

One-Pot Synthesis of Highly Luminescent CdTe Quantum Dots by Microwave Irradiation Reduction and Their Hg²⁺-Sensitive Properties

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ABSTRACT

A facile one-pot microwave irradiation reduction route has been developed for the synthesis of highly luminescent CdTe quantum dots using Na₂TeO₃ as the Te source in an aqueous environment. 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 Cd²⁺, have considerable influence on the particle size and photoluminescence quantum yield of the CdTe quantum dots. The photoluminescence quantum yield of CdTe quantum dots prepared using relatively short reaction times (10–40 min) reached 40%–60% (emission peaks at 550–640 nm). Furthermore, the resulting products could be used as fluorescent probes to detect Hg²⁺ ions in aqueous media. The response was linearly proportional to the concentration of Hg²⁺ ion in the range 8.0×10⁻⁹ mol/L to 2.0×10⁻⁶ mol/L with a detection limit of 2.7×10⁻⁹ mol/L.

KEYWORDS

CdTe, quantum dots, microwave irradiation, fluorescent, sensor, mercury

Introduction

Quantum dots (QDs), due to their variety of size-and shape-dependent optical and electrical properties [1, 2], have been attracting extensive attention for numerous applications such as in biological labels, photovoltaic devices, and optoelectronic devices [3–5]. In particular, they are very attractive as QD-based optical sensors for detecting biomolecules, small molecules, and cations or anions due to their high fluorescence quantum yields, good photostability, and negligible photobleaching in comparison with dyes [6–10]. CdTe is an important semiconductor material and CdTe QDs show increasing promise due to their large exciton Bohr radius (7.3 nm) and narrow

bulk band gap of 1.475 eV. CdTe QDs have been widely studied as luminescence probes and sensors [11–14]. However, their poor photoluminescent properties may hinder their applications as sensors. To date, two rudimentary approaches have been reported for the synthesis of CdTe QDs. One is the organometallic route [15–18]. The as-prepared QDs are generally capped with hydrophobic ligands, and hence cannot be directly employed in bioapplications. The other is the aqueous synthesis route, producing QDs with excellent water solubility, biological compatibility, and stability [19]. However, due to the long reaction times (from several hours to several days) in aqueous phase, a large number of surface defects are often generated, which results

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in low photoluminescence quantum yield (PLQY). To synthesize high-quality CdTe QDs rapidly, both hydrothermal and microwave irradiation methods have been utilized instead of traditional reflux methods [20–24]. Compared with conventional heating techniques, rapid homogeneous heating can be realized through the penetration of microwaves, which allows the concentration of surface defects of the QDs to be minimized and produces uniform products with high QY. NaHTe, an unstable compound under ambient conditions, has usually been utilized as the tellurium precursor for synthesis of CdTe QDs or Te nanostructures [20–25]. The synthesis of CdTe QDs generally has to be performed in inert reaction systems. In this paper, we report the synthesis of CdTe QDs using sodium tellurite (Na_2TeO_3) as the Te source and 3-mercaptopropionic acid (MPA) as stabilizer via a facile one-pot microwave irradiation reduction route. Since Na_2TeO_3 is air-stable, all of the operations were performed in air, avoiding the need for an inert atmosphere. The synthetic pathway is thus free of complicated vacuum manipulations and environmentally friendly. The growth of CdTe QDs should be rapid due to the microwave dielectric heating and it is expected that CdTe QDs with high PLQY could be obtained through this simple reaction route, whereas CdTe QDs synthesized by the flux method and by the hydrothermal method have PLQYs of only 35% and 14%, respectively [26, 27]. The as-obtained CdTe products are tested as fluorescent probes for the detection of mercury(II), a species which has severe adverse effects on human health and the environment even at very low concentration levels.

1. Experimental

1.1 Materials

MPA was obtained from Aldrich Chemicals. Rhodamine 6G was obtained from Acros Organics. Other analytical grade chemicals were purchased from Shanghai Chemical Reagents Company. All of the chemicals were used as received without further purification.

1.2 Synthesis of CdTe QDs

In a typical synthesis, 4 mL of cadmium chloride solution (CdCl_2 , 0.04 mol/L) was diluted to 42 mL

with ultrapure water, and then trisodium citrate dihydrate (100 mg), Na_2TeO_3 (0.01 mol/L, 4 mL), MPA (119 mg), and NaBH_4 (50 mg) were added successively under magnetic stirring. The molar ratio of Cd^{2+} /MPA/Te was 1:7:0.25. 10 mL of the resulting CdTe precursor was put into a Teflon vessel with a volume of 60 mL. A series of CdTe QDs were prepared at different temperatures (80–140 °C) and times (10–40 min) under microwave irradiation (400 W). After microwave irradiation, the mixture was allowed to cool to lower than 50 °C and the CdTe QDs sample was removed.

