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Preparation of CdSe Quantum Dots with Full Color Emission Based on a Room Temperature Injection Technique

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High quality CdSe quantum dots are synthesized through a room temperature injection technique by using $CdAc_2$ and Na_2SeSO_3 as precursors. In this synthesis approach, small CdSe clusters are formed after the injection at room temperature. Thereafter, CdSe quantum dots with emissions from the green to the red region can be obtained by transferring these clusters to different temperatures (40−150 °C) for particle growth. Meanwhile, CdSe quantum dots with emission in the blue−violet region (500−430 nm) are gained by an oxidation etching approach using H_2O_2 as oxidant. The advantage of this method is the natural separation of the nucleation and the growth process, which can provide a longer time for the preparation of the nuclei in simple operations and a well controlled fluorescence of the products, as the evolution of the fluorescence is slow at this low particle growth temperature.

Introduction

In the past two decades, II−VI semiconductor nanocrystals have attracted much attention from researchers working in various disciplines because of their quantum confinement effects and the size-dependent optical characteristics.[1–3] Their size-dependent optical property, which can be easily measured, provides a convenient means for the fundamental research on the intrinsic size effects in the nanometer region and on monitoring the growth process of small particle clusters. Their well controllable photoluminescence property, along with stability and narrow emission bandwidths, has made them potential candidates in light-emitting diodes (LEDs),[4–6] solar cells,[7,8] and biological labeling.[9–11]

As an important semiconductor material, CdSe has been extensively studied because of its well-established synthetic methods and the ability to tune its emission in the visible range simply by changing its crystal size. Over the past two decades, various synthetic approaches have been reported to synthesize CdSe nanocrystals, such as hydrothermal, sonochemical, electrochemical route, and so forth.[12–16] However, only few of them could be used to synthesize colloid CdSe nanocrystals with satisfactory size and size distribution. At present, the organometallic approach and its alternative routes[17–21] have been mostly adopted because they can prepare CdSe colloid nanocrystals with high quality morphology and also with good optical quality. The hot injection technique is often employed in this organometallic approach as it can produce a "burst nucleation" event, which

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is a crucial factor for the narrow size distribution of the products; high reaction temperature is also required to decompose the precursors. However, the hot injection process and the high reaction temperature often lead to a complicated manipulation which may limit the application in the scaled-up manufacture. At the same time, the high reaction temperature will bring a fast reactive rate. A rapid growth rate of the nanocrystals leads to a quick temporal evolution of the optical property, which may often finish in several minutes or tens of minutes;[19–23] thus, it is not easy to get the target products with the expected fluorescence. Compared with the organic system, the aqueous included system routes have the advantage of being environment friendly and low cost. Pan et al. designed a water−toluene two-phase[24] method to synthesize high quality CdSe quantum dots in a mild reaction condition. While this approach also requires a careful design of the Cd precursor, the CdSe products lack a wide range of controllable fluorescence. Hence, it is still challenging work to develop a method to prepare high quality CdSe quantum dots with an effective controllable fluorescence under mild conditions and with a simple operation technique.

In recent years, our group has devoted some efforts on a facile and general hydrothermal and amine-assisted solvothermal method to obtain II−VI group semiconductor nanocrystals using simple inorganic compounds as precursors.[12,25–27] Furthermore, a versatile liquid−solid-solution synthetic strategy and an interface-mediated approach have been developed to synthesize various high quality inorganic nanocrystals including metal chalcogenide,[28,29] but they showed a low fluorescence property. Herein, we will report the rational synthesis of CdSe quantum dots with a controllable emission property based on this interface-mediated method. In this synthesis process, $CdAc_2$ and Na_2SeSO_3 are chosen as precursors, and oleic acid is used as the capping ligand. The nucleation of the CdSe occurs and forms small particle clusters when the precursors are mixed together at room temperature. The subsequent growth of these small clusters is interrupted by the steady adhesive capping oleic acid as the dynamic activity of oleic acid is low at this temperature, which actually separates the nucleation and growth process of the nanocrystals. Thus, the mixing process of the precursors could be called a room-temperature injection compared to the hot injection. This room-temperature injection process offers a longer time for the preparation of the nuclei and also offers a simple operation. The growth of these small CdSe clusters could then be transferred to different temperatures for the growth of the crystals. By controlling the particle growth temperature, which can influence the attaching and detaching rate of the capping ligand to the particles, CdSe products emitting from green to red region can be obtained. The particle growth process could be extended to tens of hours as the reactive rate is relative slow because of the low crystal growth temperatures, which offers a convenient chance to get the target products leisurely. Meanwhile, CdSe with blue and violet emissions is obtained by a chemical oxidation method using H_2O_2 as the oxidant. This oxidation process is found to be relative to the crystallinity of the CdSe nanocrystals. By carefully controlling the oxidative condition, the photoluminescence peak can be gradually tuned from 500 nm to about 430 nm.

