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Synthesis and Characterization of CdSe Quantum Dots by UV-Vis Spectroscopy

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1. Introduction

CdSe nanocrystals are effective visual aid to demonstrate quantum mechanics, since their transition energies can be explained as a Particle in a Box, where a delocalized electron is the particle and the nanocrystal is the box. Kippeny and co-workers¹ have provided more background information and theoretical discussion. Additionally, Ellis et al.² have stated that modern science is becoming increasingly interdisciplinary. One example is material science, a broad, chemically oriented view of solids that results from the combined viewpoints of chemistry, physics, engineering, and for biotechnology, the biological sciences. Schulz³ has suggested that nanotechnology is an exciting emerging field that involves the manipulation of the atoms and molecules at the nano scale. It is projected that important advances in engineering will come from understanding of the properties of matter constructed from building blocks whose size and shape is uniform and on the 1-100 nm scale. These consequences include technologies to be used in medicine⁴, advances in computer technologies⁵, defense⁶ and everyday applications³.

Several methods exist for synthesizing Cd-Se Quantum Dots. The Molecular Beam Epitax (MBE) is expensive and not readily accessible. Kippeny et al. have used dimethyl cadmium, which is expensive, explosive, and pyrophonic making the system difficult to control and reproduce. Peng and others⁷⁻⁹ have pioneered the kinetic synthesis of Cd-Se nanocrystals from CdO and elemental Se. Boatman et al.¹⁰ have prepared Cd-Se nanocrystals using a kinetic synthesis with a quenching technique where the temperature was 225°C. The visible absorption and emission spectra of individual samples collected at various time intervals during the experimental run were recorded and the maximum wavelength peak were determined. In this paper we report a modified technique of kinetic synthesis of Cd-Se nanocrystals that is safer, simple and can easily be carried out by students in the normal chemistry lab.

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2. Experimental

UV-Vis spectrophotometer (Perkin Elmer Lambda 950) was used for spectroscopic measurements. The scan speed was 11.54 nm /min, integration time was 0.52 s and the data interval was 0.10 nm. The hotplate was Labcongo (115 V, 12 A) and the heating was set at level 3. All chemicals used were bought from Sigma-Aldrich and were of analytical grade. 60 mg of Se, 10 cm³ of 1-octadecene and 0.8 cm³ of trioctylphospine were mixed together in a round-bottomed flask. The solution was then continuously stirred with a magnetic stirrer on a hot-plate and warmed for a few minutes in a fume-hood. Separately, 26 mg of CdO was added to a 25 cm³ round-bottomed flask and clamped in a heating mantle. 1.2 cm³ of acid and 20 cm³ of octadecene were added and mixed together. The solution was heated until CdO dissolved. The CdO solution was then sub-divided into 5 Erlenmeyer flasks each containing 4 cm³ of the stock solution. 0.5 cm³ of Se stock solution was then transferred into the CdO solution with pipette. The samples were heated for 50 s, 60 s, 70 s, 80 s and 120 s, respectively.

3. Results and discussion

The colloidal suspensions of Cd-Se quantum dots of increasing size from left to right are shown in Figure 1.

