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Optimization of certain synthesis parameters for CdSe quantum dots and synthesis of CdSe–ZnSe nano-tetrapods through facile hot injection method

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ABSTRACT This paper reports a modified version of hot injection method for the synthesis of CdSe quantum dots and CdSe–ZnSe nano–tetrapod structures. In the present method, certain synthesis parameters such as injection temperature, growth temperature and precursor ratio were tuned to influence the growth kinetics and thereby the optical properties of the prepared CdSe QDs were enhanced. The influences of these parameters were studied and suitable conditions were optimized. Using the optimized parameters, CdSe–ZnSe nanostructures were synthesized with tetrapod morphology and their structural and optical properties were studied. The adopted modified version of synthesis is a facile method without stringent conditions such as inert atmosphere and therefore adaptable for the large-scale synthesis in industry.

KEYWORDS Growth kinetics, Hot injection, CdSe, CdSe–ZnSe, Nano-tetrapod.

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1. Introduction

Quantum dots (QDs) have attracted a great research interest around the globe for the last few decades due to their excellent tunability, versatility and chemical processibility [1–3]. Among II-VI QDs, cadmium selenide (CdSe) is a widely explored material for various applications [4,5]. The quality of CdSe QDs is greatly influenced by the precursor chemicals as well as the capping ligand used in the synthesis and other various synthesis parameters such as precursor ratio, ligand ratio, injection temperature, growth temperature, etc. By understanding the role of these parameters on the growth kinetics of the QDs, it can be optimized to yield highly monodisperse, emissive and smaller QDs suitable for various applications such as fluorescence labelling, light emitting devices, etc.

In the early '90s Murray et. al., reported the synthesis of good quality CdSe QDs using dimethyl cadmium as cadmium (Cd) precursor and trioctylphosphine oxide (TOPO) as capping ligand [5,6]. But the chemicals used in these syntheses such as dimethyl cadmium were pyrophoric, extremely toxic, explosive and unstable at room temperature. Stringent conditions such as inert atmosphere and equipment such as glove box, Schlenk lines etc., were mandatory to conduct these syntheses as the dimethyl cadmium releases a large amount of toxic and explosive gas at high temperatures [7,8]. To overcome this problem, Peng et. al., introduced a "greener" synthesis for CdSe QDs, using less toxic cadmium oxide (CdO) as a precursor of Cd [8]. This synthesis involves heating of Cd precursor upto a temperature around 300 °C under inert atmosphere followed by the injection of selenium (Se) precursor, termed as hot injection method. It is also called as kinetic growth method since the particle size increases with the progress in reaction time. Through this method, CdSe QDs were prepared in various solvents such as fatty acids, phosphonic acids, amines, phosphine oxides, and certain compositions of these solvents [7–10]. Nordell et. al., modified this method to an easier, safe and facile synthesis of CdSe QDs dispensing inert atmosphere [11,12]. This facile method was further modified by our group, in which the large scale synthesis of a particular size of QDs was achieved [13]. This simple, large scale production method combined with the large scale purification process proposed by Lim et.al, can be adopted for industrial and R&D applications of CdSe QDs [13,14].

Various morphologies of nanostructures such as dot, rod, tetrapod, etc., facilitate researchers to further manipulate the optical and electronic properties of nanomaterials. Wang et al. reported the synthesis of CdSe nano-tetrapods by incorporating HCl, $MnCl_2$, $FeCl_2$ and H_2SO_4 separately into a system of spherical CdSe QDs under inert atmosphere. Wang suggested that the protonic acidity in the precursor medium influences the yield of tetrapods [15]. Synthesis of

CdSe tetrapods were also reported through the addition of halides during the reaction [16]. Alivisatos et. al., synthesized rod, arrow, tetrapod and teardrop shaped CdSe nanocrystals using alkylphosphonic acid as a shape controlling factor [17].

The influence of oleic acid concentration as well as injection temperature on the growth kinetics was studied by Mulvaney et. al., and reported that decrease in oleic acid concentration result smaller dots. The synthesis was carried out under inert atmosphere with two injection temperatures, 275 and 265 °C, out of which smaller initial dot size was obtained at 275 °C [18]. The present work is the detailed study on the role of injection temperature, precursor ratio and growth temperature on the growth kinetics of CdSe QDs synthesized in ambient conditions. The effect of these parameters was studied to optimize the same and to tune the desirable optical properties with good repeatability. With the optimized conditions, CdSe quantum dots and CdSe-ZnSe nano-tetrapods of improved quality were synthesized through the facile hot injection method. In the synthesis of CdSe-ZnSe nano-tetrapods any additional chemicals were not used for shape controlling. This modified and optimized synthesis is a one-pot method excluding inert atmosphere and complex facilities so that the technique is adaptable to industry for the large scale synthesis of nanostructures.

