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Microwave-Mediated Nonaqueous Synthesis of Quantum Dots at Moderate Temperature

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The use of microwave irradiation to accelerate both inorganic and organic chemical reactions has attracted widespread attention. Generally, microwave-mediated synthesis of quantum dots (QDs) has been conducted in aqueous solution. Here, using commercial diesel and glycerol as reaction medium, a microwave-mediated nonaqueous method toward CdSe QDs with size-tunable photoluminescent properties produces oleic-acid-protected QDs at moderate reaction temperatures of 50–140 °C, which are much lower than the current temperature necessary for the synthesis of CdSe QDs in organic solvents. The appropriate condition optimization for high-quality CdSe QDs shows that different sizes of CdSe QDs with emission wavelengths between 450 and 600 nm have been synthesized through varying time, temperature, feed ratio, and reaction medium.

Introduction

Photoluminescent semiconductor nanocrystals or quantum dots (QDs) have great industrial application potentials due to their size-dependent photoluminescent properties.^{1–5} In the past decades, great efforts have been dedicated toward the synthesis of CdSe QDs. Among them, a strategy to high-quality CdSe QDs undergoes promising power, in which cadmium oxide and organic phosphine have been utilized as raw material and ligand, respectively. The popular synthesis of QDs is generally achieved by utilizing organic phosphine ligands at the laboratory scale at a high temperature between 280 and 300 °C.^{6,7} However, commercial preparation of CdSe QDs has been limited by the costs associated with high-temperature oxygen-free equipment and the risks of using toxic organic phosphines. Therefore, the application of a novel, greener chemistry strategy, which uses lower temperatures, a faster reaction process, and nontoxic chemicals, to large-scale preparation of CdSe QDs is highly desirable, particularly in view of their significant applications in biomedical engineering, lasers, and photovoltaic solar energy cells.^{8,9}

To address these problems, a novel green method to prepare CdSe quantum dots, which decreases reaction temperature down to 200 °C, was introduced recently.¹⁰ In our laboratory, more efforts to other potential preparation methods of QDs have been taken. An alternative access to simplistic and prompt control of reaction temperature is microwave-assisted heating method. While the current oil-bath heating reaction transfers heat inward

from the heating source, radio waves in microwave-mediated heating directly penetrate the container wall and excite polar molecules, such as water, alcohol and fatty acids. They are absorbed by polar molecules and converted directly into atomic motion that is successively converted into heat. The whole process is different from conventional oil-bath heating because the heat is produced in a local domain through exciting atoms or molecules rather than conducting heat. Therefore, no heat needs to migrate toward the interior by conduction, and heat is ubiquitous when all the molecules are excited together in the microwave field. The use of microwave heating to accelerate chemical reactions has attracted widespread attention during the past decade. The advantages of this technology include not only faster reaction time but also higher production yields, cleaner reactions, better controllability, and reproducibility. Recently, there have been many reports on microwave-assisted organic synthesis,^{11–13} which stimulates our interest in the microwave-mediated synthesis of QDs in organic phase. Although there are some reports about microwave-assisted synthesis of QDs, all those synthesis methods involving inorganic nanostructures were conducted in aqueous phase^{14,15} or ionic liquids.¹⁶ As far as we know, little research on the microwave-assisted synthesis of CdSe QDs in common organic solvent, such as diesel, octadecene (ODE), and alcohols, has been reported. An essential problem encountered in microwave-mediated synthesis is the reaction media. In the conventional high temperature method performed in organic medium, nonpolar solvents such as ODE are used. Such solvents are difficult to agitate in the microwave field. Only those polar solvent molecules could be interacted within the microwave field and excited to generate molecule friction. Therefore, an alternative of ODE as reaction media that could be excited by microwave