1.3 Apparatus and characterization of samples

A microwave digestion system (WX-4000) from Shanghai Yi-Yao Instruments, equipped with a controllable temperature unit, was used to synthesize CdTe QDs. The system works at a power of 0–1000 W and operates at a frequency of 2450 MHz. The resulting samples were diluted for optical characterization. All optical measurements were performed at room temperature. UV-vis absorption spectra were obtained using a TU-1901 UV-vis spectrophotometer (Beijing, China). Photoluminescence (PL) spectra were recorded with a WGY-10 spectrofluorimeter (Tianjin, China). Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) images were recorded on a JEM 2100 (JEOL, Japan) electron microscope operating at 200 kV. TEM and HRTEM samples were prepared by dropping aqueous CdTe QDs on copper grids and drying at room temperature. Samples were precipitated by 2-propanol and dried under vacuum for X-ray powder diffraction (XRD) characterization. XRD patterns were recorded on a Bruker AXS D8 Advance X-ray diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The QY of CdTe QDs was determined by using Rhodamine 6G in ethanol (QY = 95%) as PL reference [28].

1.4 General procedure for detection of metal ions

0.5 mL of CdTe QDs stock solution (the concentration of CdTe QDs was estimated to be 0.8 mmol/L), 1.0 mL of 0.05 mol/L Tris-HCl physiological buffer solution (pH=8.5), and different amounts of mercury(II) or

other metal ions were sequentially added to a series of 10 mL colorimetric cells. The mixture was diluted to the mark with ultrapure water, shaken thoroughly and equilibrated at room temperature for 20 min. All of the excitation wavelengths in the subsequent experiments were fixed at 400 nm.

2. Results and discussion

2.1 Characterization of CdTe QDs

Figure 1 shows TEM and HRTEM images of as-prepared CdTe QDs. The TEM image, depicted in Fig. 1(a), showed that the as-obtained CdTe QDs appeared as spherical particles with excellent monodispersity. As shown in the HRTEM image (Fig. 1(b)), the CdTe QDs had a diameter of about 3 nm, with the existence of well-resolved lattice planes showing that the CdTe QDs possessed a highly crystalline structure. The interplanar distance was about 0.326 nm, which corresponds to the (200) plane of cubic CdTe (JCPDS Card No. 65-0880). A typical XRD pattern for the MPA-coated CdTe QDs is shown in Fig. 2. The excitonic absorption peak of the CdTe QDs occurs at 538 nm. Calculated according to a reported method [29], the particle diameter determined by the first absorption maximum was 3.1 nm. The powder XRD pattern showed broad peaks typical for QDs. The average particle size of the product, calculated by the Scherrer formula, was 3.3 nm, which is similar to the TEM result and the value calculated from the first absorption maximum. The reflections could be indexed to the (111), (220), and (311) planes of cubic (zinc blende) CdTe.

2.2 Optical characteristics of the MPA-capped CdTe QDs

Figures 3(a), 3(b), and 3(c) present images of MPA-capped CdTe QDs with various sizes irradiated under ambient light, the corresponding absorption spectra, and photoluminescence (PL) spectra, respectively. The crude precursor solution was pale brown. There was no luminescence observed from the solution. Under microwave irradiation (400 W), various sizes of high quality CdTe QDs with tunable emission spectra were prepared by controlling the reaction temperature (80–140 °C) and time (10–40 min). Five samples 1–5 were prepared under different conditions

