

## Research Article

# Rapid Microwave Synthesis of Tunable Cadmium Selenide (CdSe) Quantum Dots for Optoelectronic Applications

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Quantum dots (QDs) are a hot topic in optoelectronic device research, due to tailorable absorption and emission properties. Unfortunately, the conventional methods of QD synthesis are hazardous and time-consuming. In this work, we present an alternative method of fabricating cadmium selenide (CdSe) QDs (via rapid microwave synthesis). This novel fabrication method provides a quick and efficient way to synthesize QDs that are almost identical to those commercially available. We also demonstrate the tuning of QD sizes by varying time and temperature during the growth process. Optical spectroscopy was used to measure the emission profile of QDs of various sizes. With ease repeatability, tunability, and scalability, this QD synthesis method can be integrated into a wide range of applications and optoelectronic devices.

## 1. Introduction

Under the nanotechnology umbrella, there are many materials that differ in behavior due to size and composition [1]. Typically known as nanoparticles, these materials have a variety of properties and have been integrated in many areas of current technology. In medicine, protein-filled nanoparticles have been used for drug delivery [2]. In manufacturing, nanoparticles are in sunscreens that protect from UV rays or clothing to prevent color fading [3]. Nanoparticles are even being utilized in environmental cleaning efforts, reducing contamination and pollution in water and air [4]. QDs (a special subset of crystalline nanoparticles ranging between 2 and 20 nm in size) can be used in all the aforementioned applications; however, conventional fabrication methods produce toxic byproducts and involved processing steps that are potentially harmful.

Here, we present a synthesis method that cuts fabrication time into a fraction and eliminates the need for 3-neck wet chemical synthesis under a fume hood. CdSe is an inorganic crystalline material whose QD properties are well understood

[5–20]. CdSe is popular due to its tailorable emission throughout the visible spectrum. In this study, we fabricated CdSe QDs using a one-step microwave and compared their optical properties to those commercially available. QD emission usually blue shift as the nanocrystallite size is reduced [21]. We used optical spectroscopy to quantify the change in the optical properties of the QDs as it relates to dot size. In this work, we first demonstrate the successful fabrication of QDs via rapid microwave synthesis. Then, we show the ability to tune the size of the QDs as mentioned through parameterization of microwave synthesis conditions. Our results show that we are able to obtain similar results to QDs synthesized via the traditional “hot injection method” but without the inherent health risks and byproducts.

## 2. Experimental Methods

Acetone was purchased from VWR Analytical. Cadmium oxide powder (99.5 wt. %) was purchased from Sigma-Aldrich. Oleic acid (tech, 90%), 1-octadecene (tech, 90%), selenium powder (-325 mesh, wt. 99.5%), and toluene



FIGURE 1: (a) CdSe quantum dots synthesized by the rapid microwave synthesis (from left to right) 509 nm, 523 nm, 554 nm, 581 nm, and 605 nm PL emission. (b) CdSe quantum dots of the same PL emission wavelengths under 365 nm UV light.

(ACS, 99.5%) were purchased from Alfa Aesar. Tri-n-octylphosphine (97% TOP) was purchased from Strem Chemicals. All materials were used as received. A recipe for one-pot microwave synthesis was adopted and modified from a conventional “hot-injection” synthesis method that utilizes a three-neck round bottom flask and heating mantle under an inert gas atmosphere [22]. Time and temperature play critical roles in nanocrystal size and emission [23]. Injections of TOP-Se at high nucleation temperatures describe the conventional “hot-injection” method commonly used in this area of research [24]. These methods are very time consuming, as heating mantles can take time to stabilize. These methods may also be harmful as CdO and Se powders are extremely toxic. The stoichiometric ratios of each material in this study were adopted from reference [24]. Stock solutions were prepared ahead of synthesis to reduce preparation time. All material preparation was done in a glove box. All heating was done on a hotplate outside of the glove box. Both precursors were left to cool in ambient temperatures in the glove box. Detailed preparation protocol is listed in the Supporting Information S1 Section a.

Synthesis was performed in an Anton Paar Monowave 300, a single mode microwave reactor with a small self-tuning cavity. For CdSe synthesis, power and stirring RPM for all samples was kept constant at 300 W and 600 RPM, respectively. Hold time and temperature was varied to produce CdSe QDs with different sizes and respective photoluminescence (PL) wavelengths. Time and temperature variations and their corresponding PL wavelengths are presented elsewhere. Following synthesis, the resulting QD solutions were cleaned and diluted for characterization. Detailed synthesis steps are listed in the Supporting Information S1 Section b.