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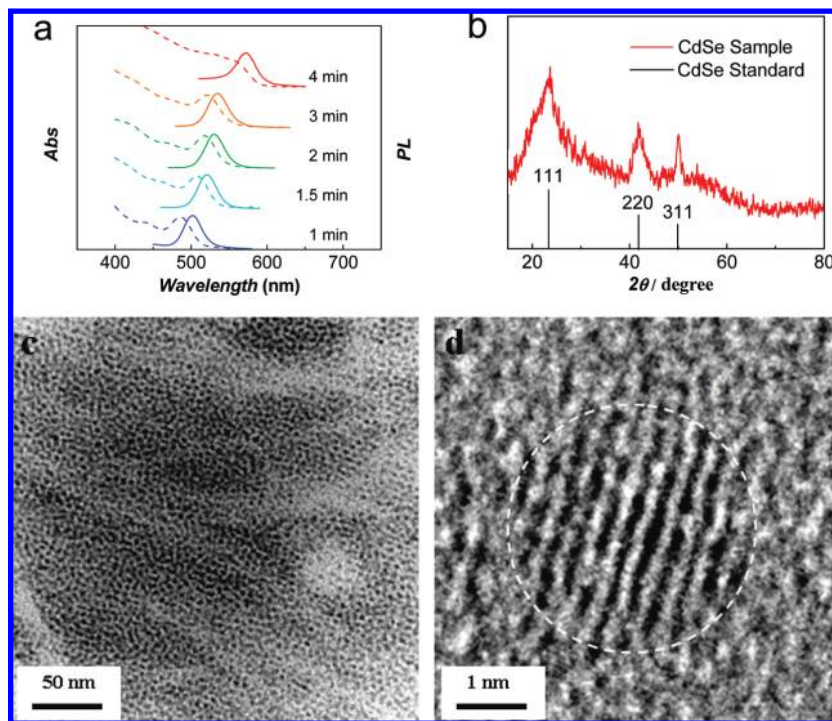


Figure 1. Optical properties and TEM characterization of CdSe QDs. (A) Temporal evolution of UV-vis and PL spectra of CdSe QD growth at 110 °C in microwave field. Conditions: [Cd]/[Se]/[OA] = 2:1:6, [Se] = 15.0 mM, temperature at 110 °C, diesel/glycerol = 1:1 (v/v), microwave power at 800 W. (B) XRD pattern of CdSe QDs indicating the zinc blende structure of QDs. (C) Representative TEM image and (D) HRTEM image of CdSe QDs, indicating the lattice fringe separation of cubic CdSe.

should be introduced. Here, we develop and demonstrate a microwave-mediated, green method of preparing oleic-acid-protected CdSe QDs with size-tunable photoluminescent properties: (1) microwave-mediated scheme in a two-phase reaction media consisting of commercial diesel plus glycerol, (2) a moderate reaction temperature at 50–140 °C, and (3) oleic acid as non-phosphine ligand. The microwave-mediated nonaqueous QD synthesis represents a direct and powerful method for photoluminescent semiconductor QDs.

Experimental Section

Materials. CdO (99%), Se powder (analytical grade), Te powder (analytical grade), sulfur sublimates (99%), and oleic acid (OA) (analytical grade) were obtained from Beijing Chemical Reagent Ltd., China, and used as obtained. Trioctylphosphine (90%) was obtained from Aldrich Co. Diesel (0#) was purchased from a commercial gas station. Rhodamine 6G (with a photoluminescence quantum yield of 95% in ethanol) was obtained from Sigma Co.

Characterization. Transmission electron microscopy (TEM) was conducted on a JEM JEOL 2010 transmission electron microscope operating at 100 kV. The sample was prepared by dropping a 3 μ L solution onto a carbon-coated Formvar copper grid (300 mesh) followed by solvent evaporation at room temperature. UV-vis spectra were recorded with a UV2300 spectrophotometer (Techcomp, China). Fluorescence spectra were recorded with a F2500 spectrophotometer (Hitachi, Japan) with excitation at 488 nm (slit width = 5 nm, 450 W Xe lamp). The fluorescence (FL) quantum yield (QY) of QDs was measured under the same setting of the F2500 spectrophotometer using Rhodamine 6G in ethanol as standard (QY = 0.95). The optical density at 488 nm (excitation wavelength) of Rhodamine 6G and QDs was set as a similar value. The first absorption peak of QDs and main absorption peak of Rhodamine 6G were kept under 0.1 to decrease the reabsorption of samples. The fluorescence quantum yields of quantum dots were calculated by comparing

the integrated intensity of QDs and Rhodamine 6G. The QY of QDs with emission lower than 520 nm was calculated using QDs ($\lambda_{em} = 535$ nm) as reference.