Experimental Section

Materials. All reagents used in this work, including Se powder, Na_2SO_3, NaOH, oleic acid (OA), $Cd(Ac)_2 \cdot 2H_2O$, H_2O_2(30%), ethanol, and *n*-hexane, were A. R. reagents (>99.99%) from the Beijing Chemical Factory, China. The selenium source Na_2SeSO_3 solution was prepared by refluxing 5 mmol selenium powder and 6 mmol Na_2SO_3 in 50 mL of distilled water for 3 h.

$$\mathrm{Se} + \mathrm{Na_2SO_3} \xrightarrow[\Delta]{\text{reflux}} \mathrm{Na_2SeSO_3} \qquad (1)$$

Synthesis. In a typical synthesis, 0.7 g of NaOH and 8.0 mL of oleic acid (OA) were dissolved in the mixture solution of 10 mL of deionized water and 30 mL of C_2H_5OH to form a transparent solution, 266 mg $Cd(Ac)_2 \cdot 2H_2O$ dissolved in 10 mL of deionized water was added to this solution by stirring, and then 10 mL of Na_2SeSO_3 solution (0.1 M) was added. The solution changed from white to yellow after about 5 s and was allowed to react for about 3−5 min, then transferred to several autoclaves and sealed. These autoclaves are taken in a designed temperature (40−150 °C) and reacted for a given time. When the reaction was finished, the autoclaves were cooled down by flowing water. The products that deposited in the bottle of the autoclaves were dissolved in *n*-hexane, ethanol was added subsequently to precipitate the nanocrystals by centrifugation, and then redissolved in *n*-hexane and precipitated with ethanol three times. Finally, the CdSe quantum dots were redispersed in *n*-hexane to form a transparent solution.

Oxidation Process. An appropriate amount of H_2O_2 (10%) was added to 10 mL of CdSe solution with a CdSe monomers concentration of about 0.1 mol/L. Typically, the amount of the oxidant H_2O_2 (10%) added in the CdSe sample with 2.2 nm is 0.5 mL for a 10 mL of solution with a monomer concentration of 0.1 M; the CdSe nanocrystal growth at higher temperature needed larger amounts of H_2O_2 (10%) for the oxidation. Then, the solution was left in a dark environment, and an aliquot solution was taken at different times to monitor the oxidation process.

Characterization. The samples were characterized by a Bruker D8 Advance X-ray diffractometer with Cu Kα radiation ($\lambda = 1.5418$ Å) and high-resolution transmission electron microscopy (HRTEM, FEI Tecnai G2 F20 S-Twin working at 200 kV). The electronic absorption spectra were obtained on a Hitachi U-3010 UV−vis spectrometer. Luminescence spectra were recorded with a Hitachi F-4500 fluorescence spectrophotometer.