Resulting QD colloidal solutions were transferred into the glove box and divided evenly into centrifuge tubes. Since CdSe is nonpolar, CdSe was “washed” in acetone by centrifugation [25]. Samples were taken out of the glove box and placed into a Beckman Coulter Allegra 25R Centrifuge. A VWR Analog Vortex Mixer was used to agitate CdSe for 10 seconds each. This washing process was repeated until the acetone was visibly transparent. Toluene was used a resuspension solvent for CdSe QDs, since toluene and CdSe are both nonpolar [26]. The CdSe QDs disperse into the toluene solvent creating a colloidal suspension. The concentration of the final solution is a function of the amount of toluene in the suspension; more toluene will produce a more diluted final

TABLE 1: Temperature and time combinations for microwave recipes and their respective PL wavelengths.

Temperature (°C)	Hold time (min)	PL wavelength (nm)
150°C	3:30	509 nm
175°C	1:30	523 nm
225°C	0:30	554 nm
225°C	5:00	581 nm
280°C	4:00	605 nm

solution. Final solutions were transferred back into the glove box. Each 2.5 mL colloidal solution (from the same time/temperature synthesis set) was combined and stored in a small amber vial for characterization. Detailed cleaning steps are listed in the Supporting Information S1 Section c.

A Jobin Yvon Horiba FluoroMax 3 Spectrometer was used to characterize the photoluminescence (PL) of CdSe colloidal quantum dots. An excitation wavelength of 400 nm was kept constant for all samples. Samples were measured in the visible range, from 400 to 700 nm at 1 nm increments. A PerkinElmer Lambda 1050 spectrophotometer was used to measure UV-VIS absorption data. Toluene was used as a reference for UV-VIS characterization since it is the suspension solvent. UV-VIS data was also collected in the visible region, from 400 to 700 nm at 1 nm increments. Photos of CdSe quantum dots were taken under both ambient and 365 nm UV light, respectively. Data and photos are presented in Results and Discussions.

### 3. Results and Discussions

Cadmium selenide (CdSe) colloidal QDs were successfully synthesized by rapid microwave synthesis and characterized using UV/Vis and PL spectroscopy. Figure 1 shows CdSe QDs with PL wavelengths of 509 nm, 523 nm, 554 nm, 581 nm, and 605 nm. Diameters of QDs for each emission are 4.6 nm, 4.8 nm, 5.4 nm, 6.2 nm, and 6.8 nm, respectively. The top image shows QD solutions in ambient light. The bottom image shows CdSe QDs under a 365 nm UV light. Table 1 shows the time and temperature combinations that were loaded into the Monowave 300 to synthesize CdSe QDs shown in Figure 1. Commercially available CdSe QDs have a stable range between 500 and 650 nm. The Monowave 300 has a temperature limit of 300°C. An infrared ruby thermometer can simultaneously monitor the temperature inside

