

STRUCTURAL AND OPTICAL PROPERTIES IN NEAR INFRARED OF CdTeSe COLOIDAL QUANTUM DOTS FOR POTENTIAL APPLICATION IN SOLAR CELLS

Pham Nam Thang^{1,3}, Le Xuan Hung^{2,3}, Nguyen Thi Minh Chau^{1,3},
Vu Thi Hong Hanh⁴, Nguyen Ngoc Hai⁵, Nguyen Thi Thuc Hien²,
Pham Thu Nga^{1,2,*}

¹*Institute of Materials Science, VAST, 18 Hoang Quoc Viet Road, Cau Giay District, Hanoi*

²*Institute of Research and Development, Duy Tan University, Da Nang*

³*Graduate University of Science and Technology, VAST, 18 Hoang Quoc Viet Road, Hanoi*

⁴*Faculty of Physics, Thai Nguyen University of Education, Thai Nguyen*

⁵*Department of Education and Formation, Quang Ninh, Viet Nam*

*Email: phtnga@ims.vast.ac.vn, ngalamvn@gmail.com

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ABSTRACT

This report presents the results on the optical properties of CdTeSe ternary alloyed quantum dots (QDs) fabricated by colloidal chemical method. We have used the micro-Raman spectra as a tool to determine the formation of CdTeSe alloy phase, the composition ratio of Te:Se when fabricated at different temperatures from 180 °C to 280 °C. The intensity ratio of two vibration lines at 159 cm⁻¹ and 188 cm⁻¹ allows the determination of Te:Se composition ratio available in QD samples. The Raman spectra of CdTe_xSe_{1-x} when x changes between 0, 0.2, 0.4, 0.5, 0.6, 0.8 and 1, fabricated at 260 °C, shows clearly the change in intensity of the optical vibration lines. The absorption and emission spectra of CdTeSe QDs show that the absorption edge of these QDs shifts up to 880 nm and up to ~ 860 nm for the emission. Detailed studies on size, structural, vibrational and optical characteristics of ternary alloyed QDs are also discussed. The absorption region of fabricated CdTeSe QDs is completely suitable for usage in solar cells.

Keywords: CdTeSe alloyed quantum dots, Raman spectra, PL spectra, absorption, TEM images.

1. INTRODUCTION

Colloidal quantum dots (QDs) are fast-improving materials for next-generation solution-processed optoelectronic devices such as solar cells, photo catalysis, light emitting diodes, and photo detectors [1]. One of the most promising approaches involves the use of semiconductor quantum dots (QDs) as light absorbers. QDs exhibit attractive characteristics as sensitizers due to their tunable band gap by size control, which can be used to match the absorption spectrum to

the spectral distribution of solar light [2]. Studies on using strong-absorbing quantum dots in the near-infrared (NIR) range (700 – 900 nm) to absorb solar radiation have been carried out recently, to apply in quantum-dot-sensitized solar cells (QDSSCs) or colloidal quantum dot solar cells. In this work, we present the optical properties of CdTeSe ternary quantum dots fabricated in solution at a suitable temperature ($< 300\text{ }^{\circ}\text{C}$), with changing absorption and emission spectra up to $\sim 870\text{ nm}$, that can be used well in solar cells. This is the type of alloy QDs that have been announced to achieve efficiency over 9 % in QDSSCs with CdTeSe/CdS [3]. In a recent publication, we have presented a study on this type of QDs which is coated with different shells such as ZnSe or ZnTe [4]. Recently, Poplawsky et al. showed the role of the CdTeSe layer as a NIR photoactive layer in CdTe-based solar cells [5].