Result and Discussion

Capping ligands which attach to the surface of nanocrystals are important to the formation of the nanocrystals. They can

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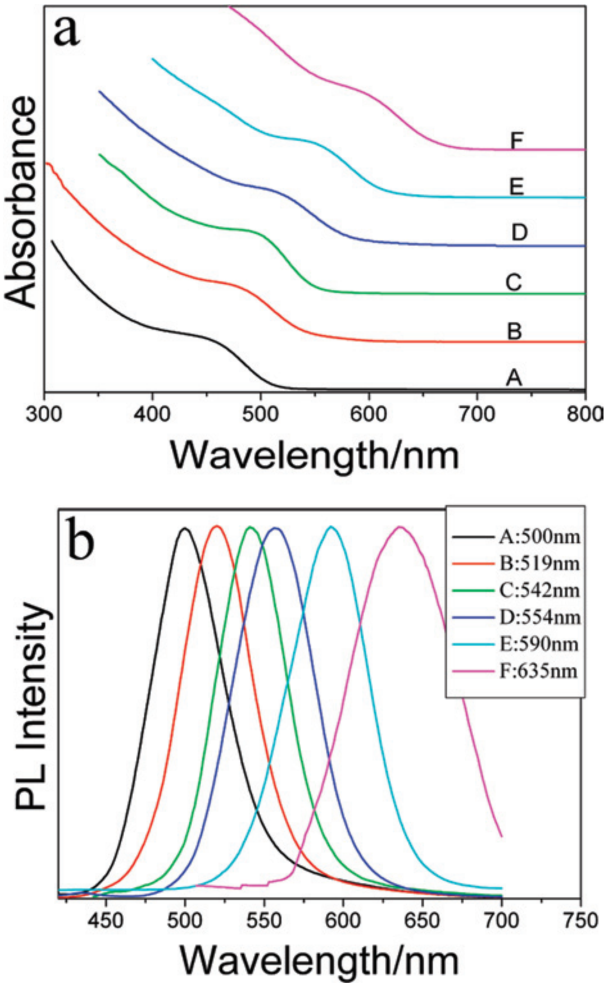

Figure 1. UV−vis absorbance (a) and PL (b) spectra of the CdSe quantum dots at different growth temperatures and at the same reaction time of 10 h (A, 45 °C; B, 60 °C; C, 80 °C; D, 100 °C; E, 120 °C; F, 150 °C).

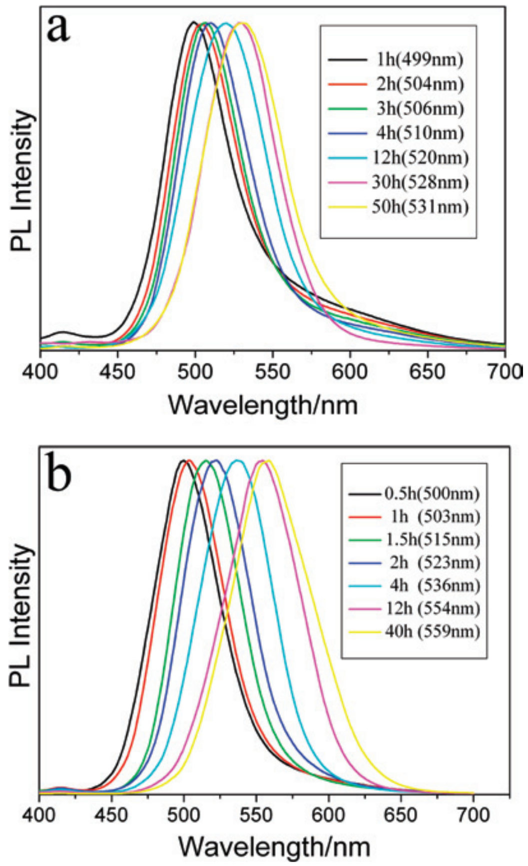

Figure 2. Temporal evolution of PL spectrum of the CdSe quantum dots taken at the growth temperatures of 60 °C (a) and 100 °C (b).

not only prevent the aggregation of these small crystals but also influence the morphology of the nanocrystals because of their different complexation to the crystals. The capping ligands are often bulky, and the behavior of these surface ligands is usually similar to their bulk materials; that is, the dynamics of the surface ligands will be influenced by the temperature and the ligand's chain length.[3,30] For example, when the reaction temperature is close to or higher than the boiling point of the bulk ligand material, the dynamics of the surface ligand is violent, similar to a quasi-gas phase, whereas while the temperature is close to the melting point, the surface ligand behaves much more calmly, similar to a quasi-solid phase. Thus, the reaction temperature, which influences the behavior of the capping ligands, can be used to control the growth of the nanocrystals.

