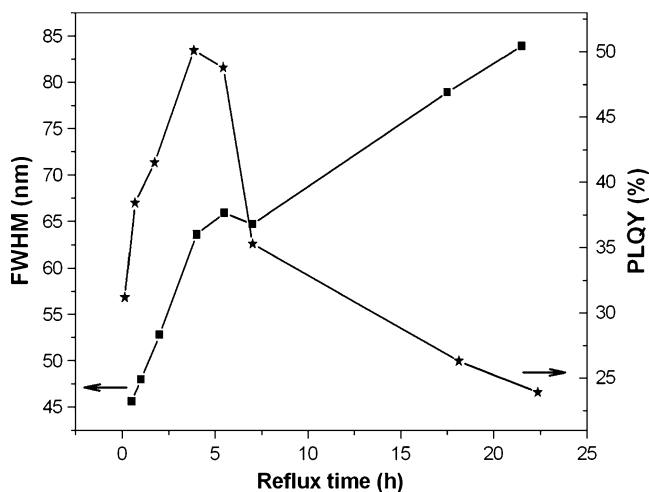

Fig. 4 Full width at half maximum of photoluminescence spectra and photoluminescence quantum yield of MPA-capped CdTe QDs versus reflux time

trap states would generate. As a result, the MPA-capped CdTe QDs showed only a maximum PLQY value of about 50%, even lower than that of TGA-stabilized CdTe QDs. The above results also suggested that the complex reaction might greatly affect the growth of CdTe QDs and their properties. The growth kinetics of QDs is determined by the affinity of the precursors for the capping molecules and the stability of the capping ligand precursor intermediate complex. If the affinity with the capping molecules is too weak, the control over the crystal growth is poor, resulting in uncontrolled growth and formation of large particles. If the complex is too stable, no QD formation occurs. Therefore, it was also approved that complex reactions between ligands and monomers were important to the growth kinetics of CdTe QDs in aqueous solution. In MPA-stabilized system, the poorer stability of thiol-Cd complexes resulted in the quick growth of QDs and the increase of surface defects. This poorer stability also led to the formation of larger QDs with longer PL peak wavelength. Thus, the use of MPA as a stabilizer enables us to obtain CdTe QDs larger than 3.7 nm, which is hardly possible for TGA-capped CdTe QDs, offering a facile synthesis method to get large size CdTe QDs.

PL lifetime measurement is another useful approach to confirm a change in the surface state (e.g. ligand exchange) of QDs [22]. Figure 5 shows the time-resolved luminescence decay curves of TGA-capped CdTe QDs, which can be satisfactorily fitted into biexponential function, which has been observed frequently for different kinds of II–VI nanocrystals. The decay curves can be well fitted to a biexponential model described by $F(t)=A+$
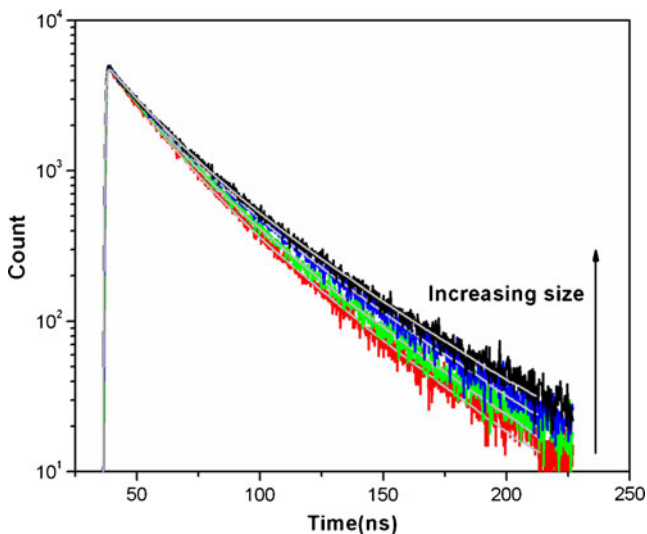
Table 2 Time constants τ_1 and τ_2, components B_1 and B_2, and average lifetime τ of TGA and MPA-capped CdTe QDs

Reflux time (h)	Capping agent	τ_1(ns)	τ_2(ns)	B_1	B_2	τ(ns)
2.5	TGA	16.6	34.7	50.08	49.92	28.8
6.0	TGA	17.1	35.9	46.81	53.19	30.3
28.5	TGA	17.5	37.6	41.47	58.53	32.6
52.5	TGA	17.8	40.4	41.38	58.62	35.0
4.0	MPA	13.4	63.1	34.22	65.78	58.2
7.0	MPA	15.7	57.8	36.58	63.42	52.2
17.5	MPA	15.2	54.1	34.64	65.36	49.2
21.5	MPA	14.5	49.2	22.78	77.22	46.4

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

$B_1 exp\ (-t/\tau_1)+B_2\ exp\ (-t/\tau_2)$, 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 $\tau = (B_1\tau_1^2 + B_2\tau_2^2)/(B_1\tau_1+B_2\tau_2)$ [21, 23]. The fitted values of the parameters B_1, B_2, τ_1, τ_2, and τ are summarized in Table 2. The fast component of the PL decay in the initial CdTe QDs is associated with exciton recombination. The slow component is considered to originate from the surface-related emission of the CdTe QDs. The fast component (B_1) of PL decay for the QDs decreased with reflux time while the slow component (B_2) increased. In addition, the average PL decay lifetime of the QDs increased with the increase of reflux time.

