

Research Article

Green Light Emission of $\text{Zn}_x\text{Cd}_{1-x}\text{Se}$ Nanocrystals Synthesized by One-Pot Method

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We present a facile one-pot synthesis to prepare ternary $\text{Zn}_x\text{Cd}_{1-x}\text{Se}$ ($x = 0.2, 0.5, 0.8,$ and 1) nanocrystals (NCs) with high emission quantum yield (QY, 45~89%). The effect of Zn content (x) of $\text{Zn}_x\text{Cd}_{1-x}\text{Se}$ NCs on their physical properties is investigated. The NCs have a particle size of 3.2 nm and face centered cubic structure. However, the actual compositions of the NCs are $\text{Zn}_{0.03}\text{Cd}_{0.97}\text{Se}$, $\text{Zn}_{0.11}\text{Cd}_{0.89}\text{Se}$, and $\text{Zn}_{0.38}\text{Cd}_{0.62}\text{Se}$ when Zn content is 0.2, 0.5, and 0.8, respectively. In terms of the optical properties, the emission wavelength shifts from 512 to 545 nm with increasing Zn content from 0 to 0.8 while the QY changes from 89 to 45, respectively. Partial replacement of Cd by Zn is beneficial to improve the QY of $\text{Zn}_{0.2}$ and $\text{Zn}_{0.5}$ NCs. The optical properties of ternary NCs are affected by compositional effect rather than particle size effect.

1. Introduction

IIB-VIA group semiconductors with energy gaps covering the visible spectral range are suitable candidates for optoelectronic devices. Semiconductor nanocrystals (NCs), also known as quantum dots (QDs), have been widely investigated in the last decade due to their unique chemical and physical properties [1, 2]. Their particle sizes are in the nanometer range (less than 10 nm in diameter) and have very unusual properties. The continued interests in semiconductor NCs can be attributed to the size-dependent optical and electronic properties displayed by these quantum-confined materials. The small size of these compounds results in the three-dimensional confinement of the bulk charge carriers and the corresponding transformation of the bulk energy bands into discrete molecular energy states. This bulk-to-molecule transition is continuous, so that the band gap of the nanocrystalline material can be tuned to a desired energy by controlling the particle size [1, 2]. Besides, for the NCs with sizes of several nanometers, the huge surface energy is noted because most of the atoms are located on the surface. Therefore, surface modification is used in order

to increase the quantum efficiency of photoluminescence. Hence, a proper handling of the surface is essential to obtain highly luminescent NCs. The adjustable and sharp emission characteristics of these materials make them viable candidates for light emitting diodes (LEDs) [3–6], laser [7, 8], biological labels [9–11], and solar cells [12, 13].

In those applications especially for LEDs and bioimages, the luminous efficiency (or quantum yield, QY) of NCs is one of the most important demands. The QY is defined as the ratio of the number of photons emitted to the number of photons absorbed. Hence, how to improve the QY of NCs is the critical task. The QY of NCs can be improved by the following four ways. (1) Remove the lattice defect by heat treatment under high temperature in order to decrease the probability of trapped electron [14]. (2) Passivate the particle surface by bonding with an organic molecule and to improve the surface stability of NCs by reducing the lone pairs [15]. (3) Modify the surface by inorganic molecules (called core-shell structure) for the sake of increasing the electron-hole recombination efficiency [16, 17]. (4) Form alloyed NCs so as to eliminate the lattice mismatch and stress on the core-shell structures [9, 12].

Much work has been published regarding the optical property of NCs with core-shell structure, and the luminous efficiency of NCs can be enhanced significantly [18–20]. However, these processes are time consuming, and the particle size of NCs increases slightly, resulting in a red shift of emission wavelength. On the other hand, the ternary alloyed systems such as $Zn_xCd_{1-x}Se$ [9, 21, 22], $CdSe_xTe_{1-x}$ [10], $Zn_xCd_{1-x}S$ [12], CdS_xTe_{1-x} [23], and CdS_xSe_{1-x} [24] are obtained by a two-step route; that is, the alloyed NCs are formed by a core-shell structure through an annealing process. Therefore, two-step route also has the same disadvantage compared to that of core-shell NCs. Moreover, ternary $Zn_xCd_{1-x}S$ and $Zn_xCd_{1-x}Se$ NCs can be also synthesized by using a low temperature molecular precursor approach [25]. Therefore, the current focus on the synthesis of semiconductor NCs is to develop efficient routes for the synthesis of high quality and monodispersed NCs materials.

Because of the interesting optical properties of ternary NCs which can be controlled without changing the particle size, a large number of experiments have been performed on developing a fast procedure to produce high quality NCs in the range of visible light [9, 10, 12, 21–24]. However, little attention has been devoted to the one-step route and high emission QY of green light, as well as the role of Zn atom during synthesis. The highly luminescent ternary NCs, in particular of green emission, are potential ideal materials for white LED and QD lasers, which could solve problems occurring in the promising, but highly strained and time-consuming, CdSe/ZnSe core-shell structure. As we have mentioned above, the most important application of NCs is the white LED, but the QY of green light NCs is quite low [26]. Therefore, the aims of this report are to develop an effective one-pot synthetic strategy to make a series of high-quality ternary $Zn_xCd_{1-x}Se$ NCs with green light emission and to explore the Zn effect on the physical property of the prepared ternary NCs.