The UV−vis absorption and the photoluminescence (PL) spectra in Figure 1a,b display the results of the CdSe products; the samples are shown at different crystal growth temperatures from the same crude nucleated solution and at the same reaction time. The absorption and PL spectra illuminate that, at the same initial nucleated condition and reaction time, the particles grow faster at higher temperature and attain a larger size. The formation of larger particles at higher growth temperatures can be explained by taking into account the kinetics of the particle formation. The particles can grow when the ligand molecule detaches and the exposed surface site reacts with Cd or Se precursors in the solution. A higher rate of attaching and detaching at higher temperatures will result in a faster growth rate and thus larger particle sizes. By tuning the particle growth temperature, the CdSe products emitting from green to red color can be obtained, the quantum yield of these fluorescence is about 9∼14%, and the quantum yield of green is a little higher than the orange and red.

Besides the growth rate, the temperature can also influence the final diameter of the CdSe products. Figure 2a,b shows the PL spectra of samples of CdSe quantum dots taken at different time intervals at the particle growth temperature of 60 and 100 °C, respectively. The PL peak shifts gradually in the first 12 h for these two growth processes. A smaller change is observed after reaction for 12 h, which reflects a slower growth of the nanocrystal toward a nearly constant particle size. The reaction temperature has been observed before as an important factor determining the size of the products.[31,32] Different from these reports where the particle growth is initially fast and subsequently slows down quickly,

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the growth process in this study is much milder; the particle growth can be extended to about tens of hours before it reaches the nearly constant size. The results in Figure 2a,b imply that the temperature is the crucial factor to determine the size of the final products in this synthesis approach. Obviously, this slow growth rate can offer a more convenient chance to control the growth dynamics and thus get the target product. CdSe quantum dots with green emission for application in LEDs, for example, requires that the emission peak lies in the range of 525−530 nm.[33] This requirement needs careful operation in a high reaction synthesis approach because the evolution of the optical property is fast. While in Figure 2a, at the particle growth temperature of 60 °C, we can see that when the emission peak reaches 528 nm, after the reaction has lasted for about 30 h, it still allows much time for the particles to grow till the emission peak reaches 531 nm, which means that we can obtain the target products more leisurely. Or in Figure 2b, while it takes about 2−3 h for the reaction to meet the requirement of the emission range in the green region, this also provides enough time to collect the target products.

Oleic acid is widely used as a low cost and green capping ligand for the semiconductor nanocrystals. It has a melting point of 16.3 °C. Thus, at a low reaction temperature, the activity of the oleic acid is low. When the nucleation occurs at room temperature (20 °C), the fallow capping oleic acid actually prevents the nuclei from growing, resulting in the separation of the nucleation and the growth process, and it can also makes the "burst nucleation" process last for a longer time, which is important for the formation of high quality quantum dots and also for scaled-up production. Herein, with this experimental condition, the yield of CdSe quantum dot products can be up to 1 g in about 250 mL of reactive solution. With the rise of the temperature, the dynamics of the capping ligand becomes more active. A faster attaching and detaching rate of the capping oleic acid is gained with the higher temperature. Without the complete protection of the capping ligand, the small nanoparticle seems more unstable, and it tends to grow bigger to reduce the surface energy. Thus, the higher particle growth temperature results in a larger particle size along with the higher particle growth rate. The reaction slows down with the consumption of the precursors. The small change of the PL spectrum at the last reaction stage means that the particles reach a nearly constant size, which can be considered as the end of the reaction. Therefore, by controlling reaction temperature, we get the CdSe quantum dots with green, yellow, orange, and red emissions (Figure 1b). Figure 3 shows the transmission electron microscopy (TEM) of the samples corresponding to the different emissions mentioned above. The average diameters of the particles in Figure 3a−f are 2.2, 2.6, 3.0, 3.3, 4.0, and 4.6 nm, respectively. Meanwhile, the low activity of the capping oleic acid at relatively low temperature also depresses the Ostwald ripening process. Actually, there is no obvious ripening process because there is no broadened and asymmetry PL spectrum[22] appearing at the last reaction stage in Figure 2a,b.