Microwave-Mediated Synthesis of CdSe QDs. The mixture of cadmium oxide (76.80 mg, 0.60 mmol), oleic acid (0.40 mL, 1.20 mmol), and 0# diesel (5 mL) was heated to 180 °C to completely dissolve CdO and to obtain an optically clear Cd stock solution. The Se stock solution was prepared in a 100 mL microwave reaction flask by dissolving selenium powder (14.40 mg, 0.15 mmol) in 0# diesel (1–5 mL) and glycerol (5 mL). The mixture was heated to 170 °C in a microwave field, and the resulting homogeneous solution was cooled down to about 35–40 °C. The Cd injection solution was then added into the solution. After the injection, the microwave-mediated temperature was kept at 110 °C for 2 min for the growth of CdSe QDs. To monitor the growth of the QDs, aliquots with a needle-tip amount of the reaction mixture were taken at various reaction times for optical measurement. The temporal evolution of the size and size distribution of CdSe QDs was monitored by UV-vis absorption and fluorescent spectroscopy. The reaction was terminated when the microwave field was removed. The QDs were precipitated twice by adding the solution into anhydrous acetone, then separated by means of centrifugation at 7000 rpm for 10 min, and redissolved in hexane.

Results and Discussion

Photoluminescent semiconductor CdSe QDs were synthesized in a microwave reactor equipped with a condenser and magnetic stirrer, which were described in detail in the Experimental Section. Typically, 0.15 mmol of Se powder, 5 mL of glycerol, and 5 mL of 0# diesel were loaded into a round-bottom flask. The mixture was heated in the microwave reactor, allowing the Se to dissolve. The Cd injection solution (0.30 mmol) was then swiftly injected into the solution when the solution temperature dropped to 35–40 °C. The reaction mixture was exposed in the microwave field and heated to 110 °C, generating luminescent CdSe QDs in several minutes. To monitor the growth of the QDs, aliquots with a

needle-tip amount of the reaction mixture were sampled at a time interval of 1–4 min for optical measurement. Compared with the popular methods reported previously at high temperatures (up to 280–300 °C), monitoring of nucleation reaction kinetics became much easier as a result of the slow growth of QDs at the relatively lower temperatures of 50–140 °C using the microwave-mediated synthesis method. The reaction was terminated when the microwave field was removed. The QDs were precipitated twice by means of the addition of the solution into anhydrous acetone. The resulting QDs were separated through centrifugation at 7000 rpm for 10 min and dried in vacuum overnight. As a result of the absence of air-sensitive organic phosphine ligands, the reaction could be conducted at a temperature below 100 °C in an open-air system without nitrogen protection or glovebox.

When the reaction mixture was heated in microwave radiation at the predesigned temperature, it was observed that the color of reaction mixture changed in seconds, indicating the formation of QDs. The temporal evolution of the size and size distribution of CdSe QDs was monitored by UV–vis absorption and fluorescent spectroscopy. Figure 1a shows the time-dependent fluctuation of UV–vis and photoluminescence (PL) spectra of CdSe QDs. The photoluminescence emission wavelength of QDs shows a gradual red-shift as the reaction proceeds, indicating that QDs grow gradually at controlled speed (Figure 1a). The CdSe QDs were near monodisperse, represented by a sharp emission with full width at half-maximum (fwhm) of 25–35 nm. The X-ray diffraction (XRD) patterns indicated that the QDs displayed zinc blende characteristic features, which appeared at about 24, 42, and 49° corresponding to the (111), (220), and (311) planes of the cubic phase of CdSe, respectively (Figure 1b). The QDs were well-defined and uniform in size as demonstrated by the representative TEM image (Figure 1c). A good crystalline structure of the CdSe QDs was confirmed by the high-resolution TEM (HRTEM) image of a single CdSe QDs from the same ensemble with the existence of lattice planes (Figure 1d).

Since QD emission is size-dependent, colloidal growth must be carefully controlled in order to achieve the target average radius with low polydispersity. A variety of methods have been developed to make semiconductor quantum dots, including arrested precipitation in water. Comparably, the nonaqueous synthesis routes based on organometallic compounds or Cd salts of aliphatic acid were pioneered and modified to produce superior crystallinity and higher PL QY at higher reaction temperatures with better size control and surface decoration, all by growing QDs in organic solvent rather than in water. A major drawback to the synthetic method performed in organic medium is the limitation of conventional heating mantle, by which heat is transferred inward from an outside reaction flask. In most cases, at a temperature as high as 280–300 °C, heating from the thermal mantle is too slow and even dangerous to be easily manipulated. As an alternative to a thermal mantle, therefore, the microwave-mediated QD synthetic method in organic medium was developed to produce the QDs with high PL QY using two-phase dual solvents, namely, glycerol and 0# diesel at the moderate temperature of 50–140 °C. Under organic phase microwave conditions, high-quality QDs could then be synthesized by combining the advantages of microwave in aqueous media and heating in the organic phase.