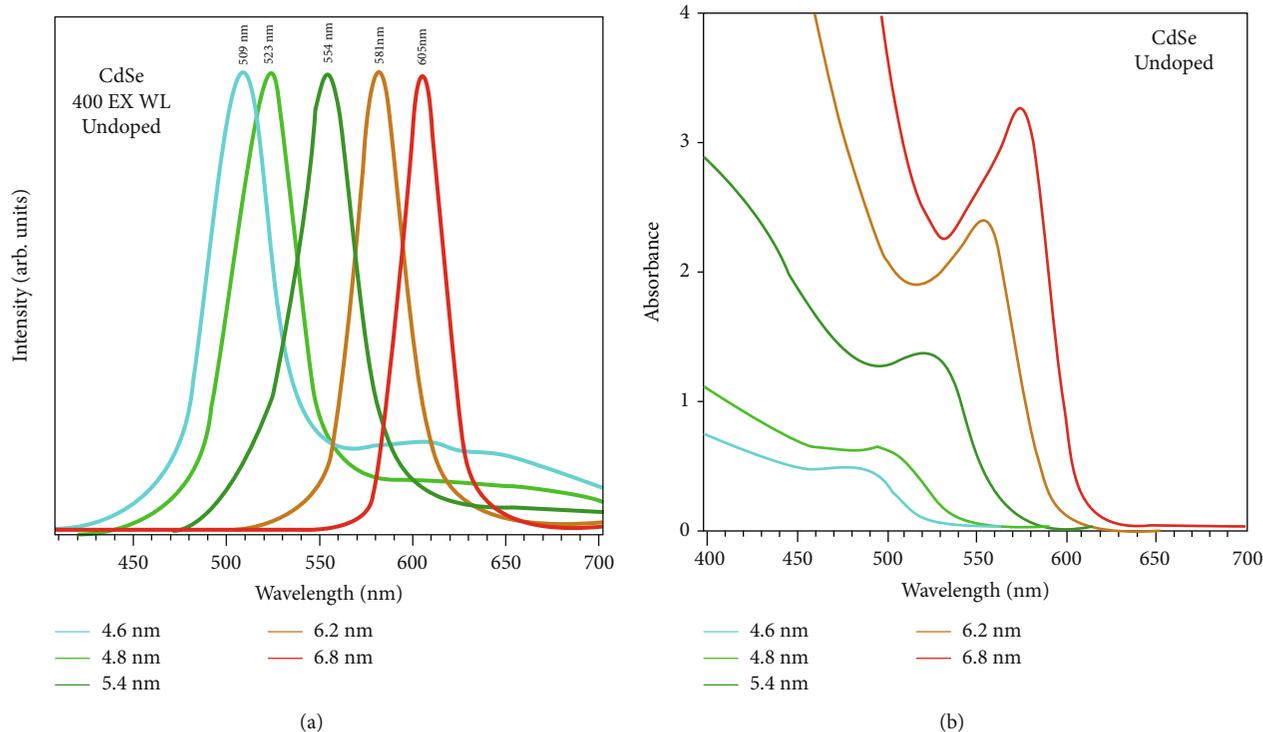


FIGURE 2: (a) Normalized photoluminescence (PL) data for CdSe colloidal quantum dots by rapid microwave synthesis. (b) UV-VIS absorption data for CdSe quantum dots by rapid microwave synthesis.

and outside of the reaction vial. As mentioned earlier, the exterior temperature of the vial was roughly 10–20°C higher than a ruby thermometer. A safety mechanism in the Monowave instrument will shut the system down if the maximum temperature limit is reached. Therefore, attempting any synthesis above a temperature of 285°C is at risk of interruption midsynthesis.

In our studies, CdSe QDs within commercially available properties were realized [27] (FWHM and emission wavelength). Figure 2 shows that CdSe QDs exhibit fluorescence properties in the visible range (~390–700 nm). Under 365 nm UV light, there is a clear shift from blue-cyan to orange-red as the PL shifts to each color's respective wavelength. CdSe QDs are highly reproducible using times and temperatures in Table 1. Figure 2 shows the PL and UV-VIS absorption data for CdSe colloids that were synthesized by rapid one-pot microwave synthesis. The PL data shown (top) is normalized to visually display the FWHM and peak emission wavelength. CdSe QDs synthesized in this study had emission in the range of 509–605 nm. The emission shifts towards the IR region while the band gap decreases, all properties tied to quantum confinement [28]. Time and temperature during synthesis are directly related to these properties, as the nucleation temperature dictates the initial nanocrystal formation [29]. Mao et al. describes a nanocrystal nucleation temperature around 225°C [30]. To obtain smaller quantum dots near the blue emission region, near the edge of the stability range of CdSe, the aliquot extractions must occur within 5 seconds following hot injection. This is due to rapid increases of nanocrystal size at high nucleation

TABLE 2: Visible range chart for color and respective PL wavelength range [31].

Color	Wavelength range (nm)
Red	625–740 nm
Orange	590–625 nm
Yellow	565–590 nm
Green	520–565 nm
Cyan	500–520 nm
Blue	435–500 nm
Violet	380–435 nm

temperatures. A lower temperature would increase the time for smaller size nanocrystals to be extracted. For this study, safe synthesis methods that utilized recipes with temperatures of 225°C did not yield a PL wavelength lower than approximately 550 nm (~5.4 nm diameter), even for synthesis times between 0 and 30 seconds. It is known that the wavelength range for the color green in the visible spectrum is roughly between 520 and 565 nm [31]. Therefore, the 225°C microwave synthesis temperature was incapable of producing smaller QDs closer to the blue region.