Figure 4 shows the X-ray diffraction (XRD) patterns of the CdSe quantum dots with different particle growth temperature. All of the peaks in the XRD patterns are apparently broadened to some extent. The deep valley at around 2θ angle of 35° and 45° indicates that the samples are of a zinc blende structure. The main diffraction peaks can be identified as (111), (220), and (311) crystal planes (JCPDS Card no. 19−0191). The gradual narrowing down of the XRD patterns with increasing temperature reflects an increasing size of the products, which matches well with the results in Figure 3. In addition, the stronger intensity of the diffraction peaks and the appearance of the minor diffraction peak (331) mean that the higher reaction temperature leads to a better crystallization of the products. The crystallinity will affect the oxidation process of the products, which will be studied below.

Different from the case at the longer wavelength emission, CdSe quantum dots with blue emission are not easy to produce. The optimal peak wavelength for blue emission is 460−470nm,[33] which requires CdSe particles smaller than 2 nm. Besides the fast reaction rate, the low melting point of the particles also increases the difficulty to obtain such small CdSe quantum dots. At a typical CdSe synthesis temperature (593 K), particles with radius smaller than about 1 nm are in the liquid state,[32] which means that only the particles larger than about 2 nm in diameter can be formed at 593 K, otherwise the particle will melt. Qu et al. have observed that the initial PL peak of the CdSe quantum dots is about 500 nm at the reaction temperature of 250 °C by an in situ method,[22] and the subsequent growth of the particle leads to the PL spectra shifting to the red region. Thus, it is not easy to get CdSe quantum dots with blue or shorter emissions through a direct synthesis approach. Reduction of the radius of the CdSe quantum dots by oxidative etching may provide an approach to get smaller sized nanoparticles while it often leads to the fluorescence quenching of the CdSe. Very recently, we have reported that CdSe quantum dots with blue emission can be prepared by a mild photochemical oxidation process, but it needs a relative long time.[34] Herein, we utilize a chemical etching method to get small CdSe quantum dots with blue emission. By carefully controlling the oxidative condition, CdSe quantum dots with blue emission can be obtained. Meanwhile, the experiments show that the oxidative results are relative to the crystallinity of the nanocrystals.

Figure 5a−c shows the PL spectrum evolution of the samples with different sizes during the oxidative process. It can be seen that not all of the samples can be oxidatively etched effectively to a smaller size with shorter emission while still retaining good fluorescence. The sizes of the samples before the oxidation in Figure 5a−c are about 2.2, 3.3, and 4.0 nm, which are gained at the growth temperature of 45, 100, and 120 °C. The amount