2. Experimental

2.1. CdSe QDS

Cadmium precursor was prepared by adding CdO powder and oleic acid (OA) into octadecene (ODE). The Cd precursor was heated to a temperature higher than 150 °C to dissolve the brown Cd powder in ODE and to form a clear solution. The Se precursor, prepared by mixing Se powder and trioctylphosphine (TOP) in ODE was then swiftly injected into the Cd precursor at a specific temperature, called injection temperature. Immediately after the Se injection, CdSe QDs nucleate and begin to grow. As the Se precursor was injected, the temperature of the mixture decreases from the injection temperature. The resultant temperature, called growth temperature was maintained at a particular level for each synthesis of QD sample. Aliquots of samples collected at different intervals of time such as 5 s, 20 s, 60 s, and 120 s after the Se injection and it was found that the particle size increased as time progresses. The experiment was repeated under different conditions such as injection temperature, molar precursor ratio and growth temperature in order to optimize and yield good quality CdSe QDs.

2.2. CdSe–ZnSe nano-tetrapods

The synthesis of CdSe-ZnSe nano-tetrapods, the same hot injection method was adopted with ZnSe grown over the CdSe core through a single step-continuous chemical reaction. Thus, the extra step for the formation of ZnSe layer was skipped and the complexity of the synthesis was significantly reduced [3]. The only difference in the synthesis was the addition of Zn precursor in continuation to the nucleation of CdSe. The method can be extended to grow different shell layers one over the other to yield core-multishell system [19,20]. The values of parameters such as injection temperature, molar precursor ratio and growth temperature optimized for the synthesis of CdSe QDs were also used in the synthesis of CdSe-ZnSe nano-tetrapods. For this, Zn acetate (1 mmol) is mixed with 3 ml of OA and 6 ml of ODE to form the Zn precursor. For the synthesis of CdSe, the optimum identified value of Cd and Se precursors were selected. Selenium precursor was prepared by mixing 5 mmol Se powder, 4 ml of TOPO and 5 ml of ODE. For the Cd precursor, CdO powder (0.5 mmol), OA (3 ml), and ODE (10 ml) were stirred thoroughly and heated up to 225 °C to obtain a clear solution. At this temperature, the Se precursor was swiftly injected to initiate the nucleation of the CdSe QDs. After 5s of Se precursor injection, drops of Zn precursor were added at a rate of 2 ml per minute up to 4.5 min. In order to restrict the nucleation of ZnSe QDs, the precursor concentration was controlled during the injection process and the reaction was maintained in the kinetic growth regime. CdSe-ZnSe nano-tetrapods of different sizes were synthesized and samples were collected at 10 s, 1 min, 2 min, 5 min, and 8 min after the injection of Se.

For the particle size and morphological studies, TEM images were obtained from the JEOL/JEM 2100 transmission electron microscope. Absorption spectra were recorded using Jasco V-570 UV/Vis/IR spectrophotometer. It consists of two light sources, a Deuterium lamp of wavelength ranges from 190 nm to 350 nm and a Halogen lamp of wavelength ranges between 330 nm to 2500 nm. The monochromator contains two plane gratings of 1200 lines/mm for the UV-Vis region and 300 lines/mm for the NIR region. A Photomulitiplier tube and a PbS photocell are the detectors. Fluorescence spectra were recorded using Varian-Cary Eclipse fluorimeter in which a xenon lamp was used as an excitation source. The emission from the sample was collected by a PMT detector.

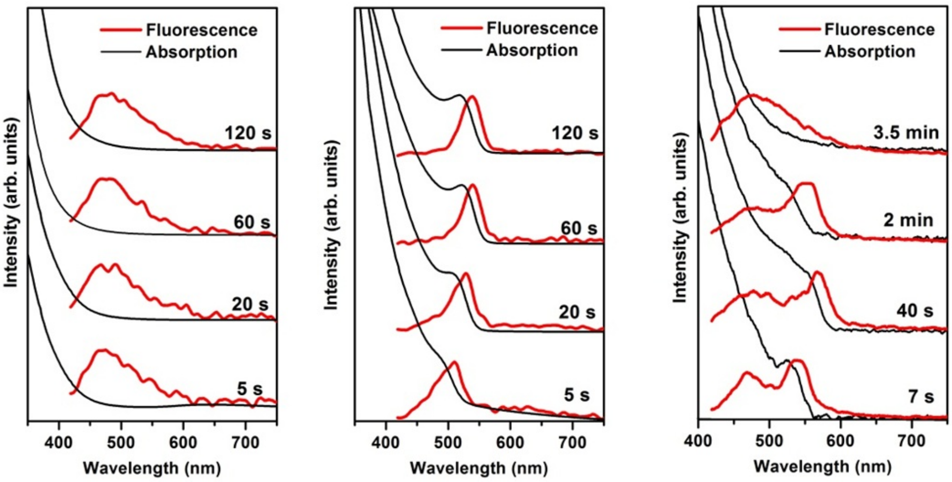
3. Results and discussion

3.1. Optimization of injection temperature

The injection temperature was varied from 160 to 240 °C at selected intervals. The temperatures above 240 °C were not considered since the optically clear Cd oleate solution becomes darker due to the high reaction of chemicals with atmospheric oxygen at elevated temperatures. Moreover, at these high temperatures, excess evaporation of the solvent leads to the drying of Cd oleate solute for which inert atmosphere is preferred.