2. Experimental

2.1. Chemicals. Cadmium oxide (CdO, 99.998%) was purchased from Alfa Aesar. Zinc oxide (ZnO, 99.99%), stearic acid (SA, 99%), selenium powder (Se, 99.999%), trioctylphosphine (TOP, 90%), hexadecylamine (HDA, 90%), and trioctylphosphine oxide (TOPO, 90%) were obtained from Aldrich. Hexane (99.7%) and methanol (99%) were provided by Mallinckrodt Chemicals. All chemicals were used as received without further purification.

2.2. Preparation of $Zn_xCd_{1-x}Se$ NCs. Most details of the synthetic and characterizing methods were similar to the ones reported previously [27–30]. Ternary $Zn_xCd_{1-x}Se$ NCs were prepared by controlling Zn content and named Zn_x ($x = 0, 0.2, 0.5, 0.8, \text{ and } 1$). A typical synthesis is as follows. Total amount of 0.3 mmol of CdO and ZnO was mixed with 2.4 mmol of SA, which was used as a complex reagent, in a three-necked flask and then heated to 230°C under argon until a clear solution was formed to prepare the cadmium/zinc-SA precursors. After CdO and ZnO were

completely dissolved, the mixture was allowed to cool to room temperature, and a white solid precipitate was obtained. After cadmium/zinc-SA precursor was formed, the mixture surfactants, TOPO and HDA, 5.82 g of each, were added into three-necked flask and stirred together under Ar at room temperature, and the mixture was heated to 320°C under Ar flow to form an optically clear solution. At this temperature, the Se solution containing 1.5 mmol of Se dissolved in 0.9 mL of TOP and 2.1 mL of hexane was swiftly injected into the reaction flask. The reaction time was about 1 s, and the mixed solution was swiftly cooled down to stop reaction. Samples were precipitated with hot anhydrous methanol for purification process. The precipitate was dissolved in hexane for further measurement.

2.3. Characterization. The optical properties of samples were measured by fluorescence spectrophotometer (FL, Hitachi F-7000) and UV-vis spectrometer (UV-vis, Jasco V-670 spectrometer), respectively. Relative QYs of samples were determined by comparing the area under the curve of FL emission for the $Zn_xCd_{1-x}Se$ NCs with that of fluorescent dye (Rhodamine 6G in methanol). X-ray diffraction (XRD) patterns were recorded by a MacScience Co. Ltd. MO3X-HF22 powder diffractometer with Cu K α ($\lambda = 1.542 \text{ \AA}$) as the incident radiation. Transmission electron microscope (TEM, JEOL JEM-2010) was used to analyze the morphologies and size distribution of samples. The compositions of ternary NCs were conducted by inductively-coupled plasma atomic emission spectrometry (ICP-AES).

3. Results and Discussion

3.1. Optical Properties. Figure 1 displays the UV-vis and FL spectra of ternary $Zn_xCd_{1-x}Se$ NCs. Based on Figure 1(a), the band edge absorption and emission wavelength of CdSe with Cd/Se = 1/5 is 502 and 512 nm, respectively. The inset picture shows that the CdSe NCs have the green light with QY of 58%. Compared with the literature [30], it is interesting to mention that a high QY of CdSe with green light can be prepared within a very short period of time (1 s) in this study. Besides, the FL and UV-vis absorption spectra are extremely sharp with a full width of half maximum (FWHM) about 25 nm and the Stokes shift about 10 nm. Moreover, the emission peak with high symmetry and small FWHM implies that the size distribution of CdSe NCs is uniform. The nucleation occurs immediately as TOPSe is injected into the reaction flask, which can be observed from the changes of solution color. The smaller CdSe NCs with stronger quantum confinement effect are produced owing to higher nucleation rate, and this also makes them have high QYs. The results also indicate that to form a SA-Cd complex is quite important for boosting the nucleation rate of CdSe NCs. It has been reported that primary amines show the most promising results for achieving high FL efficiency for a variety of semiconductor NCs [19, 31, 32], and the function of SA is proven to be helpful for the formation of large-sized CdSe [33]. In addition, the quantum confinement effect decreases for NCs with larger particle sizes, resulting in lower

