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Water-soluble CdSe quantum dots synthesis via facile seed-mediated approach and their characterization studies

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Abstract

In this paper, we are reporting the water-soluble quantum dots exhibiting emission in visible range from 550 to 650 nm. These have been synthesized by a simple seed-mediated approach using cadmium chloride, sodium borohydrate, selenium powder and thioglycolic acid as precursors. The as-prepared cadmium selenide (CdSe) quantum dots were characterized by UV-Vis spectroscopy (UV-Vis), photoluminescence (PL) spectroscopy, Fourier transfer infrared spectroscopy (FTIR), energy dispersive X-ray spectroscopy (EDS) and scanning electron microscopy (SEM). The obtained water-soluble quantum dots exhibited excellent composition, spherical geometry, strong emission and good stability which can be suitable for biomedical imaging applications.

Keywords: Quantum dots, emission, UV-Vis, PL, FTIR, SEM, EDS

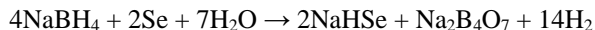
Introduction

Semiconductor nanoparticles are notable for their wide fundamental research and industrial applications ^[1, 2]. Their defining characteristics are their size, which is within the range of 1-100 nm, and excellent chemical processability. The strong confinement of excited electrons and holes leads to dramatically different optical and electronic properties compared to the bulk semiconductor ^[3]. Up till now, numerous methods have been reported for the preparation of semiconductor nanoparticles, including molecular beam epitaxy ^[4], metal organic vapor chemical deposition ^[5], solvothermal ^[6] and hydro-thermal methods ^[7]. CdSe colloidal nanoparticles are most widely investigated as their emissions can be easily tuned to cover from red to blue with decreasing nanoparticle size ^[8]. Until now, many works have been reported on synthesis of II-VI QDs, especially CdSe QDs fabricated by a high-temperature organometallic precursor route, which was first published by Murray *et al.* ^[9]. These QDs had a wide fluorescent emission spectra ranging from blue to red shift. QDs have also been expected to be used as fluorescent labels for biological applications and excellent water-solubility, stability and biological compatibility ^[10-13]. Water-soluble CdSe semiconductor nanoparticles with thioglycolic acid as capping agent have provided an interesting and alternative method to prepare semiconductor nanoparticles. Compared with the organic system, the aqueous approach is environmentally benign and very economical. In this work, we have developed a seed-mediated approach for the synthesis of good-quality CdSe QDs in water by simply mixing CdCl₂, sodium borohydrate, thioglycolic acid, and selenium powder. Thioglycolic acid acted as a capping agent and controlled the size of quantum dots. The UV-Vis spectra and PL spectra were done to identify the optical properties. Further, FTIR and SEM characterization was done to study the QDs. The synthetic parameters affecting the growth and the optical properties of CdSe QDs, such as the Cd²⁺/NaHSe/TGA ratios, precursor concentrations, pH, and growth temperatures were thoroughly investigated. Highly reproducible large-scale synthesis of CdSe QDs with good visible emission could readily be achieved.

Materials and Methods

Synthesis of CdSe Quantum Dots

In the presence of a molar ratio of 2:1 NaBH₄ to Se in water, NaHSe was formed ^[14] according to the reaction equation as follows:



Briefly, 76 mg of NaBH_4 was transferred to a beaker which was cooled by ice; then 1 mL distilled H_2O and 79 mg Se powder was put into the flask. The black Se powder disappeared and white $\text{Na}_2\text{B}_4\text{O}_7$ precipitation appeared after about 2 h. The resulting clear aqueous solution was transferred into 50 mL miliQ deionized water and used in the preparation of CdSe QDs. Cadmium precursor solutions were prepared by mixing the CdCl_2 and thioglycolic acid-TGA (as stabilizer) solutions in 50 mL ultrapure water, and then adjusted to pH 10 with 1 mol/L NaOH. The molar ratio of Cd^{2+} : NaHSe: TGA was used in our experiments is 6: 0.8: 15. Under vigorous stirring, when the prepared oxygen-free NaHSe solution was injected, the CdSe monomers were formed as seeds. And then the as-prepared CdSe monomer solution was heated for 60 min at boiling temperature. We took out the sample at certain time intervals (0 min, 30 min, and 60 min.).

Characterization of CdSe Quantum Dots

UV-Visible absorption spectra were measured by UV-1800 (Shimadzu). Samples were prepared by dispersing the samples in distilled water using ultrasonication. Fluorescence spectra were measured by RF-5301_PC Spectrofluorometer (Shimadzu). Subsequently, Fourier transform infrared spectroscopy and scanning electron microscopy experiments were also performed.

Results and Discussion

The UV/Visible absorption spectra of CdSe quantum dots are shown in Figure 1.

UV spectra shows that as the time increases, particles will grow. Spectra shift towards longer wavelength (red shift). Broader absorption is observed due to discrete energy states

in quantum dots. This effect occurs as a result of quantum confinement. Absorption lies between 380 to 460 nm wavelengths in different sizes quantum dots.