Absorption and fluorescence spectra of the synthesized CdSe QDs at various injection temperatures at 160, 200, 215, 225 and 240 °C were recorded. The molar precursor ratio of Cd:Se was selected as 1:1 and the growth temperatures were maintained 15 °C lower to the corresponding injection temperatures. The excitation wavelength for recording the fluorescence spectra used was 400 nm in all the experiments.

The CdSe QDs synthesized at injection temperatures of 160 and 200 °C show similar absorption and fluorescence spectra as that of 215 °C represented in Fig. 1(a). In the hot injection method, in general, the particle size increases as the reaction time progresses and results in a redshift in both absorption and fluorescence spectra. But for these samples excitonic absorption peak and spectral redshift with an increase in time are not observed in the absorption spectra. Since at these injection temperatures, the amount of nucleated QDs is found to be low, giving a low concentration of samples. Also, the redshift is not presented in the fluorescence spectra, in which the peak wavelengths for all the samples are around 480 nm, depicts that the particles do not grow as reaction time progresses. Moreover, the fluorescence spectra are very broad with an FWHM around 100 nm, which clearly indicates the low quality of the QDs prepared at these injection temperatures.



(a) (b) (c)

FIG. 1. Normalized absorption and fluorescence spectra of CdSe QDs synthesized at injection temperatures, a) 215 °C and b) 225 °C and c) 240 °C. Excitation wavelength used to record fluorescence emission was 400 nm

In the absorption spectra of QDs synthesized at 225 °C injection temperature, given in Fig. 1(b), prominent excitonic peaks are observed with gradual redshift from 510 nm to 540 nm, with increase in reaction time. This clearly shows that several QDs are nucleated and result a sample of high concentration. Also, the size of the particle increases with reaction time. The fluorescence spectra are narrow with an FWHM in the range of 40 nm to 60 nm in the as-prepared QDs without post-synthesis purifications. Therefore, in this facile hot injection method an injection temperature around 225 °C is suitable for the synthesis of good quality QDs of different sizes.

The synthesis was repeated for the injection temperature of 240 °C and a large amount of QD nucleation and initial growth of particle size were observed similar to the synthesis at 225 °C. But the colour of the QD solution was found to shift from green to red and then back to greenish-yellow as time progresses. The absorption and fluorescence spectra shown in Fig. 1(c) agrees with the observations. The injection temperature at 240 °C results in the nucleation at different time and cause a growth of QDs of two different sizes, which is evident in the fluorescence spectra as two narrow peaks. For the sample at reaction time of 7s, the emission peak at 470 nm has an FWHM of about 60 nm and that at 540 nm has an FWHM of about 45 nm, which clearly shows that these are not a defect induced emission. Generally, in a defect driven emission, the spectrum will be as broad as 100 s of nm [13, 21]. In the case of the sample at reaction time of 3.5 min, the two peaks merge to form a single emission peak around 480 nm. These QDs require severe post-synthesis purification processes and need refinement in their size to obtain narrow fluorescence emission. By analyzing the absorption and fluorescence spectra it was clear that the quality of QDs deteriorated as the temperature increased beyond 225 °C. The sample prepared at 240 °C was compared with that of 225 °C and found that the former is of poor quality with that of the lower temperature and hence it was concluded to maintain facile hot injection method at 225 °C. Thus, good quality QDs having narrow FWHM in the emission spectra even without stringent condition of purification and refinement were obtained.

3.2. Optimization of precursor ratio

The influence of Cd:Se molar ratio on the growth kinetics of the CdSe QDs was also studied by synthesizing the QDs at an injection temperature of 225 °C and a growth temperature of 210 °C. The precursor ratio was varied as 2:1, 1:1, 1:5, and 1:10. The absorption spectra of the synthesized samples using these precursor ratios are given in Fig. 2.