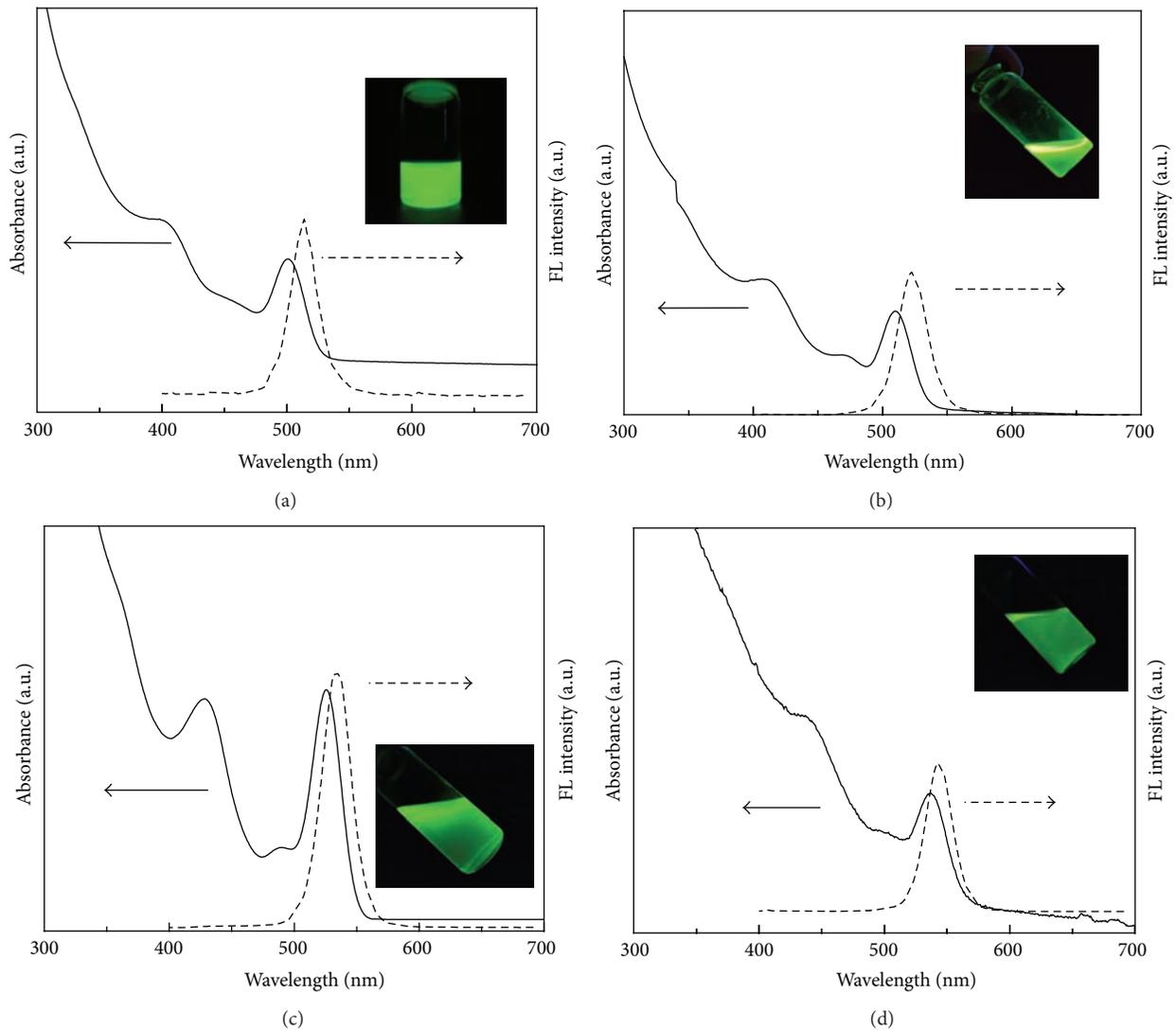


FIGURE 1: Fluorescence and UV-vis spectra of samples. (a) Zn₀, (b) Zn_{0.2}, (c) Zn_{0.5}, and (d) Zn_{0.8}. Excitation wavelength is 365 nm.

QY. Therefore, prevention of NCs from grain growth during the process is quite important. In our study, primary amine and steric phosphine oxide are used, and Cd-SA complex is preferentially formed to prepare CdSe NCs in a short time. Therefore, CdSe NCs with high QY can be obtained owing to the well-passivated surface of CdSe by those organic ligands and the stronger quantum confinement effect.

On the other hand, according to Figures 1(b), 1(c), and 1(d), the band edge absorption and emission wavelength are 510 and 523 nm, 525 and 534 nm, and 536 and 545 nm for the Zn content increases from 0.2, 0.5 to 0.8, respectively. Moreover, the Stokes shift and the FWHM of those samples is smaller than 13 and 28 nm, respectively. The inset pictures also show that the Zn_xCd_{1-x}Se NCs have the green light with QY ranging from 89 to 45%. Those results reveal that those samples have a narrow size distribution, a pure green color emission, and a nonlinear optical property. The QY of Zn_{0.2} and Zn_{0.5} NCs is 89 and 81% higher than CdSe NCs, while it is only 45% for Zn_{0.8}. Our results demonstrate that QY

is improved when compared with the samples prepared by previously reported methods [9, 21, 22]. Specifically, a high QY can be achieved by a facile one-pot synthesis. Based on Figure 1, only one emission peak with narrow FWHM can be observed for all samples, implying that either pure CdSe or ternary Zn_xCd_{1-x}Se NCs are formed. If ZnSe and CdSe nucleated separately, the corresponding FL and absorption peaks should appear. The band gap of ZnSe and CdSe is about 2.8 eV (443 nm) and 1.74 eV (713 nm), respectively. If ZnSe NCs are formed, the emission wavelengths will be less than 443 nm due to the quantum confinement effect. However, we cannot observe the emission peak around or less than 443 nm. This indicates that the ZnSe is formed but with very low QY or the ZnSe is not formed. Because the band gap energy is higher for ZnSe, meaning that it cannot emit green light. Hence, the formation of ZnSe NCs can be ruled out. Although the group II cations diffuse much easier than the group VI anions in II-VI semiconductors [34], the bond strength between Cd and Se is higher than that between Zn

TABLE 1: Physical properties of as-prepared samples.