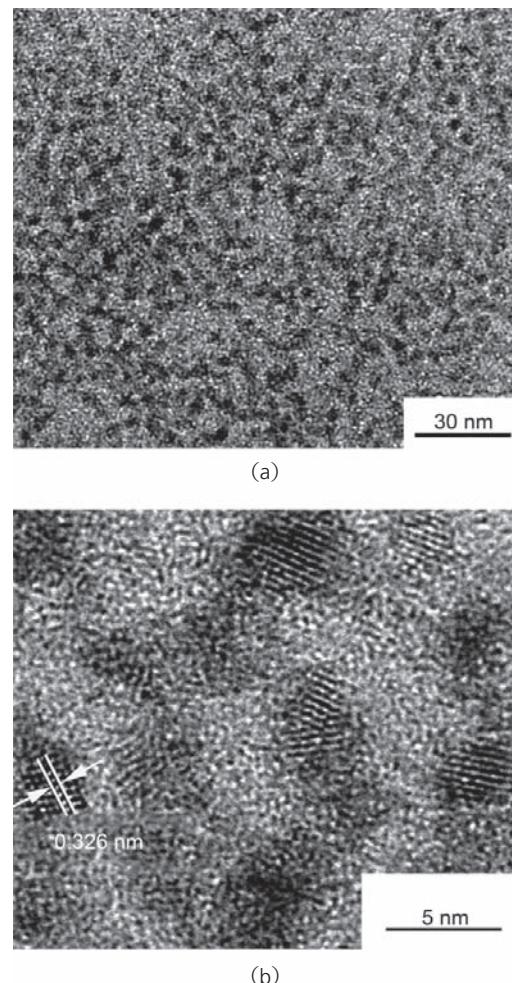


Figure 1 (a) TEM and (b) HRTEM images of CdTe QDs

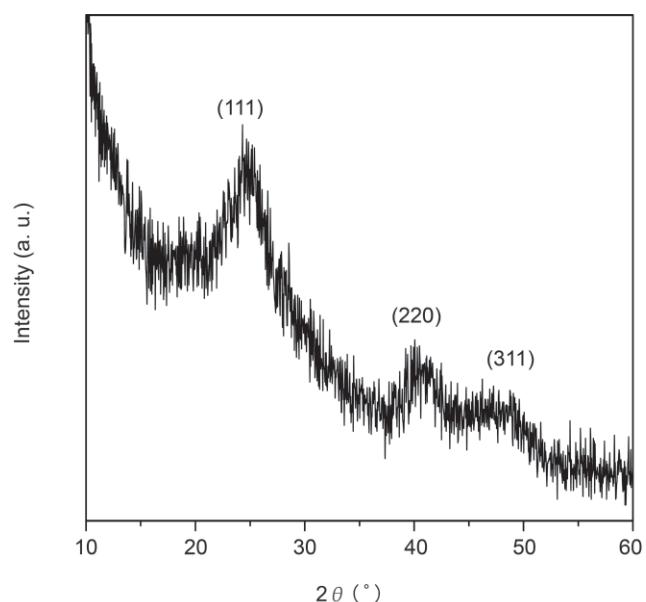


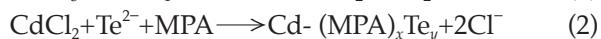
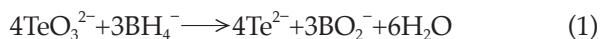
Figure 2 Typical XRD pattern of CdTe QDs



(see Fig. 3). The as-prepared CdTe colloidal solutions with different particle sizes showed different colors as shown in Fig. 3(a). The crystal growth of CdTe QDs was obvious as the reaction proceeded from Samples 1 to 5. Both absorption and emission spectra shifted to longer wavelengths with increasing the reaction temperature or prolonging the reaction time, as expected on the basis of “quantum-confined size effects”. The excitonic peak positions in the absorption spectra ranged from 496 nm (1) to 605 nm (5) and the PL emission peaks of CdTe QDs from 522 nm (1) to 632 nm (5). Their corresponding full width at half maximum (FWHM) values were between 35 nm and 54 nm. The sharp excitonic peaks in the absorption spectra and the narrow FWHM values in PL emission spectra suggest a narrow size distribution of the as-prepared CdTe QDs. The particle diameters of the above CdTe QDs determined by the first absorption maximum were around 2.3 (1), 3.0 (2), 3.4 (3), 3.5 (4), and 3.7 nm (5). The relative PLQYs of the five different CdTe samples were estimated to be 20%, 48%, 45%, 53%, and 40%, respectively using Rhodamine 6G as PL reference.