Detailed investigations have been performed to improve the optical quality of QDs by varying synthesis parameters, such as reaction solvent composition, precursor concentration, and temperature, all of which affect the growth kinetics of QDs to some extent. At 110 °C, a noticeable red-shift of the maximum emission wavelength of QDs occurred with increasing reaction time

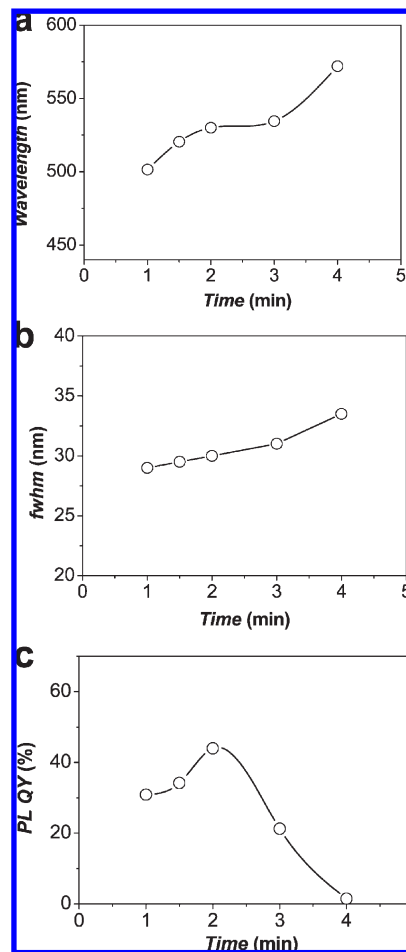


Figure 2. Effect of reaction time on optical properties of QDs: (a) emission wavelength; (b) fwhm; (c) PL QY. Standard conditions: microwave power at 800 W, temperature at 110 °C, [Cd]/[Se]/[OA] = 2:1:6, [Se] = 15.0 mM, diesel/glycerol = 1:1 (v/v).

(Figure 2a). The fwhm of photoluminescence emission gradually increased with time (Figure 2b). The PL QY of QDs fluctuated with the reaction time and attained the optimal value in about 2 min (Figure 2c). In microwave-mediated synthesis in nonaqueous polar media including glycerol and 0# diesel, reaction temperature also plays a critical role in the nucleation and growth of CdSe QDs. The UV–vis peak position of the QDs at different reaction temperatures between 50 and 140 °C after reacting for 2 min is illustrated in Figure 3. Under the present condition, the emission wavelength of QDs increases from 450 to 580 nm as the reaction temperature increases from 50 to 140 °C (Figure 3a). The fwhm of the QDs' photoluminescence gradually decreased with the increase of reaction temperature (Figure 3b), indicating the focusing of QD size. Meanwhile, the PL QY of as-prepared QDs shows a rising tendency as the reaction temperature increase from 50 to 90 °C, followed by a great drop as the reaction temperature further increases (Figure 3c). A reaction time of 2 min at higher reaction temperatures above 100 °C causes the decrease of emission intensity and even the disappearance of QD fluorescence. This indicates that 2 min of reaction time is favored to synthesize CdSe QDs with high PL QY at 110 °C. In order to synthesize CdSe QDs with high PL QY at various temperatures, however, longer reaction time is necessary at reaction temperatures below 110 °C while shorter reaction time is favored at temperatures greater than 110 °C. Therefore, when we investigate the effect of temperature on the PL QY of QDs, the effect of time