A number of syntheses were performed using various time and temperature (below 225°C) combinations to obtain QDs with a PL wavelength closer to the blue-cyan region. The blue region has a wavelength range of roughly 435–500 nm, as shown in Table 2. 500–520 nm is the cyan

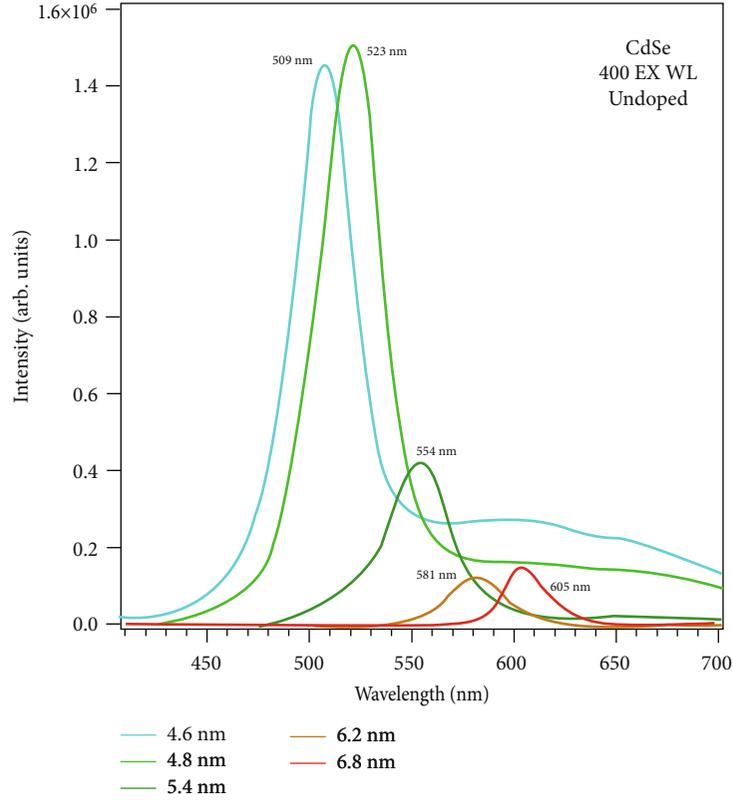


FIGURE 3: Photoluminescence (PL) data for undoped CdSe colloidal quantum dots by microwave synthesis.

TABLE 3: Temperature and time combinations, the respective PL wavelengths they produce, and the corresponding calculated size based on Brus equation for undoped CdSe [35].

Temperature (°C)	Hold time (min)	PL wavelength (nm)	Calculated radius (nm)	Calculated diameter (nm)
150°C	3:30	509 nm	2.3 nm	4.6 nm
175°C	1:30	523 nm	2.4 nm	4.8 nm
225°C	0:30	554 nm	2.7 nm	5.4 nm
225°C	5:00	581 nm	3.1 nm	6.2 nm
280°C	4:00	605 nm	3.4 nm	6.8 nm

region, a color that falls between blue and green and can be produced when the two are mixed together [32]. Synthesis temperatures lower than 225°C were used, and highly reproducible QDs in the blue-cyan region were realized. This is a safe alternative to the quick and highly toxic process of obtaining aliquots of smaller CdSe QDs by conventional synthesis methods. UV-VIS data in Figure 2(b) shows changes in optical absorption for CdSe QDs by rapid microwave synthesis. Larger absorption wavelengths equate to smaller band gaps and the lower energies. The UV-VIS data displays an increase in absorption wavelength as nucleation temperature and time increases, which is responsible for lower energy absorption and a smaller band gap.

Figure 3 shows raw PL data for CdSe at various time and temperature combinations. The plot also shows the fluorescence intensity of CdSe QDs decreases as nanocrystal size and PL wavelength increase. CdSe QDs around the blue

and cyan regions with wavelengths of 509 (~4.6 nm) and 523 nm (~4.8 nm) display higher PL intensities. This suggests that smaller QDs are well within the strong confinement region. Larger QDs from this study (~5.4-6.8 nm) in the green-red regions display much lower intensities. The CdSe Bohr radius has been reported between 5 and 6 nm [32–34]. This suggests that although these QDs are technically still within the strong confinement region, they lose some fluorescence efficiency as QD diameter increases and grows towards bulk properties.