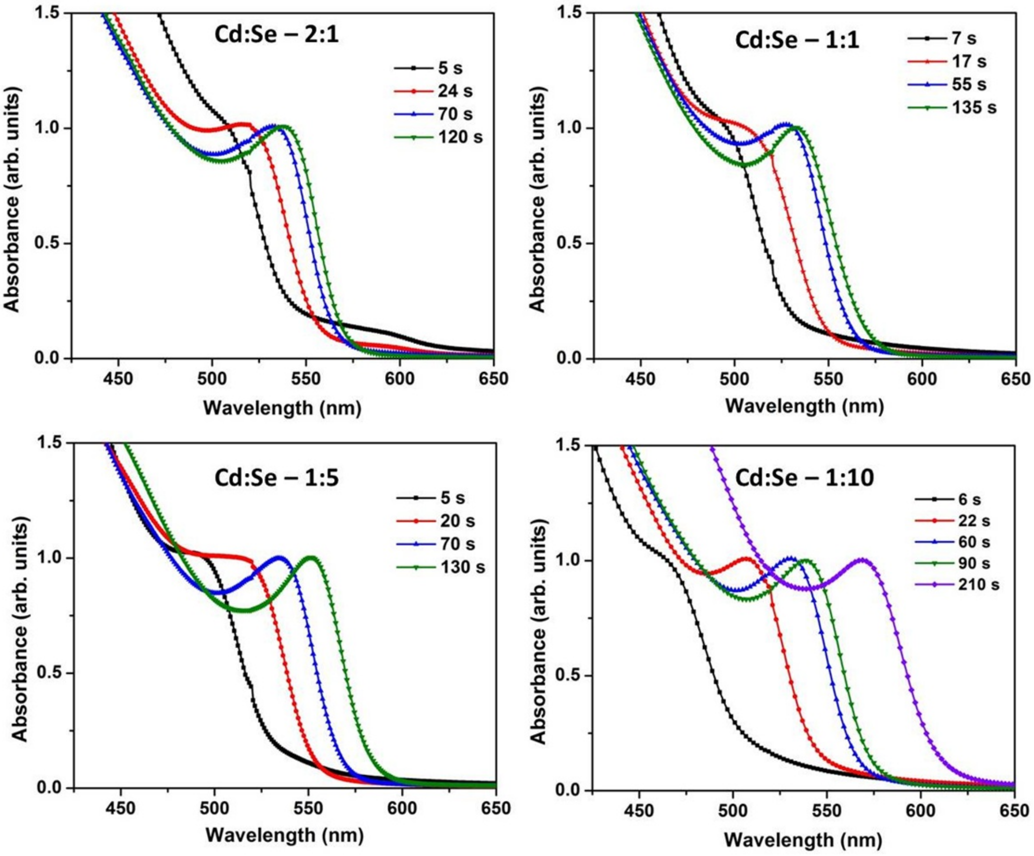


FIG. 2. Absorption spectra of CdSe QDs synthesized using different precursor ratio

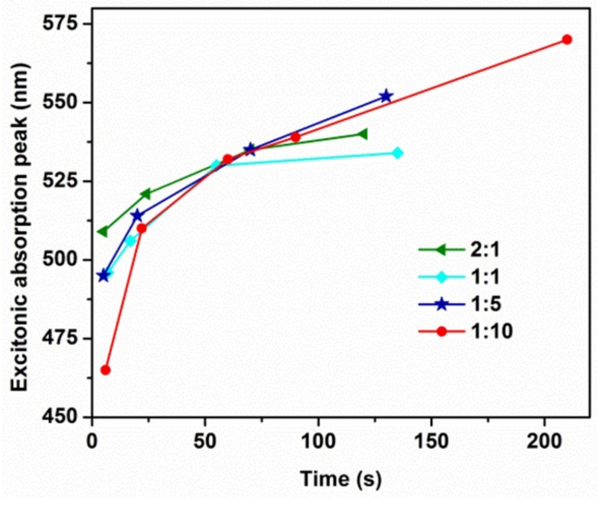


FIG. 3. Variation of excitonic absorption wavelength with increase in reaction time for different precursor molar ratios

The variation of excitonic absorption wavelength with increase in reaction time is presented in Fig. 3. As the reaction progresses, the particle size increases and the excitonic emission undergoes a redshift. In the sample with precursor ratio 2:1, the peak shifts from 510 nm to 540 nm and in samples 1:1 the peak shifts from 495 nm to 535 nm in 120 s and the particle size tends to saturate. For the sample 1:5 the particle size increases beyond 120s and the lowest excitonic peak obtained was around 495 nm. Sample 1:10 gives excitonic absorption peak around 465 nm as the particles are small and further grows similar to the sample 1:5, beyond 120 s. The results show that the presence of excess amount of Se facilitates the nucleation of smaller QDs and growth of different size ranges of QDs. Considering all these facts, for synthesis monodisperse QDs, the precursor ratio was optimized at 1:10.