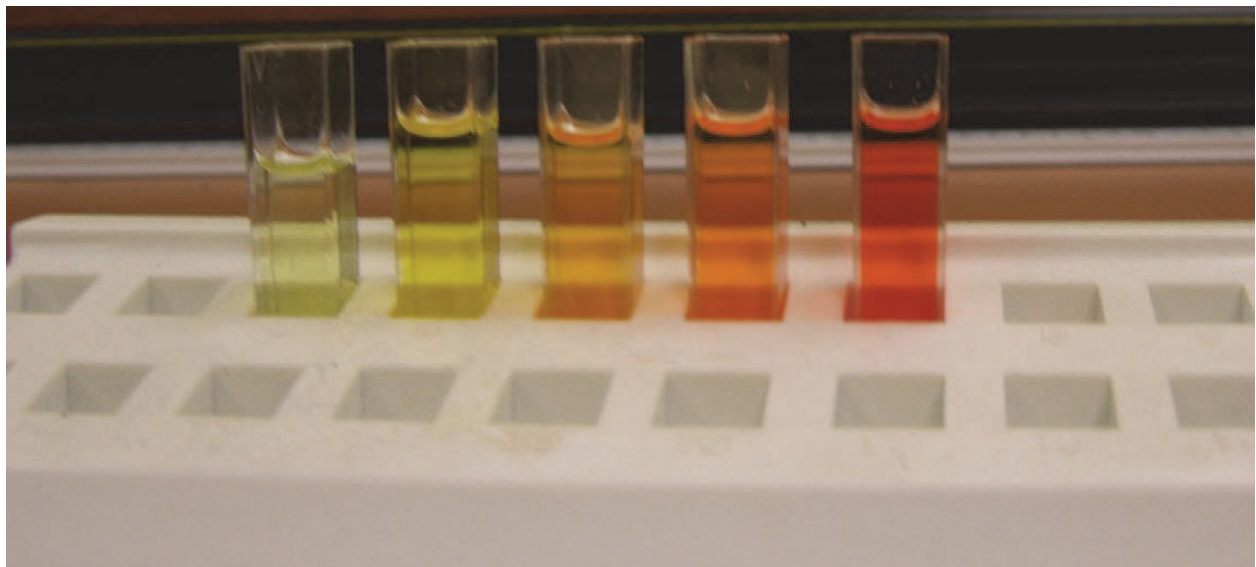


Fig. 1. Colloidal suspensions of Cd-Se quantum dots

The samples viewed in ambient light vary from green-yellow to orange-red. These changes in color which have been noted by other workers^{1, 10} are attributed to the increasing size of the Cd-Se nano-crystals.

Figure 2 presents the calculated diameter of nanocrystal with time¹. The diameter of nanocrystals increases with increasing time. As the nano-crystal size increases, the energy of the first excited state decreases.

The Cd-Se nano-crystals stay suspended in solution and cannot be filtered out. The oleic acid acts as a surfactant, binding to the exterior of the crystal lattice and allowing for the

crystal to remain soluble in the octadecene¹⁰. The diameter of the nanocrystal was calculated using Kippeny¹ method and was found to be in the range found by other workers¹⁰. The Cd-Se crystal growth has been found to be temperature dependent. Transmission electron microscope (TEM) measurements of Cd-Se nanocrystals by others suggest that such wavelengths correspond to 2- 4 nm diameter crystals¹⁰ with at most a few hundred atoms.