2.3 Effect of synthesis parameters on the QY of CdTe QDs

The following chemical reactions can be proposed in the synthesis of CdTe QDs via the microwave irradiation reduction route:



During the synthetic process, TeO_3^{2-} was reduced to Te^{2-} by NaBH_4 . The CdTe QDs were obtained by reaction between Te^{2-} and Cd^{2+} in the presence of MPA. The influence of various synthesis parameters, including the reaction temperature and time, the pH value of the reaction solution, and the molar ratio of MPA to Cd^{2+} on the QYs of QDs, were investigated. Under microwave irradiation, CdTe QDs with tunable emission wavelength from green to red were conveniently produced by varying the reaction temperature and time. Figure 4(a) shows the evolution of PL emission peak positions of CdTe

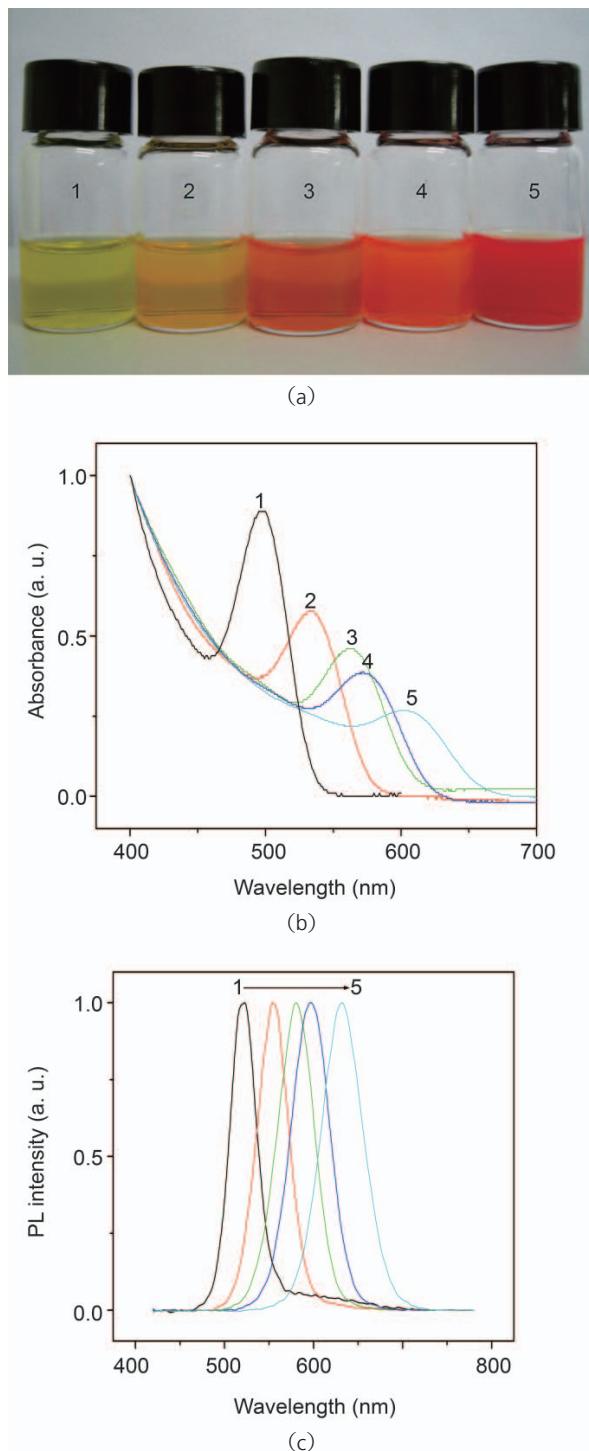


Figure 3 (a) Image of MPA-capped CdTe QDs with various sizes irradiated under ambient light; (b) the corresponding UV-vis absorption spectra; (c) photoluminescence spectra of MPA-capped CdTe QDs prepared at different temperatures and different irradiation times (1. 80 °C, 10 min; 2. 100 °C, 10 min; 3. 110 °C, 30 min; 4. 120 °C, 10 min; 5. 130 °C, 30 min). The photoluminescence emission peaks were at 522 nm (1), 555 nm (2), 582 nm (3), 597 nm (4), and 632 nm (5). PL spectra were recorded with excitation at 400 nm

QDs synthesized at four different temperatures vs microwave irradiation time. As expected, higher temperature favored an increasing growth rate of QDs and larger QDs were obtained. Consistent with previous studies, QDs with low QY were obtained at lower temperatures owing to the fact that a large number of surface defects were generated owing to the slow growth of QDs. Higher temperature leads to an increase in the crystallinity of QDs accompanied by an enhancement of QY [20]. On the other hand, a very high temperature may cause decomposition of the stabilizer [21], resultantly in a decrease in the QY. To obtain high quality QDs, the reaction temperature has to be optimized to realize an equilibrium of the attachment/detachment rate of the cadmium thiol complexes around the QDs surface [27]. As shown in Fig. 4(b), temperatures of 100–130 °C favor the formation of products with high QY under microwave irradiation. The pH value in the reaction system is another parameter influencing the synthesis of CdTe QDs. The experimental results indicated that both the fluorescence intensity and the longest emission peak of the CdTe QDs were strongly influenced by the pH value of the precursor solution (see Fig. S-1 in the Electronic Supplementary Material (ESM)). Relatively low pH values (8.0–9.0) favored PL enhancement and the stability of the CdTe solution, in accordance with previous reports