3.3. Optimization of growth temperature

In order to study the influence of growth temperature on the growth kinetics, QDs were synthesized at growth temperatures of 180, 210, 215 and 225 °C. The optimized injection temperature and the molar ratio were fixed at 225 °C and 1:10, respectively, throughout the synthesis. The absorption spectra of the samples are given in Fig. 4. The plot of excitonic absorption peak Vs reaction time is presented in Fig. 5. This graph shows that the rate of particle growth increases with an increase in the growth temperature. The growth temperature below 210 °C the rate of growth is very slow and the particle size tends to saturate. The growth temperature between 210 and 215 °C gives a moderate rate of growth and therefore better control over the particle size. The rate of growth at 225 °C is very high so that the peak wavelength reaches 590 nm in 2 minutes.

CdSe QDs synthesized at 215 °C of growth temperature is selected for further analysis. The absorption and fluorescence spectra of these QDs are given in Fig. 6. Excitonic absorption peak varies from 480 nm to 584 nm during the particle size growth. The three samples with reaction time 2 s, 13 s, 35 s have good quality of fluorescence emission with FWHM of 35 nm, usually termed as colour purity. QDs having high colour purity are useful for labelling and light emitting devices [19, 22]. For the samples with reaction time 80 s and 120 s the FWHM gradually increases to 47 nm and 60 nm, respectively. For the final sample of 200 s, the FWHM is 125 nm broad and centered around 480 nm, due to the particle size broadening effect.

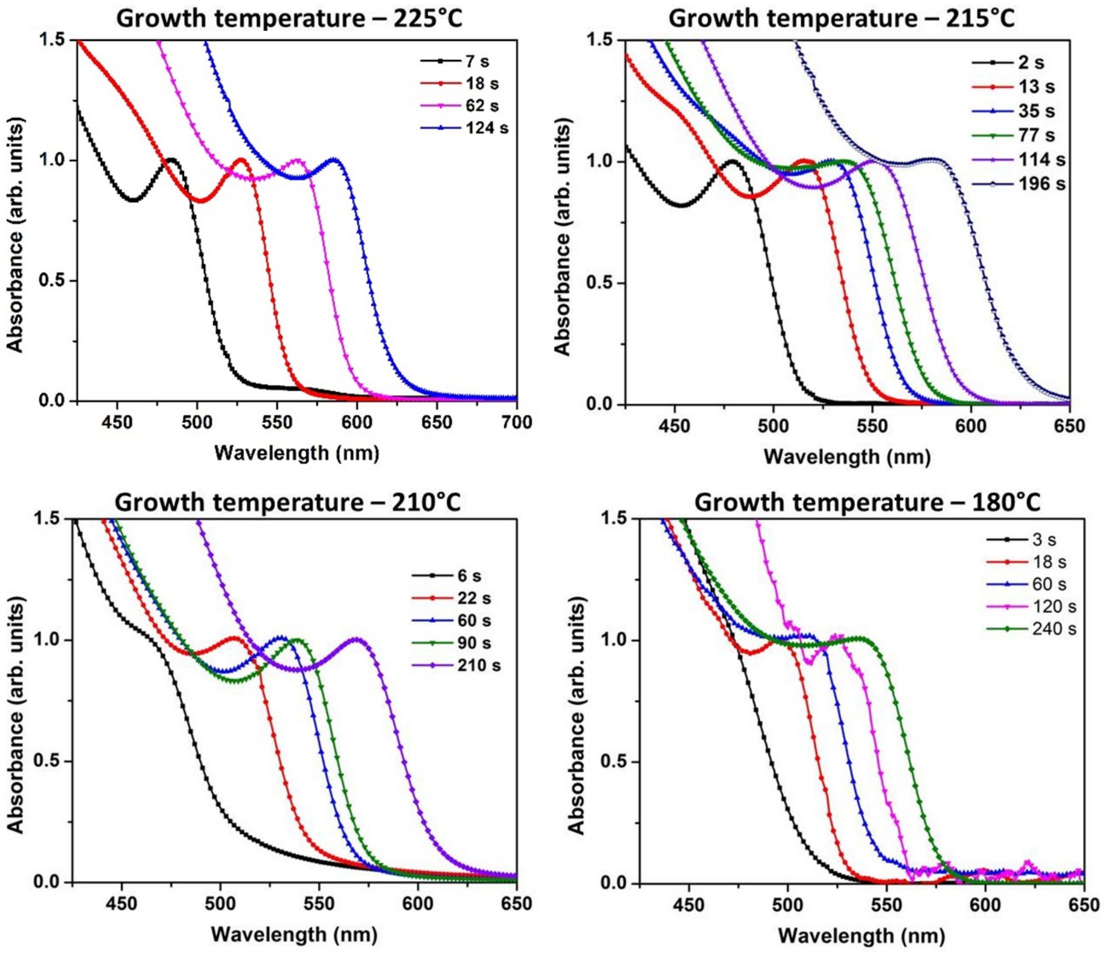


FIG. 4. Absorption spectra of CdSe QDs synthesized at different growth temperature