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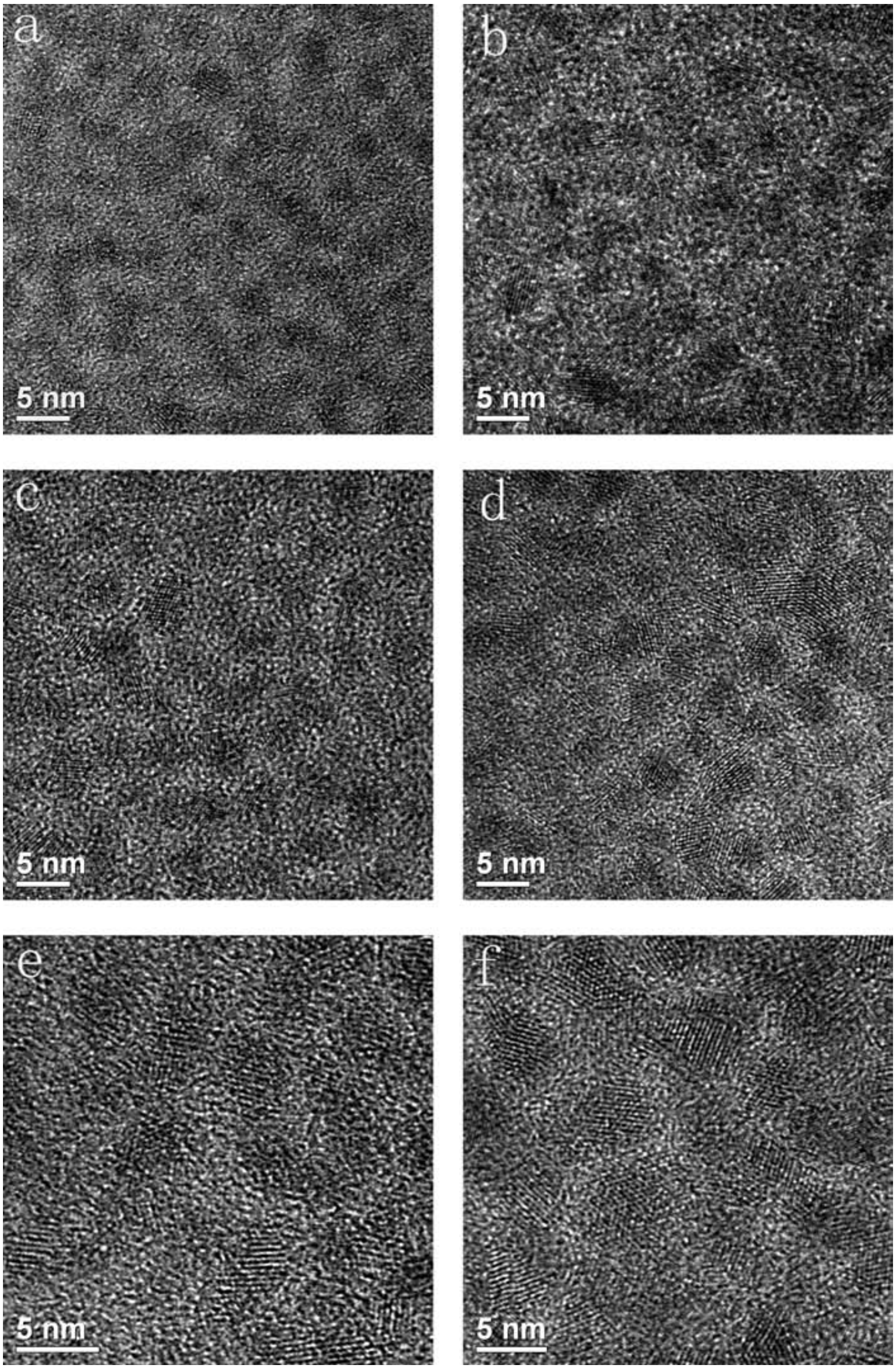

Figure 3. TEM of the CdSe samples corresponding to the emission peak of 500 nm (a), 519 nm (b), 542 nm (c), 554 nm (d), 590 nm (e), and 635 nm (f).

of the oxidant H_2O_2 (10%) added in the CdSe sample with a diameter of 2.2 nm is 0.5 mL for 10 mL of solution with a CdSe monomer concentration of 0.1 M. With this small quantity of the oxidant, the oxidation etching process is mild, and the PL peak of the sample can be adjusted well to the blue−violet region without the quenching of the fluorescence. Sometimes the quantum efficiency can increase a little during the oxidation process, which is shown in Figure 5a. CdSe quantum dots with blue emission can be successfully obtained by etching these small size CdSe materials. Compared with the smaller size samples, the larger nanocrystals are harder to etch. The same amount of H_2O_2 shows little effect when it is added to the CdSe sample with a diameter of 4.0 nm. A larger amount of the oxidant has to be added to etch these larger nanoparticles; however, the increased amount of the oxidant will lead to the quick quenching of the fluorescence and the complete oxidation of the sample, which can be seen in Figure 5c. The oxidation process of CdSe sample with medial size, see in Figure 5b, also shows a medial state between the larger particles and the small size particles. The PL spectrum can be tuned to some extent from 558 nm to about 500 nm but with broadened emission bandwidths, and the fluorescence also quenches at the end of the oxidation.