The purpose of this study is to use the colloidal chemistry method to fabricate CdTeSe quantum dots that crystallize at the zinc blende phase, at a suitable temperature ($< 300\text{ }^{\circ}\text{C}$), with absorption and emission bands that expand up to the near-infrared (NIR) region, to be applied in solar cells. Detailed results on crystal structure characteristics of CdTeSe obtained via the X-Ray diffraction (XRD) and Raman scattering methods showed that two characteristic LO phonon lines, CdTe-like and CdSe-like, are usable to identify the formation of CdTeSe alloy phase and evaluate the Te/Se content ratio in the sample. In this paper, we present in detail results on the fabrication of CdTeSe samples with unchanging initial Cd:Te:Se molar ratio, but changing growing environment and temperature. At optimal growing temperature, we varied the initial Te/Se ratio to observe changes, if any, on the shape, size and optical properties of CdTe_xSe_{1-x} QDs when x changes. The CdTeSe QDs obtained from this optimal fabrication condition all have zinc blende crystal structure. Their absorption and emission spectra expand up to near 900 nm where none of the component semiconductors CdTe and CdSe cannot reach, which is suitable for application in solar cells.

2. MATERIALS AND METHODS

We used the following reagents (from Aldrich): cadmium acetate dihydrate (Cd(Ac)₂·2H₂O, 99.9 %) as a source of Cd, zinc acetate (Zn(Ac)₂, 99.9 %) as a source of Zn, elemental selenium powder (Se, 99.99 %) as a source of Se, elemental tellurium powder (Te, 99.99 %) as a source of Te, oleic acid (OA, 90 %) and oleylamine (OLA, 90 %) as surface ligands, 1-octadecene (ODE, 90%) and trioctylphosphine (TOP, 90 %) as the reaction medium. All chemicals were used without further purification.

2.1. Synthesis method of CdTeSe, CdTe_xSe_{1-x}, CdTeSe/ZnSe and CdTeSe/ZnTe QDs

The fabrication method of CdTeSe ternary QDs have been described in [4, 6 - 8]. In this study, we have used two different mixtures which are OA-ODE-OLA-TOP and OA-ODE-TOP to create a media to fabricate and grow CdTeSe nanocrystals. Referring to the method reported in [9], we used OLA together with the OA-ODE-TOP mixture to make the Cd precursor. However, when CdTeSe samples were fabricated at temperatures lower than 230 °C, they became usable. When the CdTeSe growing temperature was higher, such as 240 °C, 260 °C, 280 °C, liquid QDs turned brown very quickly and were deposited at the bottom of the flask. Therefore, with CdTeSe QD samples fabricated at high temperatures ($> 230\text{ }^{\circ}\text{C}$), we used OA-ODE-TOP as the environment for growing nanocrystals.

To fabricate CdTeSe core QDs, it is first required to fabricate initial precursors. For fabrication of 1 mmol CdTeSe in OA-ODE environment with molar ratio Cd : Te : Se = 5 : 0.6 :

0.4, first we needed to fabricate precursor solutions. Briefly, we took 1.33 g (5 mmol) cadmium acetate dihydrate and dissolve in 1.59 (5 mmol) OA and 75 ml ODE. Cd precursor was fabricated by dissolving them in a mixture of OA and ODE. OLA is used with a mixture of OA and ODE to prepare Cd precursor for QD samples fabricated at low temperatures (< 220 °C). At higher temperatures (> 240 °C), QDs quickly change to brown and immediately deposit. TOP-Se and TOP-Te precursors were fabricated by dissolving 0.0316 g (0.4 mmol) powder Se in 0.4 ml TOP and 0.0766 g (0.6 mmol) Te metals in 1 ml TOP, at 80 - 100 °C, in N₂ gas. Then, an amount of these precursors were brought into a reaction flask. We quickly injected the mixed precursors TOP-Se and TOP-Te into a three-neck flask containing the Cd precursor solution at 120 °C for 1 h, in N₂ gas. We increased the temperature gradually to 180 °C, 200 °C, 220 °C, 240 °C, 260 °C or 280 °C, depending on the desired nanocrystal growing temperature for each sample and kept it stable at each temperature for a period from 10 mins, while vigorously stirring the reacting solution, to create nanoparticle seeds and grow them. Then we allowed the solution to cool slowly while stirring with a magnetic stirrer. These samples were simply abbreviated as CdTeSe.