Theoretical composition	Emission wavelength (nm)	Particle size ^a (nm)	QY (%)	Actual composition ^b
Zn ₀	512	3.1 ± 0.32	58	—
Zn _{0.2}	523	3.1 ± 0.27	89	Zn _{0.03}
Zn _{0.5}	534	3.2 ± 0.31	81	Zn _{0.11}
Zn _{0.8}	545	3.3 ± 0.28	45	Zn _{0.38}
Zn ₁	—	—	—	—

^aTEM characterization.

^bICP-AES characterization.

and Se, implying that CdSe NCs are much easier to form. In order to confirm the stoichiometric ratio of Cd and Zn, the mole fraction of Cd and Zn in the resulting NCs is determined by ICP method. From the ICP results (shown in Table 1), the actual composition of Zn_{0.2}, Zn_{0.5}, and Zn_{0.8} is Zn_{0.03}, Zn_{0.11}, and Zn_{0.38}, respectively, meaning that ternary alloyed Zn_xCd_{1-x}Se NCs can be prepared successfully under high Zn concentration by one-pot alloying route. For Zn_{0.2} sample, Cd is partially replaced by Zn through a doping process. The QY of ternary NCs is higher than that of CdSe when increasing the Zn content from 0.2 to 0.5. When Zn content increases to 0.8, the QY decreases dramatically, implying that even the concentration of Cd is reduced; Zn_xCd_{1-x}Se NCs with high QY can also be prepared. Qu and Peng [30] mentioned that for the CdSe with an initial Cd/Se of 1/10, its PL QY is higher up to 80%, which is consistent with our results. Based on ICP results we find that the molar fraction of Zn in Zn_xCd_{1-x}Se NCs is higher for high Zn content sample, so the lower QY of Zn_{0.8} sample may be caused by the formation of ZnSe phase. The actual composition of Zn_{0.2} sample is Zn_{0.03} with QY of 89%, suggesting that when the CdSe is the main phase, the ratio of Cd/Se as well as QY is high [30]. On the other hand, the lower fraction of ZnSe phase is noted for Zn_{0.5} sample, and the covalent bond strength can be increased by partially replacing Cd with Zn, resulting in higher QY of ternary NCs.

Figure 2 shows the FL spectra of ternary Zn_xCd_{1-x}Se NCs with reaction time of 1 s. The emission wavelength of Zn_xCd_{1-x}Se NCs shifts obviously from 512 nm when increasing the amount of Zn from 0 to 0.8. When Zn content is 1 (ZnSe), no emission wavelength can be observed, meaning that ZnSe cannot be prepared or the ZnSe NCs are with QY nearly zero under this condition. The observed systematic composition-controlled shift of the emission maximum to longer wavelength is explicable by the formation of Zn_xCd_{1-x}Se ternary NCs via intermixing wider band gap ZnSe with the narrower band gap CdSe NCs, rather than forming separate CdSe and ZnSe NCs or core-shell structure CdSe/ZnSe, because the emission wavelength of ternary Zn_xCd_{1-x}Se ($x = 0.2$ to 0.8) NCs is located between that of CdSe and ZnSe. Since the band gap of CdSe and ZnSe is 1.74 and 2.8 eV, respectively, it is implied that the ternary Zn_xCd_{1-x}Se shows a nonlinear optical property, which may be caused by the compositional or particle size effect.

3.2. Morphology and Structure Analysis. In order to understand the effect of composition and particle size on emission

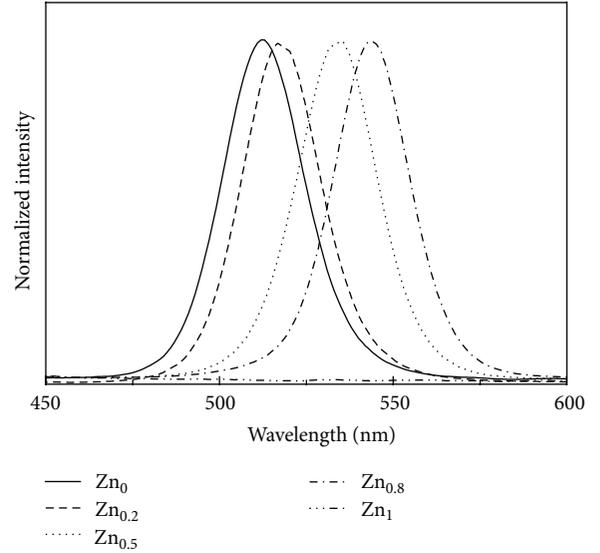


FIGURE 2: FL spectra of samples react for 1 s.