The different oxidative results may be caused by the different crystallinity of the samples. The larger size sample, which has a higher growth temperature, has a better

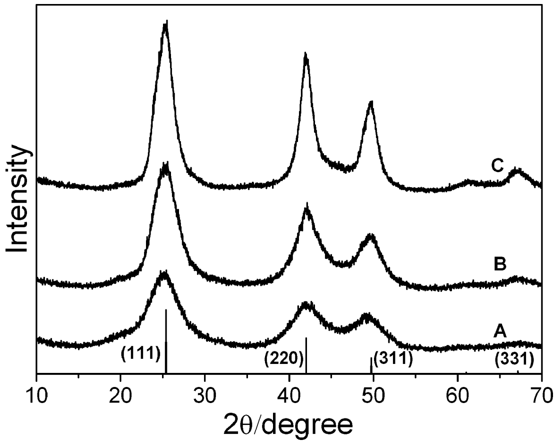

Figure 4. XRD patterns of the CdSe quantum dots with different reaction temperatures of (A) 60 °C, (B) 100 °C, and (C) 150 °C.

crystalline structure. It is accepted that crystallization of a crystal needs energy and time. Before the complete crystallization of a crystal, the outer layer structure of the crystal may stay at a quasi-amorphous state; thus, it could happen in a way where a crystallite core is covered with an amorphous shell.[35] Chen et al. report gradual crystallization on an amorphous nanoparticle of CdSe with a surface reconstruction.[36] The crystalline structure and the quasi-amorphous structure may show different responses to the oxidant. The nanocrystals here grown at low temperature have less crystallization structure than nanocrystals grown at high temperature. The completely crystallized crystal may not be easily oxidized as it has a compact structure. If the oxidant gets enough chemical potential, such as by increasing the amount of the oxidant, and begins to etch the crystal, then the crystal can be easily completely oxidized because the structure of the whole crystal is the same, which is like the situation in Figure 5c in which the CdSe quantum dots are quickly completely oxidized when the amount of the oxidant increases. On the other hand, the quasi-amorphous structure is easier to etch as the structure is not so compact; a small quantity of the oxidant can lead to the etching of the nanoparticle, which gives us the opportunity to gradually etch the particle with not a well crystallized structure to a smaller size. The mild oxidation condition can retain the fluorescence of the sample because it avoids the sharp corroding of the structures, as shown in Figure 5a. This oxidation process can be effective stopped by removing the oxidant, and the sample can stay stable in the air unless the oxidant is added again. Therefore, including the particle growth process above and this oxidation etching process, we get CdSe quantum dots with full color emissions, which are shown in Figure 6. These CdSe quantum dots stay stable with the ambient air environment and the fluorescence remains almost the same for longer than half a year, which may make them potential candidates for application in LEDs.

In Figure 5a, we can find that when the emission peak shifts to the blue region (463 nm), a small tail peak appears at around 550 nm, and it always exists even when the emission peak shifts to the violet region, which may be caused by the surface-trap emission due to the increase of the surface atom ratio. The increase of surface atoms with dangling bonds may induce additional electronic states in the band gap and thus influence the optical properties of nanocrystal. This phenomenon implies that the application