For the fabrication of the sample series CdTe_xSe_{1-x} we used a Te fraction (Te/(Te/Se) ratio) of $x = 0, 0.2, 0.4, 0.5, 0.6, 0.8$ and 1, with the precursors fabricated in a similar way as above – note that x is the fraction of Te reagent, not the fraction of Te inside the CdTeSe QD. The nanocrystals growing temperature was kept at 260 °C for 10 min.

Similar to fabricating the core when coating ZnSe for the CdTeSe cores, we also had to fabricate the precursors for the shell material. The process of preparing the precursors for Zn and Se is completely identical to that of initial Cd precursor and Se-TOP. An monolayer (ML) thickness is based on the lattice constant a of ZnSe crystals, depending on the type of shell. The masses of Zn and Se were calculated for 1 ML, 2 ML, 4 ML and 6 ML of ZnSe. The molar ratio of Zn:Se was 1:1.

We coated the CdTeSe cores fabricated at 260°C. We took 46.4 ml of the CdTeSe core QD solution (~1.6 mmol) and poured it into a three-necked flask, then quickly raised the temperature to 230 °C. At this temperature, we injected 2.8 ml of the Zn precursor solution with haste (corresponding to a monolayer of Zn ions) and stirred vigorously for 15 min. Afterward, we injected 1.3 ml of TOP-Se and stirred vigorously for 15 min to grow the shell. Next, we took out 25 ml of the solution containing QDs, which was comprised of CdTeSe/ZnTe 1 ML. With the remaining volume, we continued to inject 1.4 ml of Zn precursor, stirred vigorously for 10 min, then injected TOP-Se (0.7 ml), stirred vigorously to grow the ZnTe particles' shell for 15 min. At this point, CdTeSe/ZnTe 2 ML was obtained. We performed the same operations when coating ZnSe for CdTeSe QD cores to form CdTeSe/ZnSe with higher thickness such as 4 and 6 ML.

All alloyed ternary quantum dots were purified by several rounds of precipitation and centrifugation, then stored at room temperature for later characterization and use.

2.2. Characterization of alloyed CdTeSe ternary quantum dots

The size of the CdTeSe QDs was determined by transmission electron microscopy (TEM) with a JEOL JEM 1010 microscope operating at 100 kV. Powder X-ray diffraction (XRD, Siemens D5005) was used to confirm the wurtzite (w) or zinc-blende (zb) crystalline structure.

The ultraviolet-visible (UV-Vis) absorption spectra of the QDs in toluene were scanned within the wavelength range of 200–1000 nm using a Shimadzu (UV-1800) UV-Vis

spectrophotometer. All UV–Vis measurements were performed at 25 °C and automatically corrected for the solvent medium.

The photoluminescent (PL) spectra measurement was carried out on a Fluorolog-322 system (Yvon) by using Xenon 450W light, whose detector is a photomultiplier, with measuring range from 250 - 1000 nm.

The QD samples were analyzed by Micro Raman spectroscopy (XploRA- Horiba) using 532 nm (25 mW) excitation line from a diode-pumped 100 mW solid state laser to analyze the vibration bonds and their Raman frequencies. 10× Objectives were used to focus the excitation laser light on the right spot - 1 μm in size - of the investigated samples. The spectral resolution was 2 cm⁻¹. The acquisition time ranged from 30 s to 120 s, but normally it's 30 s. The system uses a Charge Coupled Device (CCD) receiver with four gratings - 600, 1200, 1800 and 2400 gr./mm - that measures from 100- 4000 cm⁻¹. For Raman and PL measurements at low temperature (liquid nitrogen temperature) we combined the Raman XploRA Plus system with the THMS 600 temperature control system of Linkam company that can measure from liquid nitrogen temperature (77 K) to the room temperature (293 K). The whole THMS6 00 system is controlled by the Lynksys-32 software with temperature adjustment of 0.1K.

For powder X-ray diffraction (XRD) and Raman measurements, the CdTeSe QD samples were used in solid form. These samples were purified by washing thrice with isopropanol. The sample that was used for TEM, absorption and fluorescence spectra recording was in solution in toluene, after being purified of ligands and remaining excess substances after QD fabrication.