property of NCs, the TEM technique is used. The TEM micrographs of ternary Zn_xCd_{1-x}Se NCs are shown in Figure 3. The particle size for all samples is almost the same, which is about 3.1, 3.1, 3.2, and 3.3 nm for CdSe, Zn_{0.2}, Zn_{0.5}, and Zn_{0.8}, respectively, suggesting that the nonlinear optical property of ternary Zn_xCd_{1-x}Se NCs is caused by the compositional instead of particle size effect. Bailey and Nie mentioned that the relation of the anion-cation bonding to their equilibrium positions leads to local structural disordering and a particularly large band gap reduction in CdSeTe-type semiconductor alloys [10], resulting in a nonlinear optical phenomenon. In our study, we believe that a similar mechanism is found in Zn_xCd_{1-x}Se-type semiconductor alloys when the Zn content is not high enough.

The results of emission wavelengths, particle sizes, QYs, and actual compositions of ternary Zn_xCd_{1-x}Se NCs are listed in Table 1. A large red shift of Zn_xCd_{1-x}Se NC is observed with increasing the Zn molar fractions from 0 to 0.38 (emission wavelength is between 512 and 545 nm), and the particle size is almost the same. Zhong et al. mentioned that a large blue shift of Zn_xCd_{1-x}Se NC is observed with increasing the Zn molar fractions from 0 to 0.67 (emission wavelength is between 615 and 500 nm), and the particle size also increases from 5.2 to 7.5 nm. The optical properties of Zn_xCd_{1-x}Se NCs with linear optical property and high

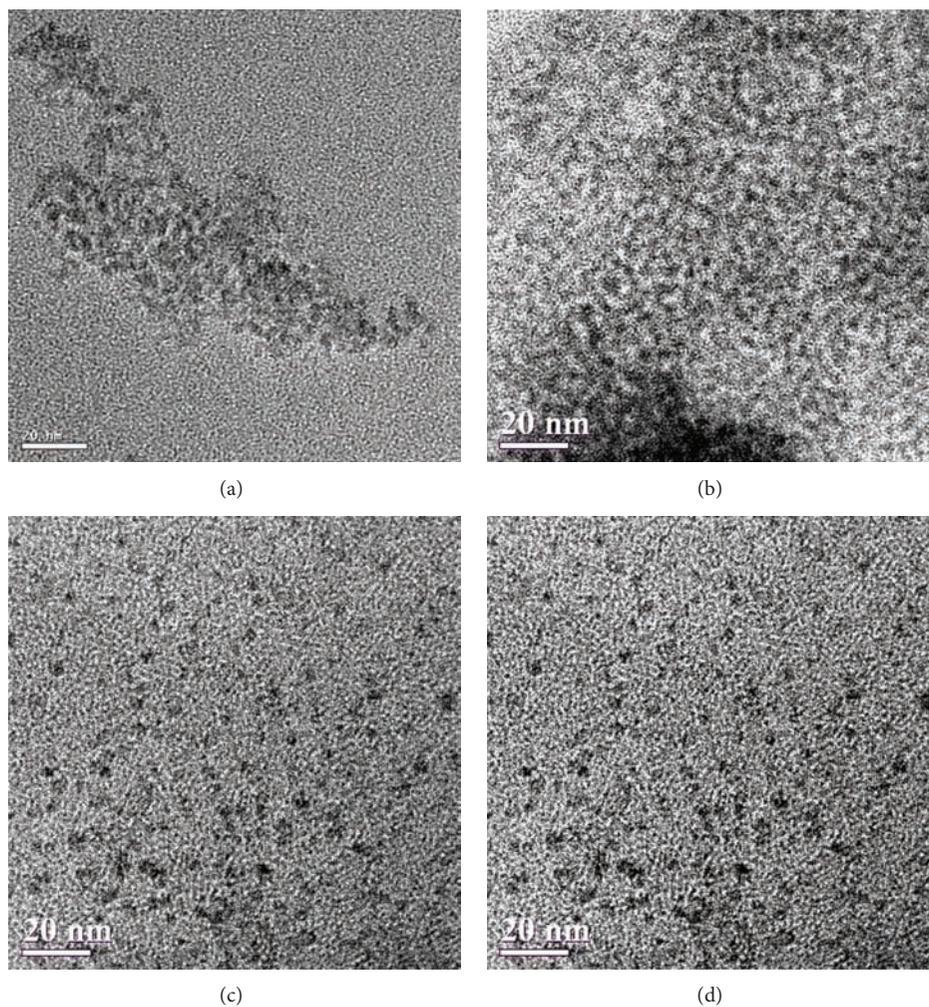


FIGURE 3: TEM images of samples. (a) Zn_0 , (b) $Zn_{0.2}$, (c) $Zn_{0.5}$, and (d) $Zn_{0.8}$.