The PL lifetime of semiconductor QDs depends strongly on their composition, size, structure, and surface state
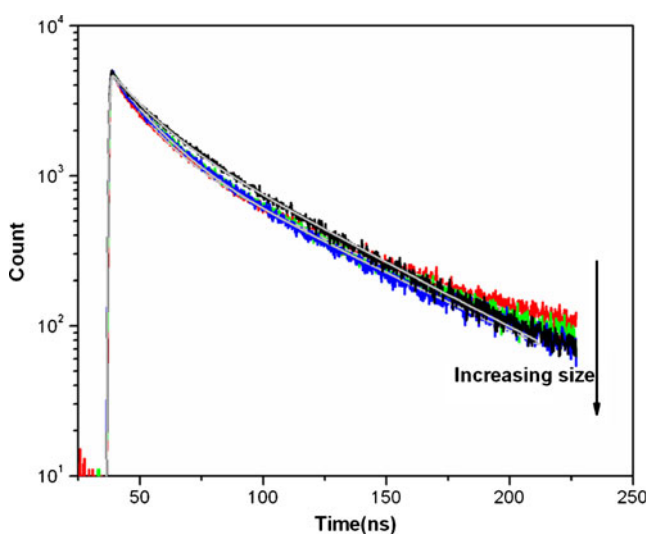
Fig. 5 Time-resolved luminescence decay curves (measured at maximum emission peak, $\lambda_{ex}=370$ nm) of TGA-capped CdTe QDs. Reproduced curves for data shown in Table 2 are plotted as thin white lines

Fig. 6 Time-resolved luminescence decay curves (measured at maximum emission peak, $\lambda_{ex}=370$ nm) of MPA-capped CdTe QDs. Reproduced curves for data shown in Table 2 are plotted as thin white lines

which also affected PLQY. In our case, CdTe QDs (2.5 nm in diameter, 2.5 h reflux time, shown in Table 1) with a TGA capping agent had an average lifetime of 28.8 ns and exhibited biexponential decay. However, when trioctylphosphine and trioctylphosphine oxide were used as the capping agents, other CdTe QDs (2.6 nm in diameter) had an average lifetime of 8 ns and exhibited single-exponential decay [24]. Furthermore, the lifetime of semiconductor core-shell QDs is also affected by the composition, morphology, and thickness of the shell. In this, due to a long reflux time, TGA-capped CdTe QDs would reveal a CdTe-CdS core-shell-like structure which resulted in the increased average lifetime.

In contrast, Fig. 6 shows the time-resolved luminescence decay curves of MPA-capped CdTe QDs, which can also be satisfactorily fitted into biexponential function. The fitted values of the parameters B_1, B_2, τ_1, τ_2, and τ are summarized in Table 2. As a result, the average lifetime of the QDs decreased with reflux time even though the fast component (B_1) of PL decay decreased with reflux time while the slow component (B_2) increased. This is ascribed to the effect of MPA on the structure and properties of the QDs.

The observed difference in PL decay curves for TGA and MPA-capped CdTe QDs could be caused by a different degree of surface passivation of the CdTe core by these two ligands. In our experiments, TGA-capped CdTe QDs exhibited a short average lifetime compared with MPA-capped CdTe QDs of similar size (e.g. emitting at similar wavelength). This is ascribed to the different surface state of two kinds of QDs because MPA has an additional -CH$_2$-group [25].

Conclusion

An optimal aqueous synthesis makes thiol-capped CdTe QDs with high PLQY (up to 60%), tunable PL peak wavelength from 520 to 660 nm, and narrow PL band (the FWHM of PL spectra of 40–50 nm). Compared with TGA-capped CdTe QDs, MPA-capped ones exhibited long average lifetime, large red-shifted PL peak wavelength with reflux time, low PLQY, and broad PL spectra. This phenomenon is ascribed to the effect of two kinds ligands on the growth kinetics and surface passivation degree of the QDs. Brightly emitting water-soluble CdTe QDs with a flexible surface chemistry determined by easy choice of thiol capping ligands have already secured and will secure in the future for a wide field of applications, ranging from life sciences to photonics and optoelectronics. Here CdTe QDs with well-defined lifetimes through the control of the size and quantum efficiency can play a crucial role.

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