Table 3 shows the size approximation calculations for CdSe QDs. Diameter approximations were calculated using PL data and the Brus equation,

$$R^2 = \frac{\hbar^2}{8E_x} \left( \frac{1}{m_e^*} + \frac{1}{m_h^*} \right), \quad (1)$$

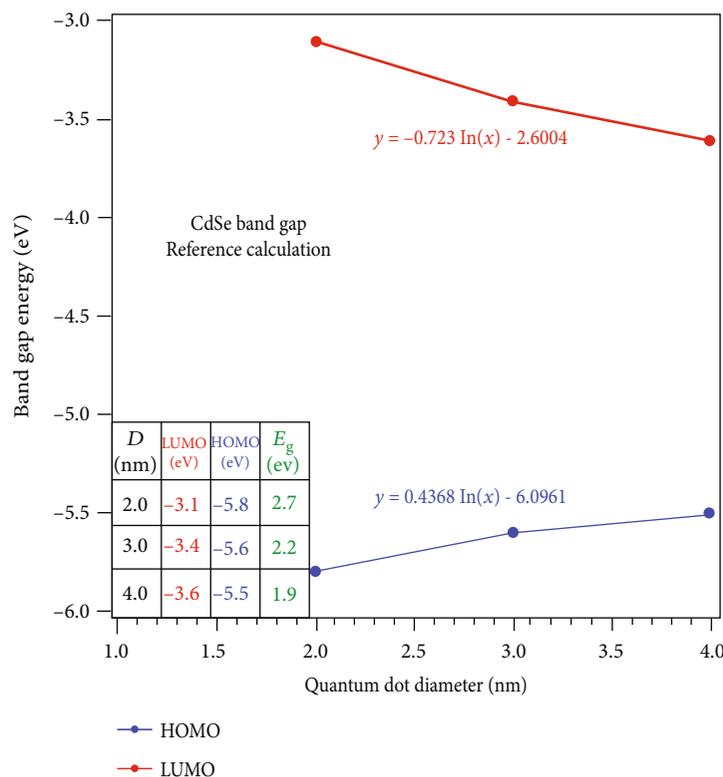


FIGURE 4: HOMO-LUMO level trend of CdSe in literature reference [39].

where  $E_x = E - E_g$  (CdSe),  $E_g$  (CdSe) = 1.74 eV,  $m_e^*$  (CdSe) =  $0.13m_o$  ( $m_o$  is the mass of free electron),  $m_h^*$  (CdSe) =  $0.45m_o$ , and  $h$  is Planck's constant [35, 36].  $E = hc/\lambda$  and is solved by using peak PL wavelengths for  $\lambda$ . These diameter size approximations explain bright fluorescence in the strong confinement region.

QDs may have large variance in size distribution [36]. A narrow size distribution is desired; yielding monodisperse QDs all close to the same size. At high cluster concentrations, critical size remains small and monodisperse size distribution can be achieved [37]. Over time, Ostwald ripening effects cause a decrease in cluster concentration and causes size distribution to widen [35–40].

Supporting information from a literature reference was used to calculate estimations for optical band gap of CdSe [41]. Tauc's equation based on UV-VIS absorption was compared to experimental cyclic voltammetry data for three quantum dot diameters [42–47]. A plot showing the trend of HOMO-LUMO levels based on diameter of CdSe in the literature reference is shown in Figure 4. Trend line equations for HOMO and LUMO levels of CdSe vs. diameter were generated from information gathered in the literature. Theoretical and experimental data from reference [41] were used to generate a line of best fit for HOMO and LUMO level approximations. As shown in Figure 5, the regression is not perfectly linear with respect to diameter. Therefore, a logarithmic fit was suitable to describe the regression of energy levels and produced the highest  $R^2$  (how close the

data is fitted) value when compared to linear and polynomial fits [48, 49].

A best fit of error between experimental and theoretical data was developed (shown in Figure 5(a) [41]). The data shows as dot diameter increase, the error between theoretical experimental data increases. Figure 6(b) shows the decrease in optical band gap as dot diameter increases. Error bars at each diameter display the standard deviation between theoretical and experimental data. The standard deviation increases with diameter size as expected.

The same logarithmic best-fit equation was used to estimate the HOMO-LUMO levels of CdSe QDs synthesized in this work, as shown in Figure 6(a). Plots show that as diameter increases, the LUMO level decreases and the HOMO level increases, effectively “shrinking” the band gap. This verifies the band gap calculation discussed earlier [19, 31]. Figure 6(b) shows the decrease in band gap for CdSe QDs, and the increase in standard deviation, which is consistent with literature [41]. High resolution transmission electron microscopy (HR-TEM) and cyclic voltammetry (CV) may be performed visual confirmation of size and distribution.