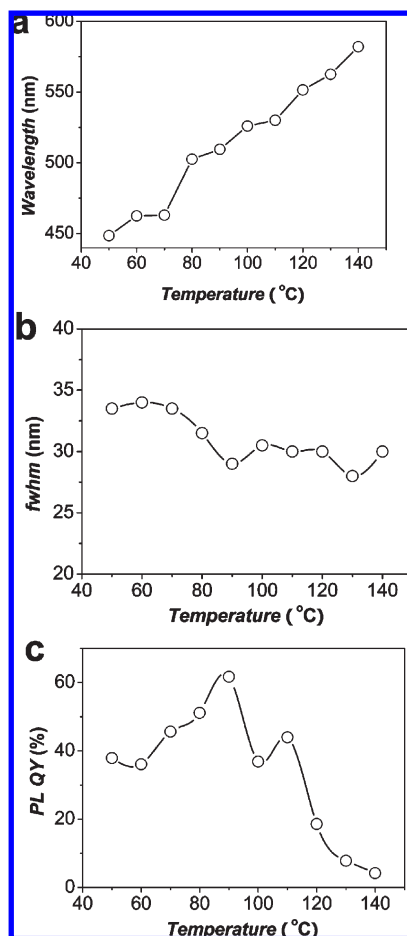


Figure 3. Effect of reaction temperature on optical properties of QDs: (a) emission wavelength; (b) fwhm; (c) PL QY. Standard conditions: microwave power at 800 W, reaction time at 2 min, [Cd]/[Se]/[OA] = 2:1:6, [Se] = 15.0 mM, diesel/glycerol = 1:1 (v/v).

should be considered along with the reaction temperature. In fact, the optimal reaction temperature of QDs was demonstrated to be significantly associated with ligands, solvents, and reaction time. The optimal PL QY of QDs fluctuates with the reaction temperature, that is, 110 °C in 2 min, 90 °C in 4 min, and 80 °C in 5 min. Notably, the CdSe QDs were produced at the temperature far below the boiling point of commercial diesel with a boiling range between 180 and 240 °C, probably because the reactivity of molecules or atoms excited by microwave irradiation is much higher than a conventional heating bath at such low temperature as 110 °C. This demonstrated that low-viscosity solvents and microwave heating can enhance the molecular diffusion and increase the reactivity of excited molecules or atoms, respectively, thus allowing the synthesis of CdSe QDs at such low temperature.

Aside from reaction temperature, the effects of other reaction parameters, such as Cd/Se molar ratio, concentration of oleic acid, and different nonaqueous solvents, on the resulting CdSe QDs were demonstrated by comparing the optical properties of QDs taken 2 min after the microwave-mediated reaction system was heated to 110 °C. Cd and Se are the essential raw materials for synthesis of CdSe QDs, and their effects on the optical quality of CdSe QDs were investigated. It was revealed that the initial Cd/Se molar ratio of the precursors influenced the optical properties, especially the intensity and fwhm of the emission peak of the as-synthesized quantum dots. Under the present conditions, the emission wavelength of QDs fluctuated between 500 and 540 nm with the increase of the Cd/Se molar ratio (Figure 4a). The

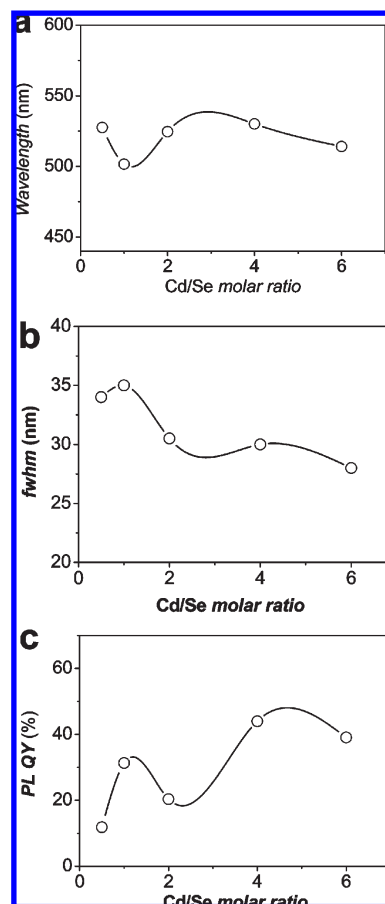


Figure 4. Effect of Cd/Se molar ratio on optical properties of QDs: (a) emission wavelength; (b) fwhm; (c) PL QY. Standard conditions: microwave power at 800 W, reaction temperature at 110 °C, reaction time at 2 min, [Se]/[OA] = 1:6, [Se] = 15.0 mM, diesel/glycerol = 1:1 (v/v).

influence of the Cd/Se molar ratio is apparently less than that of temperature on the maximum emission wavelength of the resulting QDs. The fwhm of the QD photoluminescence gradually decreased with the increase of the initial Cd/Se ratio of the precursors from 1:2 to 6:1 (Figure 4b), indicating the focusing of QD size. Meanwhile, the PL QY of as-prepared QDs fell between 10 and 50% and showed a gradually rising tendency with increasing Cd/Se molar ratio (Figure 4c).