3. RESULTS AND DISCUSSION

3.1. Study on the fabricating temperature of alloyed CdTeSe QDs

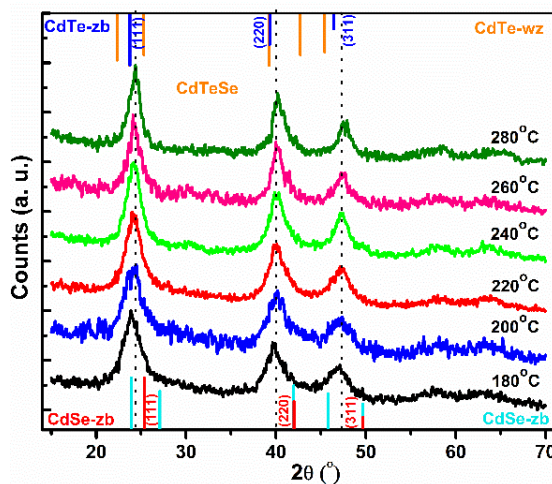


Figure 1. Powder XRD patterns of CdTeSe ternary QDs prepared at different temperatures from 180 °C to 280 °C (for Cd: Te: Se = 5: 0.6: 0.4). The tabulated values of the bulk diffraction peaks for zinc blend (zb) CdTe, (zb) CdSe are shown.

CdTeSe nanocrystal samples were fabricated with an initial molar ratio of 5Cd : 0.6Te : 0.4Se, a changing of growing temperature from 180 °C to 280°C, and their crystalline phase was identified by the XRD method. Figure 1 is the XRD diagrams of this sample series. From the diagrams, we can see that all the XRD patterns exhibit three diffraction peaks whose positions

lie between respective peaks of two zb crystalline phases of CdSe and CdTe. The peaks are located at angles 2θ corresponding to (111), (220) and (311) lattice planes. This shows that the quantum dots have formed in the form of alloyed CdTeSe. When the fabricating temperature rises from 180 °C to 260 °C, the diffraction lines remain at the same positions. However, for the samples fabricated at 280 °C, the two peaks (220) and (310) are shifted towards higher angles 2θ . The shift in position of these lines when the CdTeSe sample is grown at a high temperature (280 °C) can be explained as the following. When the temperature is low, Te reacts first due to its stronger activity, initially creating Cd-Te bonds. Data on the zb-CdTeSe crystalline lattice parameter can be changed slightly when sample fabricating temperature is changed from 180 °C to 260 °C. The lattice constant of zb-CdTe is $a_{\text{CdTe}} = 6.48 \text{ \AA}$ and zb-CdSe is $a_{\text{CdSe}} = 6.05 \text{ \AA}$ [10]. In zb-CdTe crystal, the Cd-Te bond length is 2.81 Å, while in zb-CdSe, the Cd-Se bond length is 2.62 Å [11]. When the temperature is increased to 280 °C, most of the Te ions would have reacted, and Se ions would keep linked with Cd ions creating many Cd-Se bonds, shrinking the crystal lattice structure in the same tendency with zb-CdSe and shifting diffraction lines towards angles 2θ higher - as observed on the XRD patterns of sample created at 280 °C.

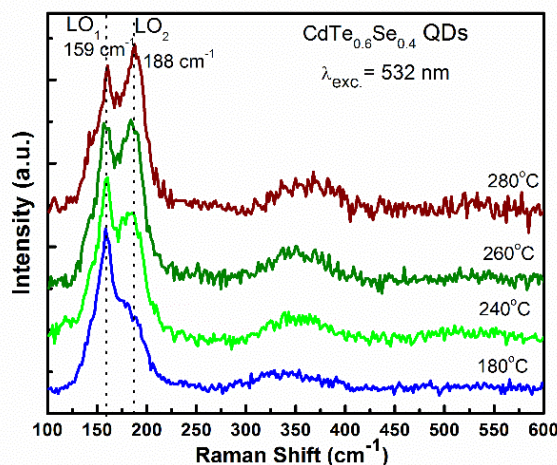


Figure 2. Raman spectra of CdTeSe QDs fabricated at different temperatures.