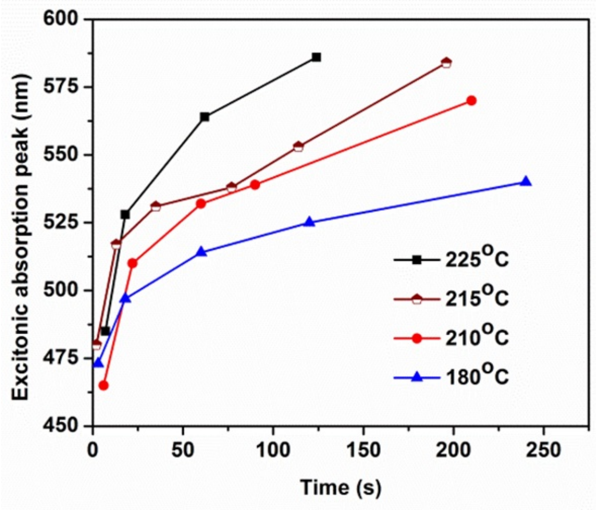


FIG. 5. Variation in excitonic absorption wavelength with reaction time for different growth temperatures

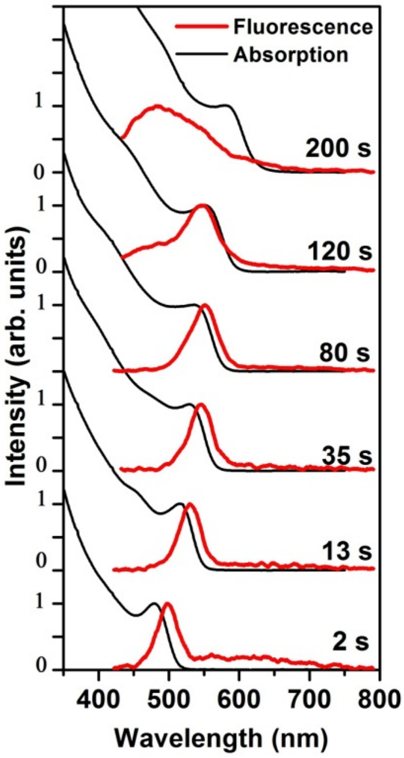


FIG. 6. Normalized absorption and fluorescence spectra of CdSe QDs synthesized at the growth temperature of 215 °C. For recording fluorescence spectra, an excitation wavelength of 400 nm was used

TEM image of one of the samples, taken at 35 s is shown in Fig. 7(a). The particles are nearly monodispersed and spherical in shape. The average particle size is measured to be 2.7 nm and the histogram of the particle size measurement is given in Fig. 7(b). The CdSe QDs synthesized with optimized conditions of injection temperature of 225 °C, precursor ratio 1:10 with growth temperature ranging from 210 to 215 °C have good optical properties.

3.4. CdSe–ZnSe nano-tetrapod structure

The optical properties of the CdSe-ZnSe nano-tetrapod was found to be depending significantly on the quality of the CdSe, and hence the same optimized conditions adopted for the CdSe QDs were used for the preparation of CdSe-ZnSe nanotetrapods [3]. With these conditions, CdSe–ZnSe nano-tetrapods of different sizes were synthesized and samples were collected at 10 s, 1 min, 2 min, 5 min, and 8 min after the Se injection time.

TEM images of representative samples at 10 s and 2 min are given in Fig. 8 and Fig. 9. The sample at 10 s consists of QDs with an average size of approximately 3 nm, exhibiting mostly spherical shapes. Some of the QDs exhibit a

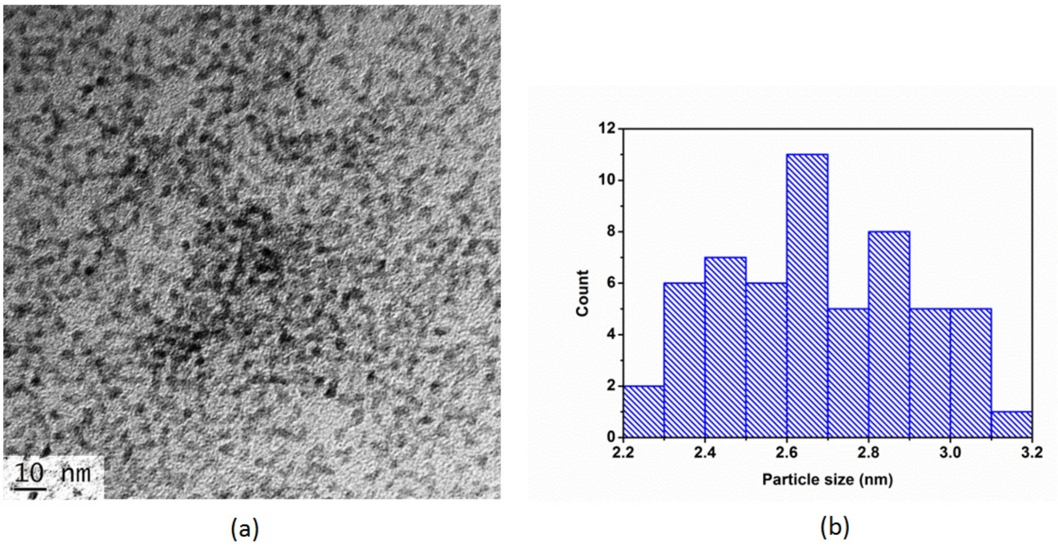


FIG. 7. a) TEM image b) histogram of particle size measurement of CdSe QD sample of 35 s, synthesized at the growth temperature of 215 °C