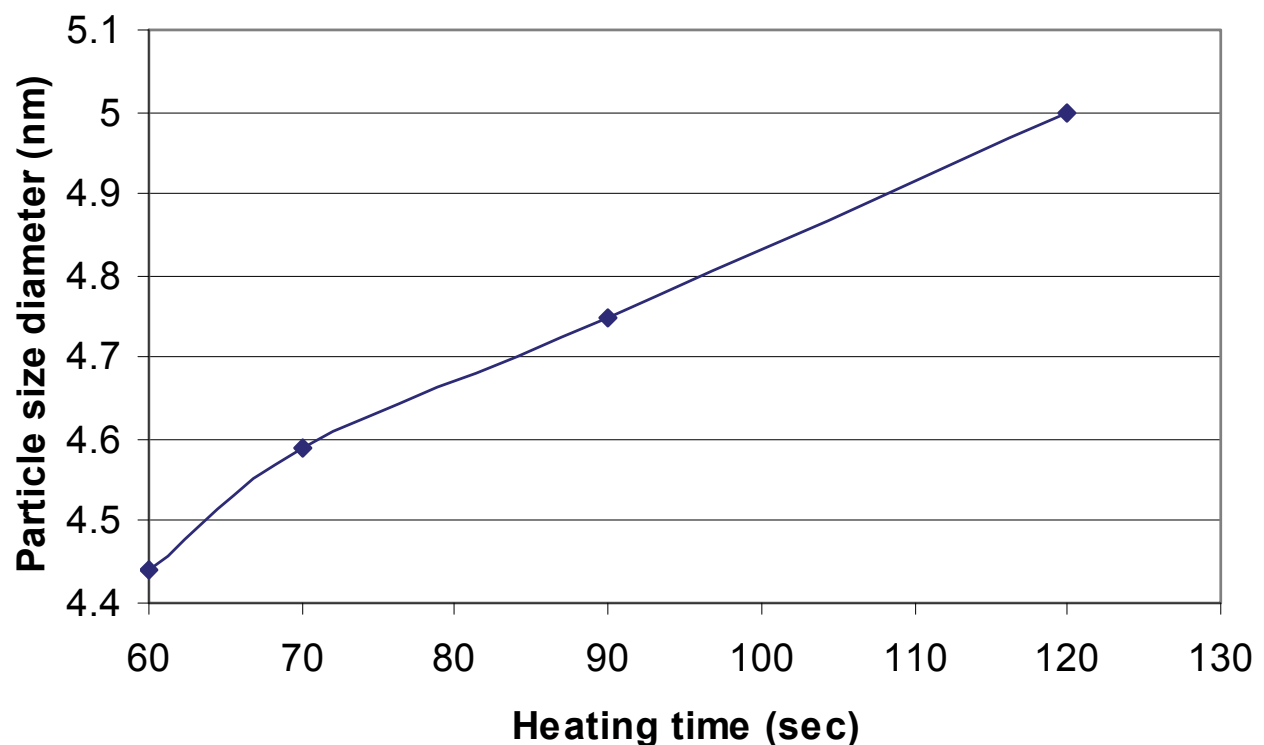


Fig. 2. The effect of particle size growth with time

Figure 3 presents ground state peak wavelengths as a function of reaction time. As the reaction progresses, the peak wavelength decreases. As nanocrystals grow, it has been suggested that their peak emission quickly approaches the band gap of bulk Cd-Se (730 nm).

The observable peak maximum shifts from violet to green with increasing crystal size. The absorption shows peak maxima with additional absorption at lower wavelengths due to the starting materials and oleic acid polymerization. Heating oleic acid and octadecene alone yields increasing visible absorption at increasing wavelengths over time as the effects of oleic acid polymerization become noticeable.

Figure 4 presents UV-Vis spectra of Cd-Se colloidal nanocrystals. The scan range was between 400 nm to 600 nm. The maximum peak shifted toward the longer wavelength. This observation is expected because as the crystal size increases, the energy absorbed or emitted decreases. The sample heated for only 50 sec did not show any peak.