[30]. A low pH value should increase the number of cadmium thiol complexes around the surface of CdTe QDs, and favor the enhancement of QYs. During the synthesis process, the molar ratio of MPA to Cd²⁺ was adjusted from 2.4:1 to 8:1 while the amounts of the other reagents were fixed. The CdTe QDs became tightly capped by Cd²⁺-MPA complexes at higher MPA to Cd²⁺ molar ratios, which passivated the nanocrystal surface and decreased the number of surface defects. Consequently, CdTe QDs with high QY and narrow FWHM value could be obtained. Meanwhile, the growth rate slowed down with increasing MPA to Cd²⁺ molar ratio (see Fig. S-2 in the ESM). As discussed above, CdTe QDs with high QY could be obtained through optimizing the reaction parameters. CdTe QDs with the maximum QY of 60% were prepared by reaction at 100 °C for 30 min, with an MPA to Cd²⁺ molar ratio of 7:1, at pH 8.0.

2.4 Sensing performance of MPA-capped CdTe QDs for mercury(II) ions

CdTe QDs with a QY of 58% were tested in detection of Hg²⁺ ions in aqueous media. The relative fluorescence intensity was measured at $\lambda_{\text{em}}/\lambda_{\text{ex}} = 562/400$ nm (see Fig. S-3 in the ESM). Figure 5 shows the influence of Hg²⁺ ions on the luminescence of CdTe QDs. The luminescence intensity of CdTe QDs was successively depressed with increasing

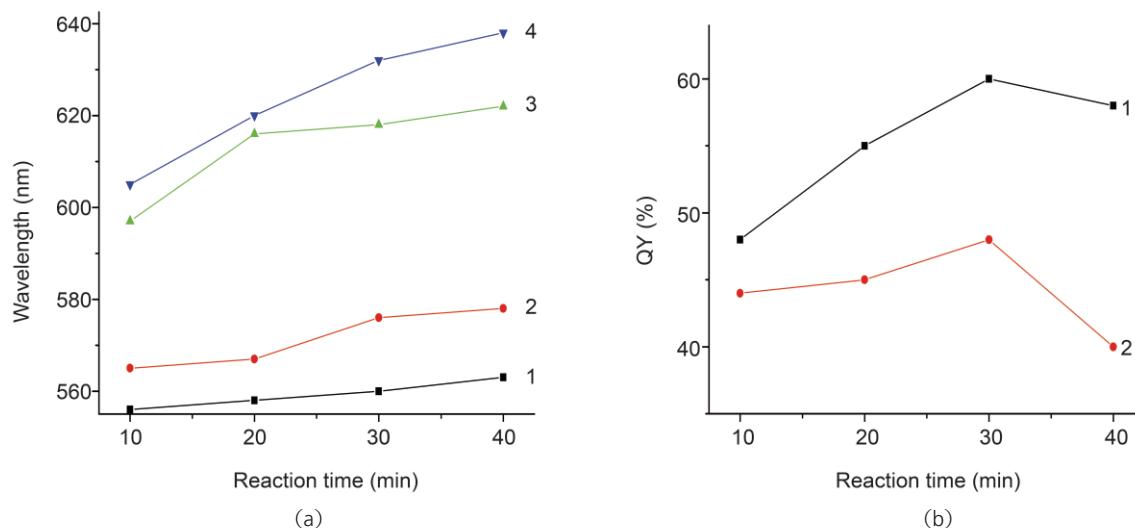


Figure 4 (a) Variation in the PL (photoluminescence) peak position of the crude solution of CdTe QDs during their growth at four different temperatures (1. 100 °C, 2. 110 °C, 3. 120 °C, 4. 130 °C); (b) PLQY of CdTe QDs synthesized at different reaction temperatures (1. 100 °C, 2. 120 °C). The pH and MPA:Cd²⁺ molar ratio were fixed