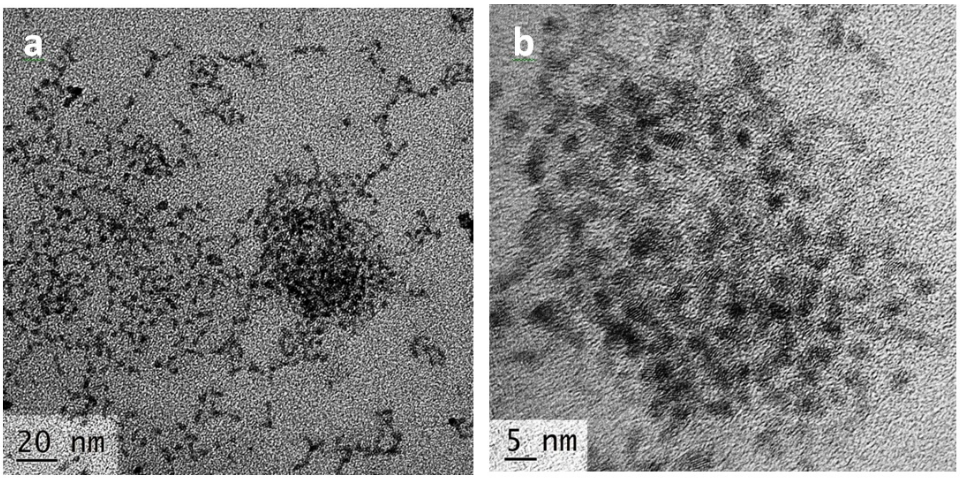


FIG. 8. a) TEM image and b) HRTEM image of sample at 10 s

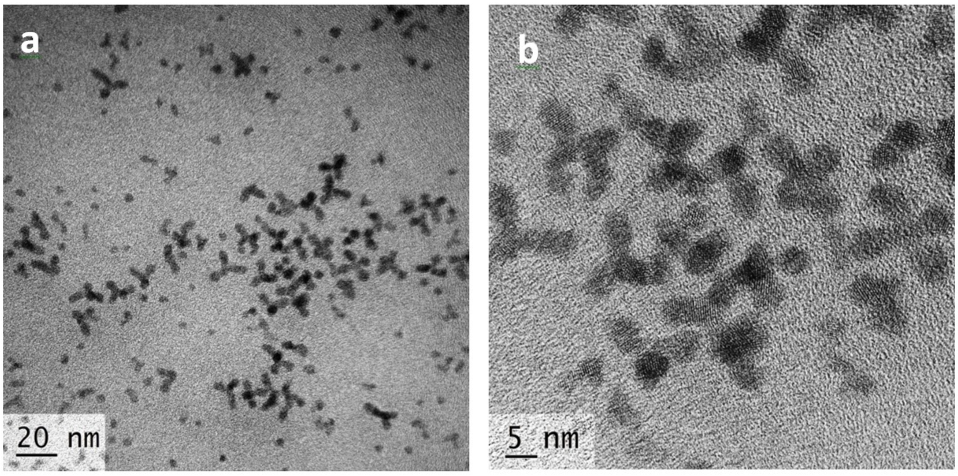


FIG. 9. a) TEM images and b) HRTEM images of sample at 2 min

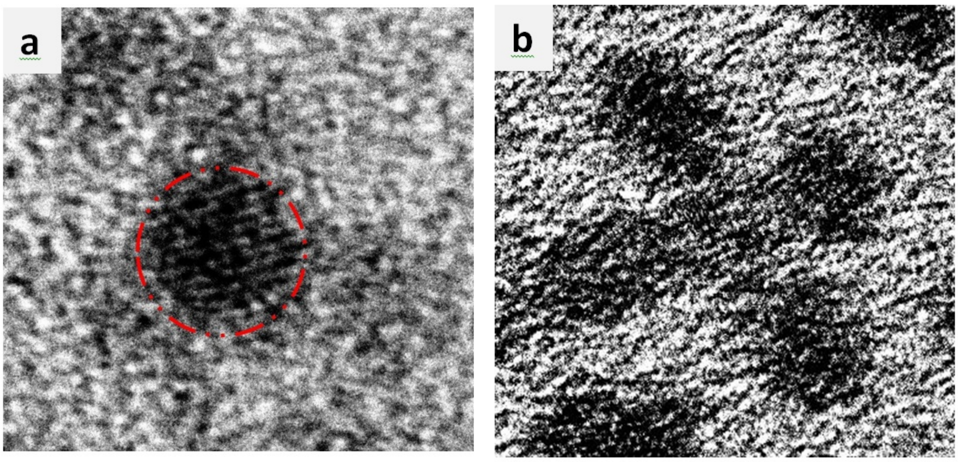


FIG. 10. HRTEM images of a) a single quantum dot from the sample at 10 s and b) a single nanotetrapod from the sample at 2 min

tendency of anisotropic growth. In contrast, sample at 2 min predominantly contains tetrapod structures characterized by a branch-lengths range from 7 nm to 9 nm and widths around 4 nm. The formation of the ZnSe outer layer is evident from the overlapping fringe patterns observed in Fig. 9b.). These patterns arise due to the difference in the d-spacing of CdSe and ZnSe, providing evidence for the presence of the ZnSe outer layer. To facilitate a better comparison, Fig. 10 displays images of a quantum dot from the sample at 10 s and a nanotetrapod from the sample at 2 min. The TEM images reveal a gradual transformation from a spherical to a tetrapod shape, providing clear evidence of the growth process.

Absorption and fluorescence spectra of samples of CdSe–ZnSe nano-tetrapods are given in Fig. 11. In the absorption spectra, second excitonic absorption peaks are clearly visible which indicate the good quality of the tetrapod structure. Fluorescence emissions of samples with 10 s, 1 min and 2 min are in the wavelength ranges from 510 nm to 550 nm, with an FWHM around 35 nm. These samples have colour purity similar to the CdSe QDs discussed in the previous section. Beyond 2 min, new emission at about 470 nm is observed while that at around 550 nm is absent. The tetrapod structures have a CdSe core of zinc blend structure with four wurtzite arms. The slow addition of Zn precursor causes growth of wurtzite structure of CdSe–ZnSe along the four (111) planes of the zinc blend structure [3,15,23]. The addition of zinc oleate eliminates the TOP surfactant and triggers an anisotropic growth by