Figure 2 is the Raman spectra of CdTeSe QD samples fabricated at different temperatures recorded with the micro Raman XploRa Plus using 532 nm laser as an excitation source. On the Raman spectra, two main vibrational lines appearing at 159 cm^{-1} and 188 cm^{-1} are characteristic for phonon vibrations of CdTe-like LO and CdSe-like LO modes as observed in [12, 13]. When the fabricating temperature is as low as 180 °C, the intensity of the CdTe-like LO phonon is much higher compared to that of the CdSe-like LO phonon, proving that there are more formations of Cd-Te bonds than of Cd-Se in the alloy. When the nanocrystals growing temperature increases, the CdSe-like LO phonon line intensity increases gradually. This might be because when temperature rises, Se atoms have more chances to react to Cd atoms, creating an alloy crystal net of larger size. With the samples fabricated at high temperatures, for example 260 °C, we can see on the Raman spectra that the intensity of CdSe-like LO phonon line is a bit higher than that of CdTe-like LO phonon. For the fabricating temperature of 280 °C, the intensity ration of these two lines in the CdTeSe QD sample are still the same as in alloy QDs grown at 260 °C, but on the CdSe-like LO phonon band there appears another small line. This proves that a new, very substance might be formed in alloyed CdTeSe QDs that the amount is too small to be identified. The intensity ratios of CdTe-like LO/CdSe-like LO of these samples

are almost the same. This can correspond to the amount of Se in this sample being a bit higher than the amount of Te. At this moment, we could assume that the amount of Se brought into the reaction flask had completely reacted to Cd. Therefore, based on the Raman spectra, combined with the results obtained from XRD, we decided to use 260 °C as the optimal temperature to fabricate alloyed CdTeSe QDs.

3.2. Study on the change in Te/Se molar ratio

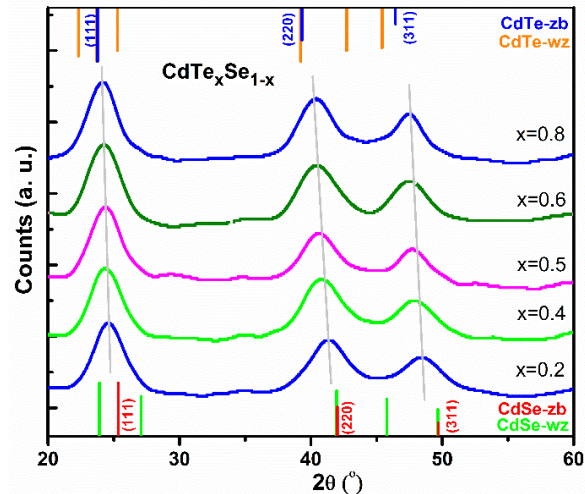


Figure 3. Powder XRD patterns of $\text{CdTe}_x\text{Se}_{1-x}$ ternary QDs prepared at temperature 26 °C (for $x = 0.2, 0.4, 0.5, 0.6$ and 0.8). The tabulated values of the bulk diffraction peaks for zinc blend (zb) CdTe, (zb) CdSe are shown