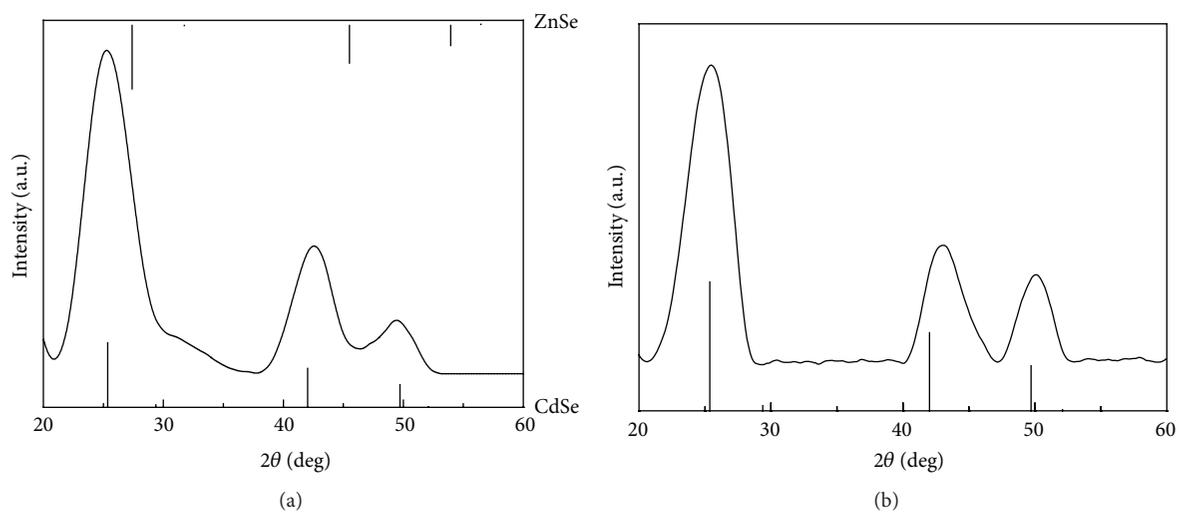


FIGURE 4: XRD patterns of samples. (a) $Zn_{0.2}$ and (b) $Zn_{0.5}$.