introducing a difference in surface energy. Once the surface coverage of ligands is reduced, arms of wurtzite structure are grown along the four (111) faces of the zinc blend CdSe 23. The four arms are grown through the C-axis of the wurtzite structure [15]. The synthesized CdSe–ZnSe nano-tetrapod structure through this facile hot injection method was completed under ambient conditions with optical properties like significant fluorescence emission.

4. Conclusion

In summary, we have investigated the facile hot injection synthesis method, excluding inert atmosphere and stringent chemical conditions, for the synthesis of CdSe QDs. The synthesis parameters such as injection temperature, precursor molar ratio and growth temperature suitable for the facile method were investigated and optimized. The optimized injection temperature was found to be 225 °C, in which highly monodisperse QDs were synthesized. The molar precursor ratio of 1:10 with excess Selenium was found to be optimum, which gives good quality of QDs in a wide range of particle sizes. The rate of growth of QDs was found to be directly related to the growth temperature. The growth temperature in the range from 210 to 215 °C gives moderate rate of growth, which gives better control over the particle size and improved repeatability. Using these optimized values, the facile method of synthesis of CdSe QDs was employed for the synthesis of CdSe–ZnSe nano-tetrapods of different sizes. Both the synthesized CdSe QDs and CdSe–ZnSe nano-tetrapods under the optimized conditions are exhibiting significant optical properties useful for the applications such as labelling, light emitting devices [19,22,24] etc.

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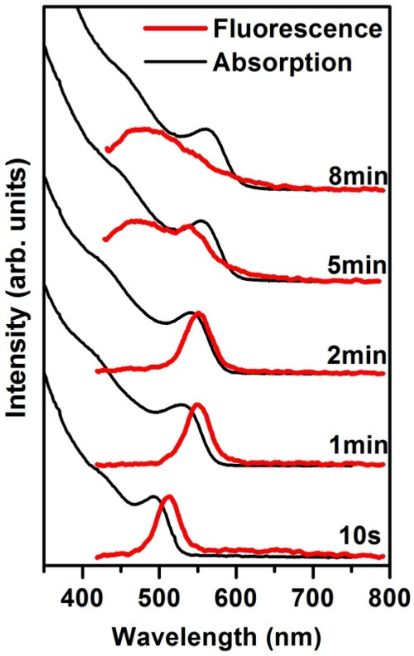

FIG. 11. Normalized absorption and fluorescence spectra of CdSe–ZnSe nano-tetrapod. Excitation wavelength of 400 nm was used for recording fluorescence spectra

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