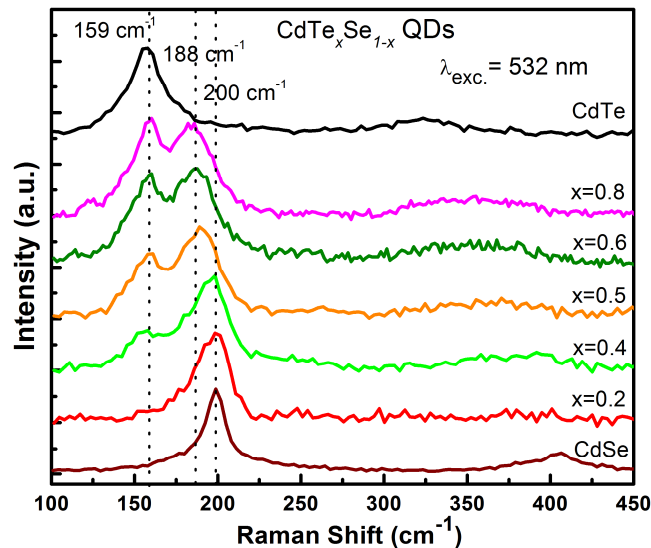


Figure 4. Raman spectra of $\text{CdTe}_x\text{Se}_{1-x}$ QDs prepared at temperature 260°C, $x = 0.2, 0.4, 0.5, 0.6$ and 0.8 .

From the XRD diagram in Fig. 3 we can see that: i) When the Te amount (x) changes, the diffraction lines lie between standard lines of two zb composition crystalline phases CdSe and

CdTe, meaning the CdTeSe nanocrystals fabricated are alloyed ternary QDs; ii) When the ‘x’ content rises from 0.2 to 0.8, the position of these lines are also shifted from near the characteristic lines of zb CdSe to near characteristic lines of zb CdTe. This proves that the fabricated QDs were three-component CdTeSe and they become more Te rich with increased Te amount.

The Raman spectra of $\text{CdTe}_x\text{Se}_{1-x}$ QD samples with changing Te amount (x) are shown in Fig. 4. From the spectra, we can see that when the Te amount changes, the intensity of vibrational lines at frequency 159 cm^{-1} (characteristic for CdTe-like LO phonon vibrational mode) and at 188 cm^{-1} (characteristic for CdSe-like LO phonon vibrational mode) changes. When the Te amount is low ($x = 0.2$), the nanocrystals have formed in alloy form, but due to the low amount of Te, the QDs mainly contain Se and on the Raman spectra only appears a peak at 200 cm^{-1} , characteristic for CdSe LO phonon mode. When the amount of Te rises to 0.4, on the Raman spectra appears a mode at 159 cm^{-1} , which is characteristic for CdTe-like LO phonon mode along side CdSe LO phonon mode at 200 cm^{-1} . When the Te amount is increased to 0.5, 0.6 and 0.8, peak intensity at 159 cm^{-1} also increases clearly, and the position of the second peak shifts, which is the peak characteristic for CdSe LO phonon mode from 200 cm^{-1} to a lower frequency - 188 cm^{-1} . When the Te amount is low, the position of CdSe LO phonon mode band is located at 200 cm^{-1} . When the Te amount increases from 0.5 and higher, the CdSe LO phonon position is shifted towards lower frequencies, 188 cm^{-1} in this case, and this peak is assigned as the zb CdSe-like LO phonon mode. This can be explain as when the Te amount is low (high Se amount in the CdTeSe alloyed molecule), in the alloy can exist wurtzite (w) hexagonal structure phase; and when the Te amount is high (low Se amount), the CdTeSe alloy change to a cubic zb phase [12, 13].

3.3. Study on the core/shell structure QDs

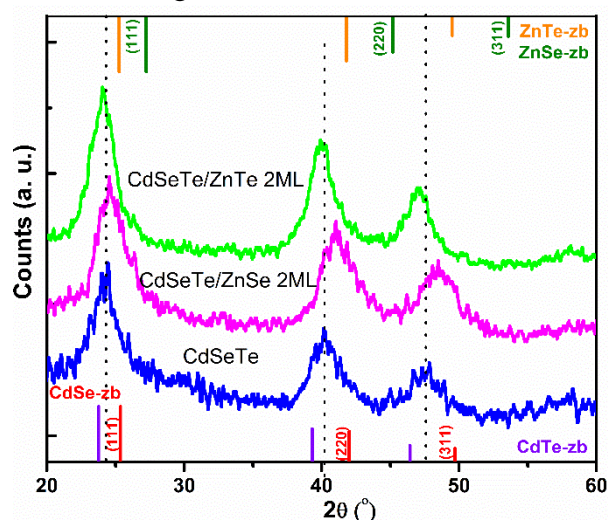


Figure 5. XRD diagram of CdTeSe core sample, and two core/shell samples CdTeSe/ZnSe 2ML and CdTeSe/ZnTe 2ML.