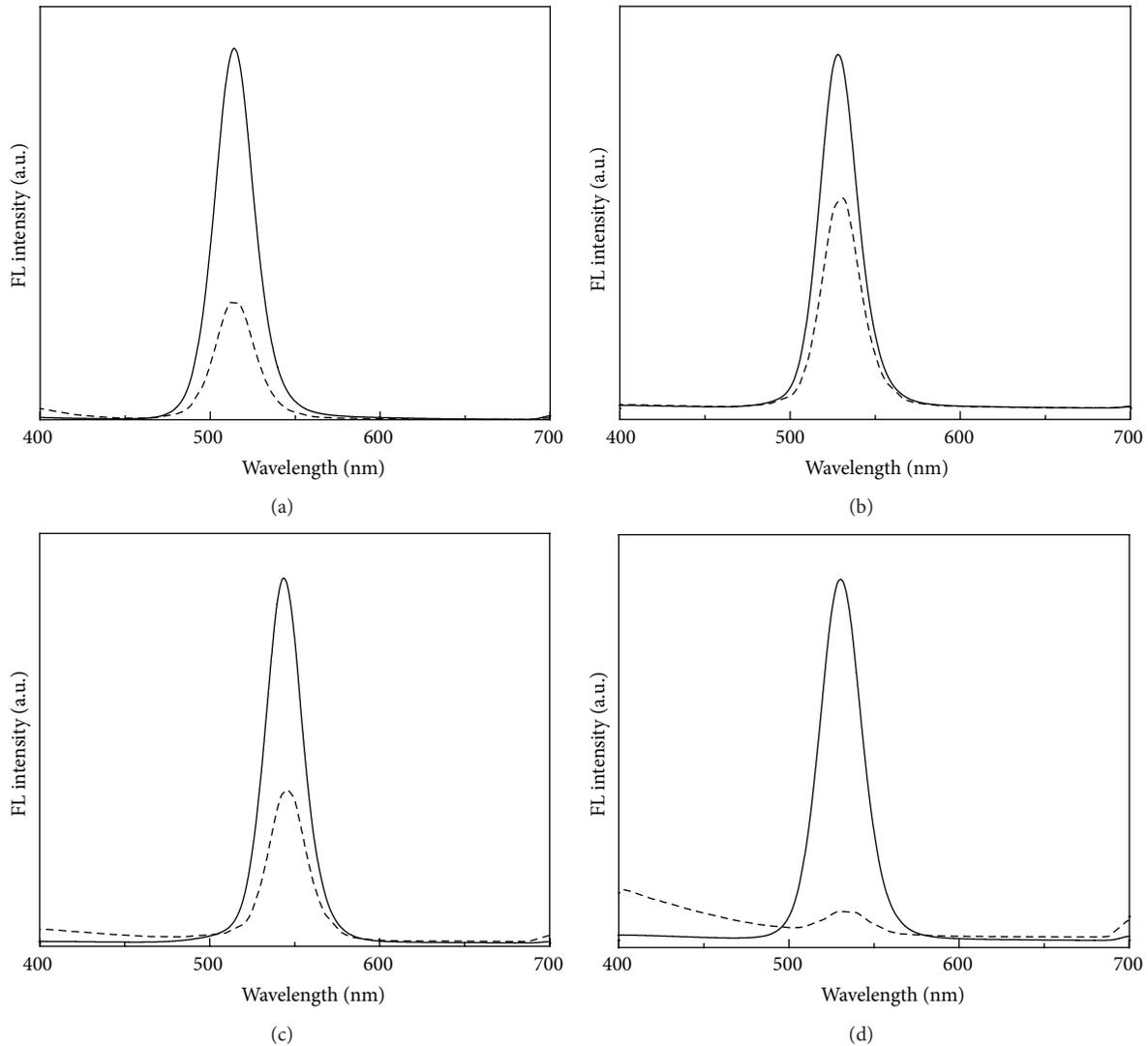


FIGURE 5: FL spectra of samples. (a) Zn_0 , (b) $Zn_{0.2}$, (c) $Zn_{0.5}$, and (d) $Zn_{0.8}$. (Solid line: as-prepared NCs; dash line: aging for 2 months.)

QY are due to larger particle size, high crystallinity, lattice structure, decreased interdiffusion, and spatial composition fluctuation [9]. The emission peak of composition-tunable $Zn_xCd_{1-x}Se$ NCs moves to short wavelength when increasing the Zn content. On the other hand, Bailey and Nie pointed out that the optical properties of $CdTe_{1-x}Se_x$ NCs are nonlinear and the emission peaks for $CdTe_{1-x}Se_x$ NCs are out of the range of that for CdSe and CdTe [10]. According to a theoretical model developed by Bernard and Wei, the nonlinear effect of alloyed NCs is due to the following factors: (1) different atomic size of different ions, (2) different electronegativity values, and (3) different lattice constants [35, 36].

In our study, the emission peaks of ternary $Zn_xCd_{1-x}Se$ NCs are between those of ZnSe and those of CdSe. However, when the actual Zn mole fraction increases from 0 to 0.38, a significant red shift of ~ 35 nm is observed. This result may be due to the different reactivity and atomic radii of the Zn

and Cd. From ICP results it can be seen very clearly that the Zn content in actual composition of ternary $Zn_xCd_{1-x}Se$ NCs is much lower than that of the theoretical composition. Therefore, the emission properties of ternary $Zn_xCd_{1-x}Se$ NCs may be dominated by CdSe under lower Zn content. When the Zn content is higher than a critical value, the emission wavelength of ternary $Zn_xCd_{1-x}Se$ NCs may either shift to short wavelength or appear as two wavelengths. In this situation, the optical properties of ternary $Zn_xCd_{1-x}Se$ NCs may be dominated by ZnSe, and the QY is also decreased. In this study, because the actual Zn content in alloy is not high enough, the nonlinear optical properties can be observed.

Figure 4 shows the XRD patterns of $Zn_{0.2}$ and $Zn_{0.5}$ sample. A face centered cubic (FCC) structure similar to pure CdSe NC is obtained for $Zn_{0.2}$, while a slight peak shift of NC is observed for $Zn_{0.5}$. As the Zn content increases, the diffraction peaks shift toward larger angles. The XRD results are also consistent with ICP and confirm again that ternary