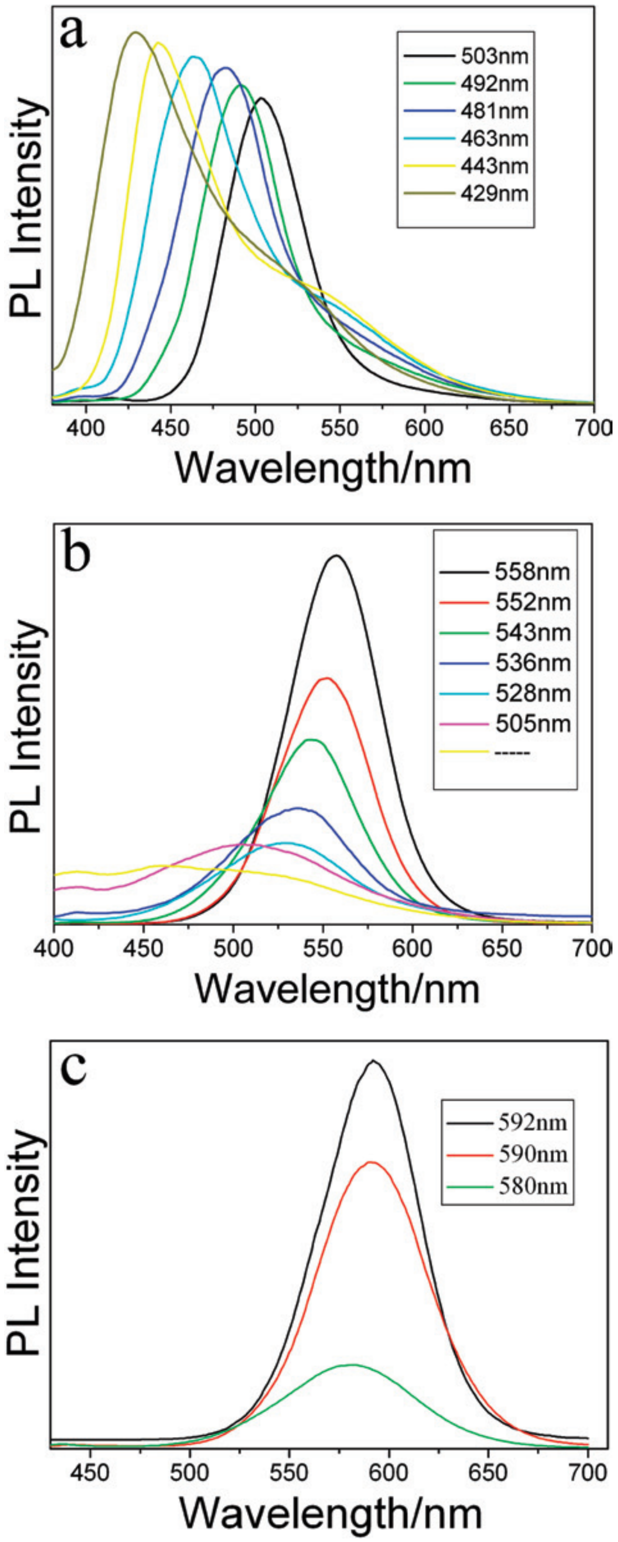

Figure 5. Evolution of the PL spectrum during the oxidative process with different sized samples.

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Figure 6. Fluorescence of the CdSe with different emission ($\lambda_{ex} = 365$ nm).

of CdSe quantum dots in the blue−violet region has to be coated with some matching materials which can saturate the dangling bonds on small CdSe surfaces.

Summary

In conclusion, we utilize a room temperature injection technique along with a temperature-controlled growth approach and the oxidative etching method to obtain CdSe quantum dots with full color emission. The room temperature injection technique and the low reaction temperature make the operation much simpler and safer. The low reaction rate gives us a convenient condition to study the dynamics of the crystal growth process and to obtain the target products easily. It is worth noting that the water solubility of Na_2SeSO_3 is more environment friendly compared with that of the organic Se sources such as TOPO-Se or TOP-Se as it avoids the use of a toxic organic capping agent. Thus, this mild and less toxic synthetic route and the well controlled optical property can be expected to bring more opportunities to nanocrystal-related research and the corresponding application field.

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