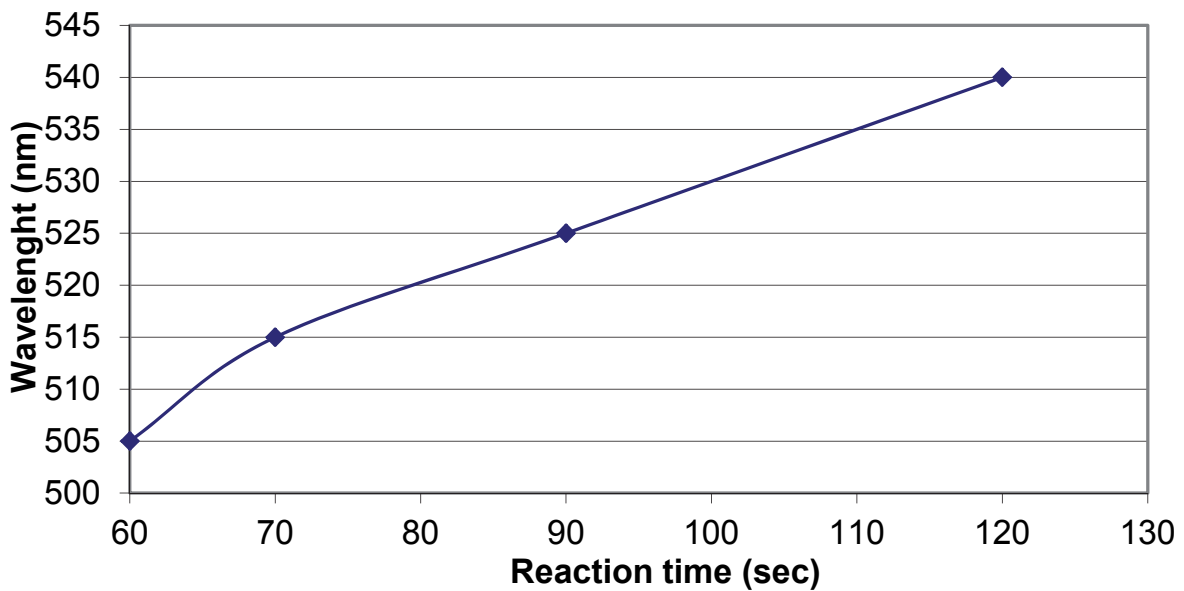


Fig. 3. The change of wavelength with reaction time

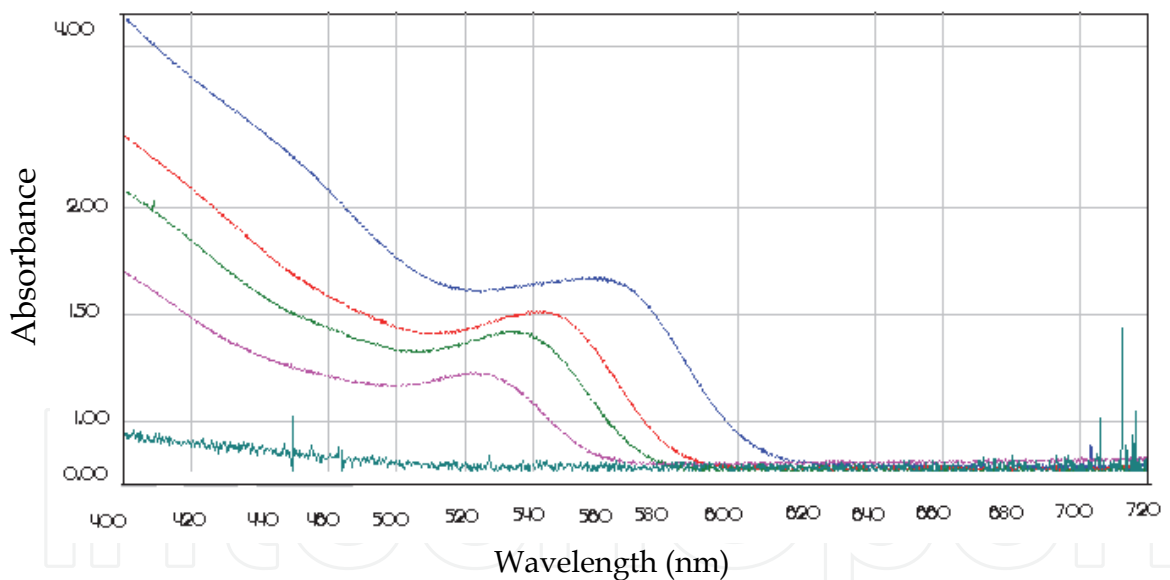


Fig. 4. UV-Vis spectra of Cd-Se colloidal suspension

4. Discussion of the optical measurements and results

As the nanocrystal size increases, the energy of the first excited state decreases qualitatively following particle in a box behavior¹. The optical absorption results using Perkin Elmer Lambda 950 spectrometer are indicated in Figure 4.

a. Energy Shift and Nano- Crystal Size.

Using the L. E. Brus^{1,11,12} model we assume the following:

1. The nanocrystal is spherical with a radius R.
2. The interior of the nanocrystal consists of uniform medium and the excited electron and hole pair.
3. The potential energy outside the radius R is infinite – the radius R defines the confining boundary of the box.