To protect CdTeSe QDs from surface defects, we wrapped a layer of ZnSe or ZnTe around them, with the thickness of about 2 monolayers (2 ML) of the shell material. Figure 5 is the

XRD diagram of CdTeSe core sample, CdTeSe/ZnSe 2 ML and CdTeSe/ZnTe core/shell. On the XRD diagram of CdTeSe core sample, we observed three peaks whose maximum peak lie between the positions of two standard lines of compositional crystalline phases which are zb CdTe and zb CdSe. This proves that the fabricated CdTeSe QDs in this study are of alloy. When coated with a thin ZnSe 2 ML shell, all three diffraction peaks of this sample are shifted towards higher angles 2θ , showing that the crystalline phase shifts towards near CdSe crystalline phase. When coated with ZnTe, only two diffraction peaks at (220) and (311) are shifted towards lower angles 2θ .

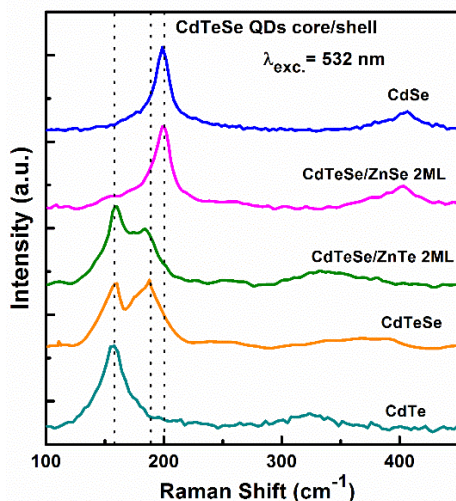


Figure 6. Raman spectra of CdTeSe core QDs, CdTeSe/ZnSe 2ML and CdTeSe/ZnTe 2ML core/shell structure.

We also recorded the Raman spectra of the core, core/shell QDs and the results are presented in Fig. 6. The Raman spectra show that the core of CdTeSe QDs revealed two peaks at 159 cm^{-1} and 188 cm^{-1} , which are typical lines for vibrational LO phonon modes such as CdTe-like LO phonon and zbCdSe-like LO phonon. When coating these QDs with a thin shell of ZnSe (2ML), we observed vibration lines at 200 cm^{-1} , which is characteristic of the vibrational LO phonon mode of w-CdSe. Besides these vibration lines, a peak with very weak intensity is located at 159 cm^{-1} frequency, characteristic of the CdTe-like LO vibrational mode. This can be interpreted as when using a thin shell of ZnSe, because the amount of Cd precursor moles is more than 10 times the total amount of Te and Se, the amount of remaining Cd in the reaction vessel reacted to create the core remains, so this amount of Cd reacted with Se in the coated shell, forming Cd-Se bonds, creating a very-CdSe-rich shell. This makes the intensity ratio of the characteristic vibration line of the w CdSe LO phonon much stronger than CdTe-like LO phonon. However, when using ZnTe shell, due to Te being chemically much stronger than Se, adding Te to create the shell immediately produced Cd-Te bonds, increasing the intensity of the vibration of CdTe-like LO phonon at 159 cm^{-1} , which helped us to obtain CdTe-rich QDs.

To survey the change in shape and size of particles before and after coating, we have taken TEM images of fabricated samples. Figure 7 is the TEM images of CdTeSe core sample and the two core/shell samples CdTeSe/ZnSe and CdTeSe/ZnTe. From Fig. 7 we can see that the samples exhibit uniform different sizes but not very spherical shape. With the ImageJ software we were able to calculate the particle average size and list the data in Table 1.