The coordinating solvents have a significant effect on QD nucleation in conventional QD preparation, which, in turn, affects the stepwise growth of CdSe QDs.^{17,18} We chose glycerol and 0# diesel as the noncoordinating solvents, so that the effects of oleic acid could be studied. As the only ligand used in the synthesis of QDs, oleic acid plays multiple roles, which has been investigated in detail by Yu and co-workers.^{19,20} First, oleic acid is used as an organic acid to neutralize CdO into Cd precursor and make it dissolvable in nonpolar solvent as a cadmium oleate, while Se powder is dissolved in 0# diesel. Second, oleic acid acts as the ligand for the resulting QDs to stabilize the nucleation and growth of QDs. In a screening of oleic acid for the nucleation and growth of CdSe QDs, it was observed that the amount of oleic acid influenced the photoluminescent properties

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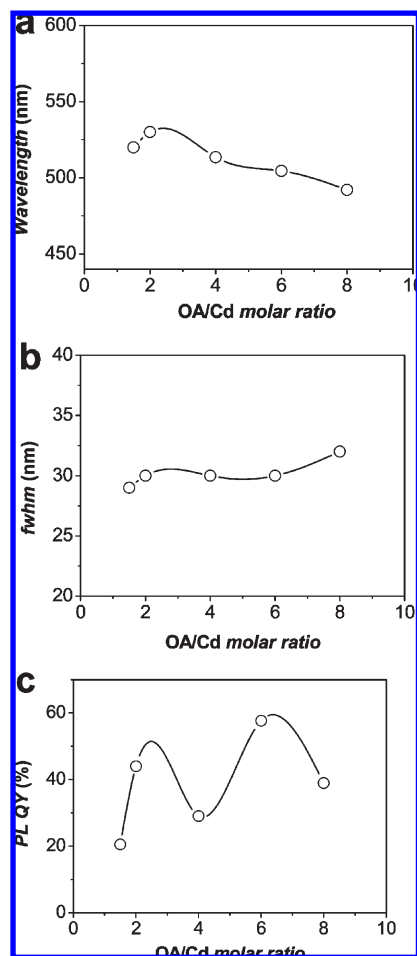


Figure 5. Effect of OA/Cd molar ratio on optical properties of QDs: (a) emission wavelength; (b) fwhm; (c) PL QY. Standard conditions: microwave power at 800 W, reaction temperature at 110 °C, reaction time at 2 min, [Cd]/[Se] = 2:1, [Se] = 15.0 mM, diesel/glycerol = 1:1 (v/v).

of QDs. Figure 5 shows, when the reaction was conducted for 2 min, the relationship between optical properties of QDs and the amount of oleic acid added. The more the amount of oleic acid added, the shorter the wavelength the emission moved toward (Figure 5a), the smaller the size of the QDs became, and the broader the size distribution of the QDs (Figure 5b). However, the FL QY of QDs fluctuated randomly with the oleic acid when reaction was conducted for 2 min (Figure 5c).

Understanding the impact parameters of PL QY and fwhm for CdSe QDs is important in optimizing the performance of QDs in downstream applications and evaluating the growth mechanism of QDs. Experimentally, the microwave-diathermanous media is indispensable to microwave synthesis. In order to demonstrate the effect of different solvents in the two-phase reaction media, other similar solvents, such as paraffin and glycol, were used to substitute diesel and glycerol, respectively. Paraffin, as an alternative to 0# diesel in the microwave-mediated system, did not cause any apparent difference in QD quality. As another alternative, glycol, which is a more polar diathermanous medium than glycerol to the microwave-mediated system, was found to inhibit the nucleation process, thus resulting in poor growth of CdSe QDs. In addition, by trying other polar solvents such as *n*-octanol, benzyl alcohol, dimethyl sulfoxide, and dimethyl formamide, we found that glycerol was the most appropriate one of the as-used media. The nonaqueous solvents such as