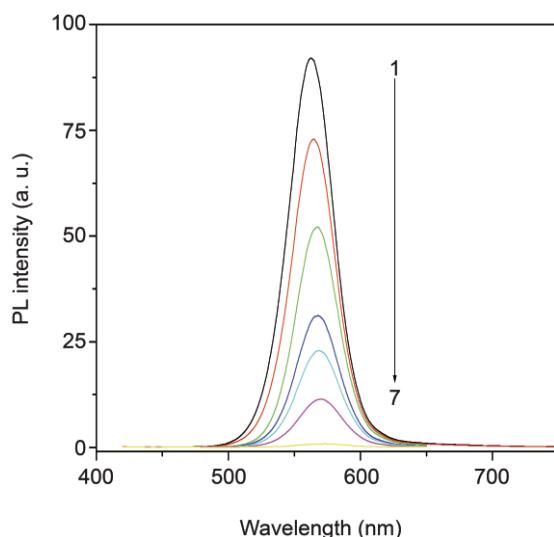


Figure 5 Effect of mercury(II) ions on the luminescence of MPA-coated CdTe QDs at pH 8.5. The concentrations of Hg^{2+} ($\times 10^{-9}$ mol/L) in 1–7 are 0, 8, 160, 400, 800, 2000, and 20000

concentration of Hg^{2+} ions. When the concentration of Hg^{2+} solution reached 2 $\mu\text{mol}/\text{L}$, the luminescence intensity was reduced to only one-tenth of that in the absence of Hg^{2+} . The quenching of luminescence of the QDs can possibly be attributed to ion binding followed by an effective electron transfer process from MPA to Hg^{2+} ions on the surface of QDs.

Figure 6 shows a Stern-Volmer quenching curve describing F_0/F as a function of Hg^{2+} ion concentration. F_0 and F are the luminescence intensities of the resulting CdTe QDs in the absence and presence of Hg^{2+} ions, respectively. The F_0/F response to Hg^{2+} ion concentration indicated a nearly linear behavior ($R=0.998$) in the range from 8.0×10^{-9} mol/L to 2.0×10^{-6} mol/L. The limit of detection (LOD) calculated according to the IUPAC definition is 2.7×10^{-9} mol/L, which is lower than the tolerance limit of 2 ppb (10×10^{-9} mol/L) for mercury in drinking water permitted by the United States Environmental Protection Agency (EPA). The method presented here has a low detection limit and high sensitivity, in contrast to most other recently described luminescent methods [31–34]. MPA-capped CdTe QDs displayed no obvious quenching response to other metal ions apart from Hg^{2+} (see Fig. S-4 in the ESM), which suggests a relatively high selectivity for this method.

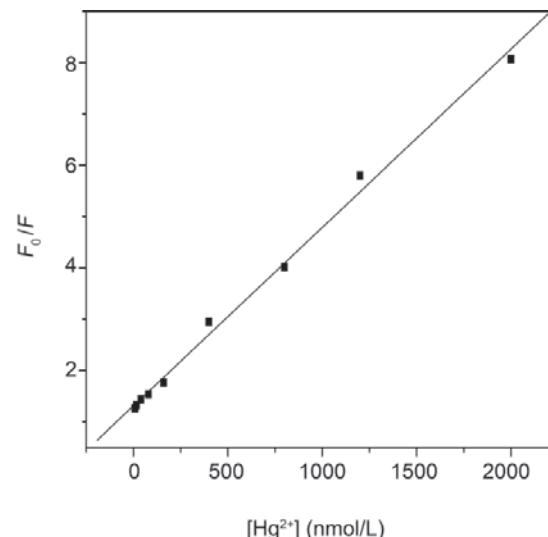


Figure 6 Stern-Volmer plot of the fluorescence intensity (F) of MPA-coated CdTe QDs vs. the Hg^{2+} ion concentration

3. Conclusions

In summary, the facile preparation of highly luminescent CdTe QDs using Na_2TeO_3 as the Te source via a one-pot microwave irradiation reduction route has been demonstrated. CdTe QDs with high QY (40%–60%) were prepared in relatively short time (10–40 min), after optimization of the synthesis parameters including the reaction temperature and time, the pH value of the reaction solution, and the molar ratio of MPA to Cd^{2+} . This synthetic approach is simple and rapid, benefiting from the use of microwave irradiation and an easily-handled tellurium precursor, Na_2TeO_3 . The as-obtained water-soluble CdTe QDs with high QY can be used to develop highly sensitive and selective probes for the detection of mercury(II) ions. The response was linearly proportional to the concentration of Hg^{2+} ion over the range from 8.0×10^{-9} mol/L to 2.0×10^{-6} mol/L with a detection limit of 2.7×10^{-9} mol/L.

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