## 4. Conclusion

In conclusion, we were able to successfully synthesize CdSe QDs quickly using a one-pot microwave synthesis method. The purpose of synthesizing CdSe QDs in-house as opposed

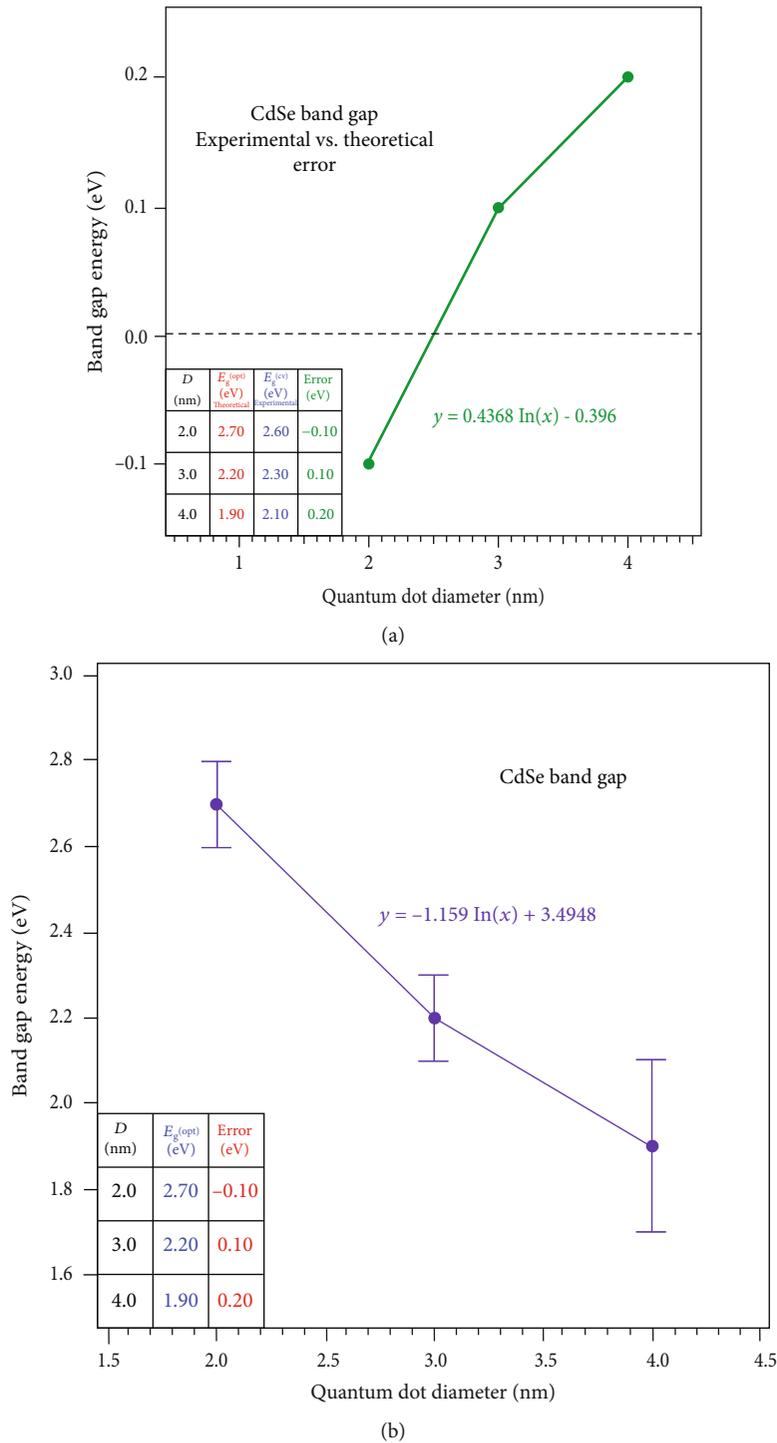
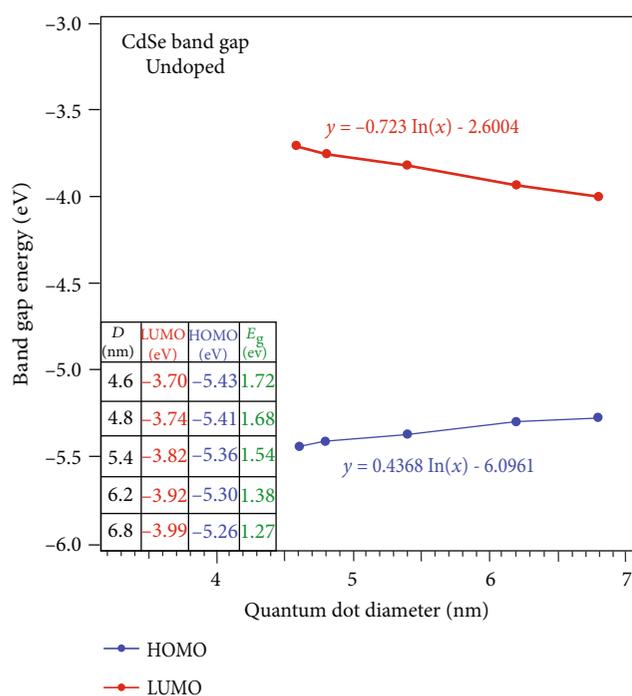