The solution to the spherical Schrödinger equation leads to the energy of the exciton – electron hole pair as¹:

$$E_{ex} = \frac{h^2}{8R^2} \left(\frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{1.8e^2}{4\pi\epsilon_{CdSe}\epsilon_0 R} + \frac{e^2}{R} \left\langle \sum_{k=1}^{\infty} \alpha_k \left(\frac{S}{R} \right)^{2k} \right\rangle$$

The first term is the kinetic energy and the second term the Coulomb potential attractive energy; and the third term is the polarization energy.

b. Using the first term to calculate the exciton energy

At small R the predominant term is the first term (because of the inverse square R dependence since $R < 1$ for a simple example $\frac{1}{(0.5)^2} > \frac{1}{0.5}$).

We can therefore use the first term to approximate R the radius of the nanoparticles as follows:

The energy needed to create the first peak - corresponding to the peak position in the spectra is $E_u = E_g + E_{ex}$ this energy corresponds to 500 nm from Figure 4.

The energy then converts to 2.48 eV (using the well known conversion formula $\frac{1.24}{1\mu m}$ for photon energy to eV.

The energy gap of bulk CdSe corresponds to 730 nm (0.73 μm) and is 1.70 eV [1,10]

This leads to the exciton energy of 0.78 eV

Using the formula:

$$E_{ex} = \frac{h^2}{8R^2} \left(\frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{1.8e^2}{4\pi\epsilon_{CdSe}\epsilon_0 R} + \frac{e^2}{R} \left\langle \sum_{k=1}^{\infty} \alpha_k \left(\frac{S}{R} \right)^{2k} \right\rangle$$

and the first term alone as the approximation for small R

$$E_{ex} = \frac{h^2}{8R^2} \left(\frac{1}{m_e} + \frac{1}{m_h} \right) = 0.78 eV$$

Using h as Planck's constant ; the electron effective mass $m_e = 0.13$ mass of a free electron and m_h equals 0.45 times the free electron mass

R can then be calculated to be the following:

$$R^2 = \frac{h^2}{8E_{ex}} \left(\frac{1}{m_e} + \frac{1}{m_h} \right)$$

$$R = \sqrt{\frac{(6.626)^2 \times 10^{-68} \times 9.91452}{9.1095 \times 10^{-31} \times 8 \times 0.78 \times 1.602 \times 10^{-19}}} = 2.18 \times 10^{-9} \text{ m}.$$

This leads to diameter of about 4 nm

5. Conclusion

We have demonstrated a more convenient synthesis method for colloidal CdSe quantum dots. This method does not involve quenching. This makes it easier for students to make the semiconductor nanoparticles. This synthesis method depends on different heating times for premixed CdO and Se solutions.

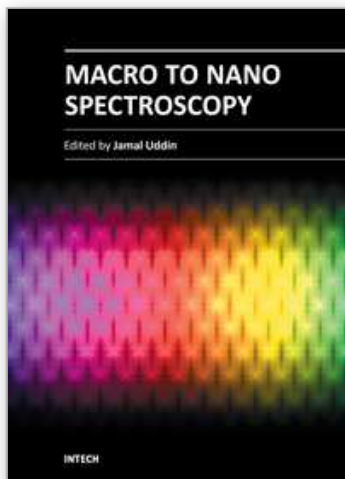
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In the last few decades, Spectroscopy and its application dramatically diverted science in the direction of brand new era. This book reports on recent progress in spectroscopic technologies, theory and applications of advanced spectroscopy. In this book, we (INTECH publisher, editor and authors) have invested a lot of effort to include 20 most advanced spectroscopy chapters. We would like to invite all spectroscopy scientists to read and share the knowledge and contents of this book. The textbook is written by international scientists with expertise in Chemistry, Biochemistry, Physics, Biology and Nanotechnology many of which are active in research. We hope that the textbook will enhance the knowledge of scientists in the complexities of some spectroscopic approaches; it will stimulate both professionals and students to dedicate part of their future research in understanding relevant mechanisms and applications of chemistry, physics and material sciences.

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