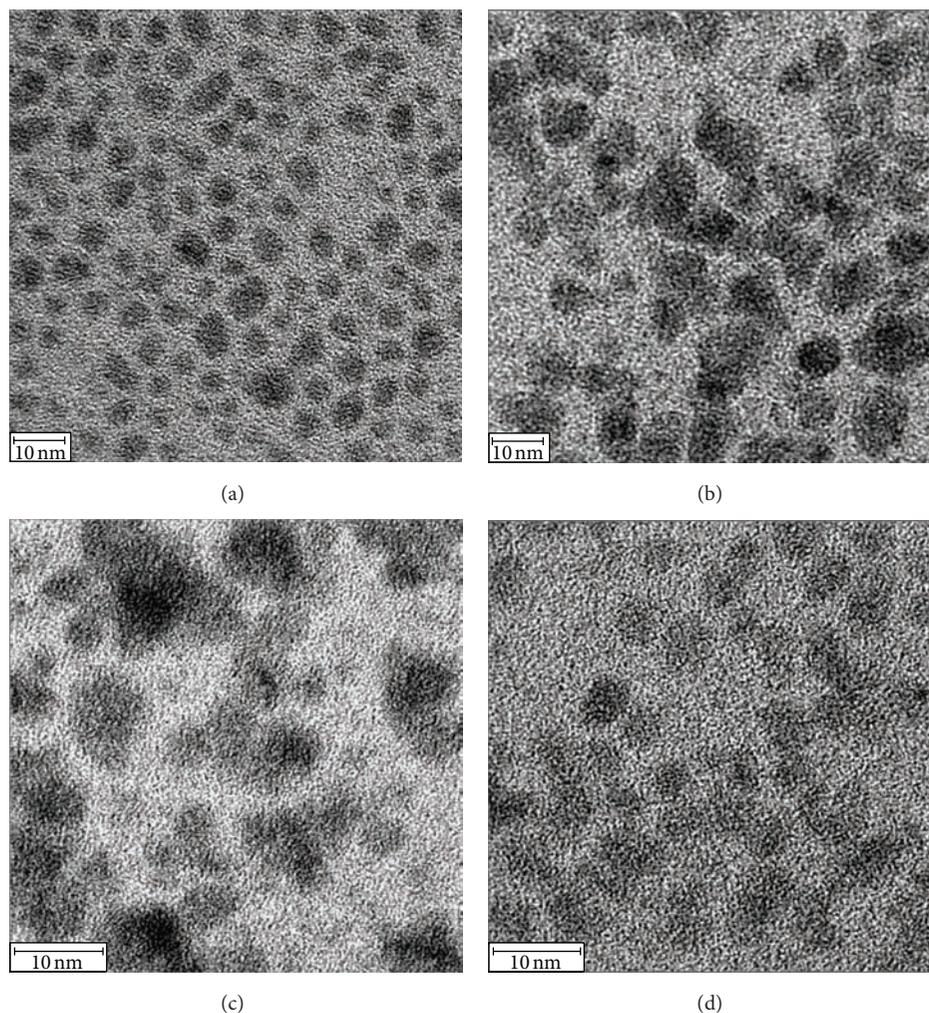


FIGURE 6: TEM images of samples aging for 2 months. (a) Zn_0 , (b) $Zn_{0.2}$, (c) $Zn_{0.5}$, and (d) $Zn_{0.8}$.

alloyed $Zn_xCd_{1-x}Se$ NCs can be prepared under high Zn content. On the other hand, a doping effect can be found under lower Zn content.

3.3. Stability of NCs. In order to investigate the stability of NCs, all of the samples were dissolved in hexane and stored in dark for 2 months. The FL spectra and TEM micrographs of $Zn_xCd_{1-x}Se$ NCs are shown in Figures 5 and 6, respectively. In Figure 5, the FL intensity for all of the samples decreases after aging for 2 months. It is interesting to find that the peak position of emission wavelengths for all samples is almost the same compared to as-prepared samples, but the intensity of peak for all samples decreases, resulting in a decrease in QY. Moreover, only one emission peak can be observed, and its FWHM increases slightly. For the TEM observation shown in Figure 6, we can find that the size distribution of all samples is not uniform. Moreover, the shape of NCs changes from spherical to elliptical and the size becomes bigger than that of as-prepared one. The QY of samples decreases after aging for 2 months as shown in Table 2. The $Zn_{0.2}$ shows the best stability in all samples. As

we know that the capping agent such as TOPO and HDA covers the surface of NCs, resulting in improving the QY. The interaction between those surfactants and surface of NCs becomes weak as aging time increases. Therefore, Ostwald ripening occurs, resulting in nonuniformed size distribution of NCs because small particles dissolve and deposit on the large particle surface. Moreover, once the Ostwald ripening occurs, the capping agents are not adsorbed on the surface of NC anymore. Therefore, only one emission peak with lower intensity can be observed, and the QY of aging sample decreases due to the concentration of capped-NCs decreases. Larger particle of NCs formed by the Ostwald ripening cannot emit light, so only one emission wavelength appears in Figure 5.

4. Conclusion

An effective and facile one-pot synthetic method has been developed for the formation of highly luminescent, composition tunable, high QY, and high-quality green emitting $Zn_xCd_{1-x}Se$ NCs. The emission wavelength from 512 to

TABLE 2: Physical properties of samples aging for 2 months.

Theoretical composition	Emission wavelength (nm)	As-prepared QY (%)	After-aged QY (%)	Size range (nm)
Zn ₀	512	58	16	2.8~5.1
Zn _{0.2}	523	89	51	2.8~5.5
Zn _{0.5}	534	81	26	2.9~5.6
Zn _{0.8}	545	45	9	3.0~5.9
Zn ₁	—	—	—	—

545 nm can be tuned by changing the composition of the Zn_xCd_{1-x}Se NCs from Zn₀ to Zn_{0.8}. Besides, not only Stokes shift but also the FWHM of ternary Zn_xCd_{1-x}Se NCs is very small, implying that a narrow size distribution and saturated color of ternary Zn_xCd_{1-x}Se NCs can be obtained. The Zn content of ternary Zn_xCd_{1-x}Se NCs affects their optical properties significantly. When increasing the amount of Zn from 0.2 to 0.8, the emission peak is red-shifted from 523 to 545 nm and the QY decreases from 89 to 45%, respectively. From the TEM analysis, those samples have almost the same particle size, indirectly suggesting that the nonlinear optical property of Zn_xCd_{1-x}Se is caused by the Zn compositional effect instead of the size effect. The one-pot preparation method reported in this study cannot only reduce the concentration of Cd but can also maintain the QY of NCs. Moreover, this would be a new class of colloidal chemistry method to prepare high quality of green emitting NCs and use them as a phosphor in white LED.

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