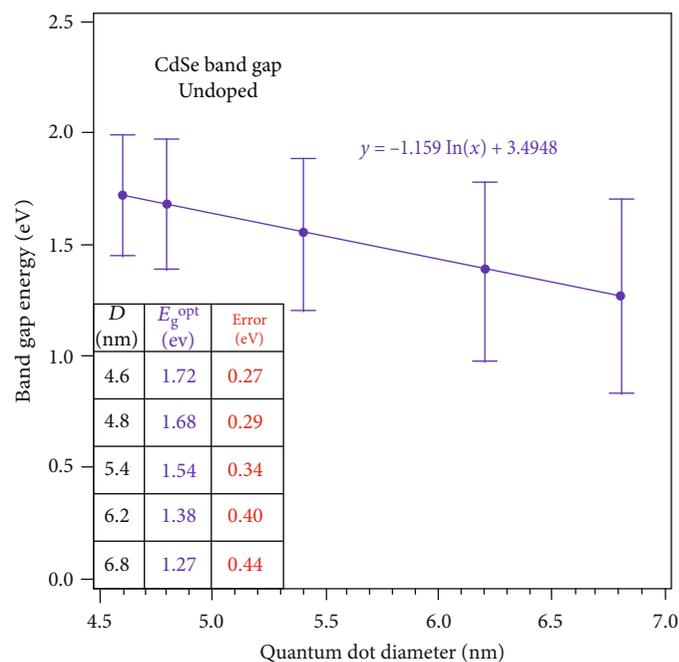
FIGURE 5: Band gap error trend from literature reference comparing experimental and theoretical data (a) and band gap vs. diameter with standard deviation from the literature reference (b) [41].

to purchasing them commercially was the ease of band gap tunability, since dot size is related to band gap due to quantum confinement as discussed. Along with quick synthesis, we were also able to tune their optical properties by varying microwave irradiation times and temperatures. We were able to synthesize CdSe dot diameters ranging from 4.5 to 6.5 nm, with corresponding emission peaks from 509 to 600 nm,

respectively. Rapid microwave synthesis is an economical alternative with high throughput; QDs can be synthesized on a need-by-need basis. The QD synthesized had the same emission profiles in terms of FWHM, bandgap, and peak wavelength as compared to those commercially available. The only drawback of this method is the temperature limitation of the microwave system, which limits the size range of



(a)



(b)

FIGURE 6: HOMO-LUMO level trend of undoped CdSe in this study (a) and band gap vs. diameter with standard deviation (b).

QDs. Other microwave systems may have a wider range of temperature control. Implementation in optoelectronic devices such as solar cell and light-emitting diodes will be the basis of future studies.

## Data Availability

The UV/Visible spectroscopy and photoluminescence data used to support the findings of this study are included within the article.

## Conflicts of Interest

The authors declare no competing financial interest.

## Acknowledgments

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## Supplementary Materials

The Supplementary Information attached to this research article includes detailed preparation, synthesis, and cleaning procedures for the formation of the quantum dots studied throughout this article. It also includes the specific heating mechanism of how the microwave synthesis provides specific advantages over the conventional hot injection method. (*Supplementary Materials*)

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