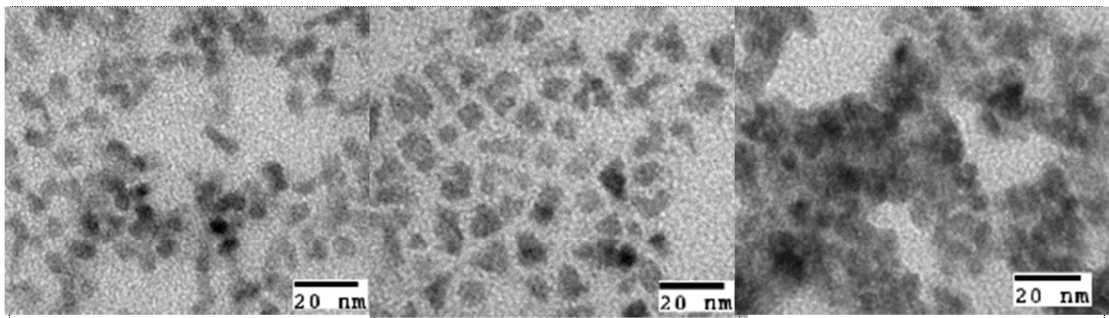


Figure 7. TEM image of the CdTeSe core QDs, CdTeSe/ZnSe 2ML and CdTeSe/ZnTe 2ML core/shell QDs (from left to right).

Table 1. Particle size of shell and core/shell samples.

Sample	CdTeSe	CdTeSe/ZnSe 2ML	CdTeSe/ZnTe 2ML
Average size	6.3 nm	8.3 nm	8.0 nm

3.4. Optical properties of alloyed CdTeSe QDs

From the fluorescent spectra (Fig. 8) we can see that for CdTeSe QDs, when fabricated at a low temperature (180 °C), the emission maximum is at 716 nm. However, for CdTeSe QDs fabricated at higher temperatures, up to 260 °C, the emission maximum is at 860 nm, and the emission band maximum almost does not move when temperature is increased to 280 °C. The shift in emission maximum peak towards long wavelengths can be originate from the increase in particle size of QDs, also because of the change in alloy composition of CdTeSe QDs, as observed on the Raman spectra. From the absorption spectra (inset image in Fig. 8), we can see that when the temperature is increased from 180 °C to 260 °C, the absorption edge is shifted towards longer wavelengths, located in the NIR area. This result shows that the fabricated CdTeSe QDs can be used as absorbing QDs in solar cells.

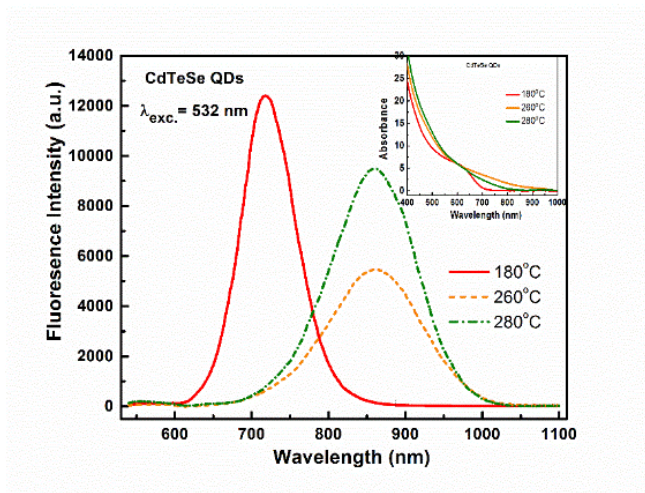


Figure 8. The emission spectra of QD samples grown at different temperatures: 180 °C, 260 °C and 280 °C. Inset is the absorption spectra of these QDs samples.

4. CONCLUSION

By the method of fabricating quantum dots at 260 °C in 10 minutes, alloyed CdTeSe QDs have been formed with good crystallization and zinc-blende single phase. Raman spectra results were completely in sync with XRD, therefore Raman spectra could be used as an effective tool to estimate the Se and Te compositions available in CdTeSe QDs. Studies on the absorption and emission spectra showed that the fabricated CdTeSe QDs were able to absorb and emit up to the ~ 900 nm region. The NIR absorbing properties make CdTeSe QDs usable in solar cells.

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