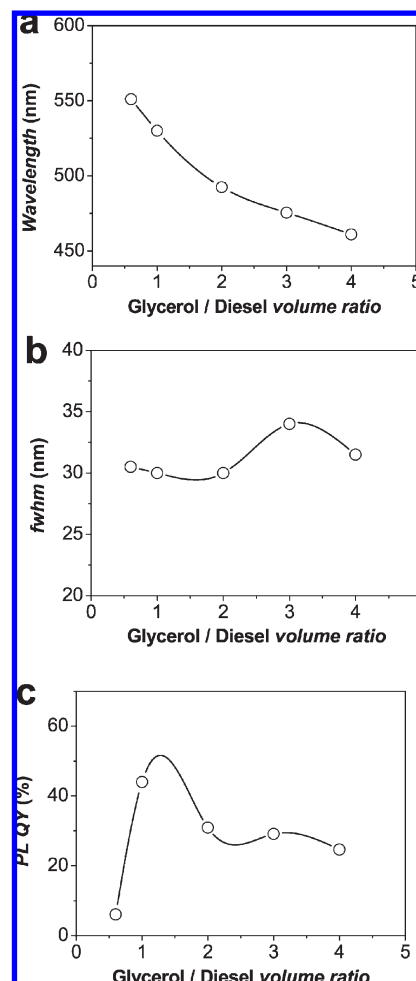


Figure 6. Effect of glycerol/diesel volume ratio on optical properties of QDs: (a) emission wavelength; (b) fwhm; (c) PL QY. Standard conditions: microwave power at 800 W, reaction temperature at 110 °C, reaction time at 2 min, [Cd]/[Se]/[OA] = 2:1:6, [Se] = 15.0 mM, diesel/glycerol = 1:1 (v/v).

glycerol can be optimized to maximize the PL QY of CdSe QDs and minimize particle size variation in certain stages of QD growth. The optical properties of QDs in the microwave-mediated system at 110 °C in 2 min with varying feed ratio of 0# diesel/glycerol are provided in Figure 6. The increase in the feed ratio of 0# diesel/glycerol resulted in an observable red-shift of maximum emission wavelength of QDs (Figure 6a). Additionally, the fwhm of photoluminescence emission is between 30 and 34 nm, which indicates the narrow size distribution of CdSe QDs (Figure 6b). Meanwhile, the FL QY of QDs fluctuates with the diathermanous media, and the optimal value of 0# diesel/glycerol is at 1:1 (Figure 6c).

In the same way, we have prepared CdS quantum dots for which absorption and emission spectra are shown in Figure 7. The strong blue emission was observed under UV excitation. This indicates that the microwave-mediated method to prepare Cd-containing quantum dots could be a promising strategy. However, the synthesis of CdTe quantum dots via microwave irradiation is a challenge, since the dissolution of Te has to be operated using organic phosphine ligands such as tributylphosphine or trioctylphosphine under water-free and oxygen-free environments, which is difficult under the current microwave reaction system. We are endeavoring to develop new system in Ar atmosphere. As a new nonaqueous microwave synthetic technique,

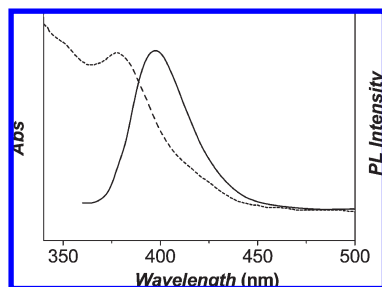


Figure 7. Typical optical properties of CdS QDs via microwave-mediated synthesis.

we are expanding microwave synthesis of quantum dots for other transition metal chalcogenides and oxides.

Conclusions

A microwave-mediated method of preparing oleic acid-protected CdSe QDs using commercial diesel and glycerol as reaction media is introduced. This method produced QDs at moderate reaction temperatures of 50–140 °C using nonphosphine ligands. This nonphosphine microwave-mediated synthetic green technique

produces CdSe QDs with size-tunable photoluminescent properties at relatively low temperature. The appropriate condition optimization for high-quality CdSe QDs shows that different sizes of CdSe QDs with emission wavelengths between 450 and 600 nm could be synthesized through varying time, temperature, feed ratio, and reaction medium. Considering the accessibility and promising industrialization prospective of microwave reactor, it is promising to accomplish microwave-mediated preparation of other transition metal chalcogenides and oxide nanostructures. We expect that the microwave-mediated synthetic method will prompt more basic research and applications including biomedical sensing and imaging, lasers, and photovoltaic solar energy cells.

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