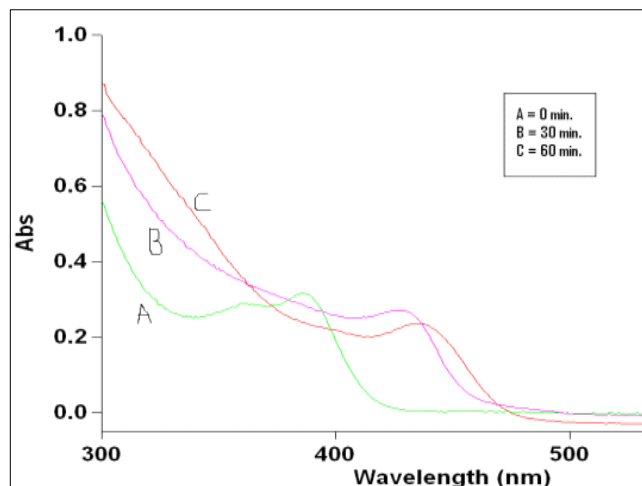


Fig. 1: UV-VIS Spectra for CdSe Quantum Dots.

PL spectra (in Figure 2) show emission lies between 550 to 650 nm (visible spectrum) in different sizes quantum dots. As the time increases, spectra shifts towards longer wavelength. Quantum dots show narrow emission peak. Due to narrow emission peak, there is multiplexing. So, we can use these quantum dots in imaging techniques in biological systems. Narrow emission peak is due to less broadening of energy levels in quantum dots. Emission occurs at longer wavelength than absorption because some amount of absorbed energy is consumed inside the defects in material.

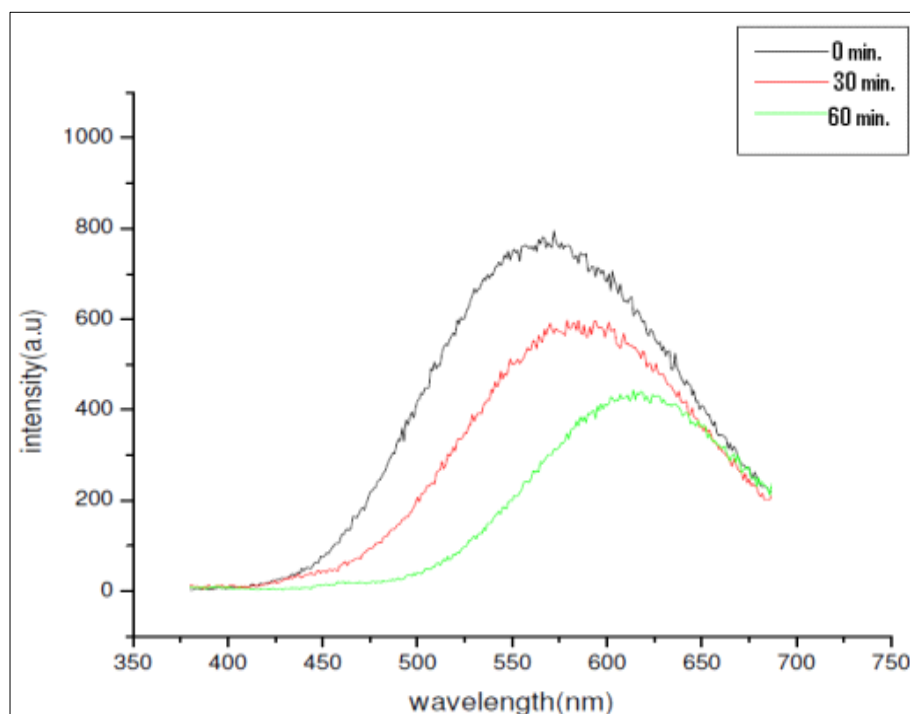


Fig. 2: PL Spectra of CdSe Quantum Dots.

By the FTIR spectra (Figure 3), we can find out as to which functional group is attached with synthesized CdSe quantum dots. FTIR spectra show different peaks for different groups. Peak at 1620.24 cm^{-1} refers to carbonyl group peak. Peak at 1390.53 cm^{-1} refers to C-O-H in the plane band. Peak at

1208 cm^{-1} refers to C-O stretching. Peak at 930 cm^{-1} refers to O-H deformation (out of plane). Peak at 3440.27 cm^{-1} is due to hydrogen-bonded O-H stretching. Peak at 2924.34 cm^{-1} is due to C-H stretching. Peak of weak C-S stretching at 750 cm^{-1} is due to TGA.

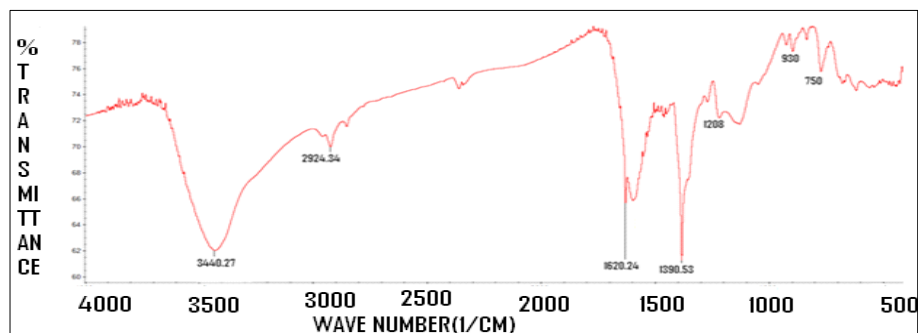


Fig. 3: FTIR Spectra of CdSe Quantum Dots.

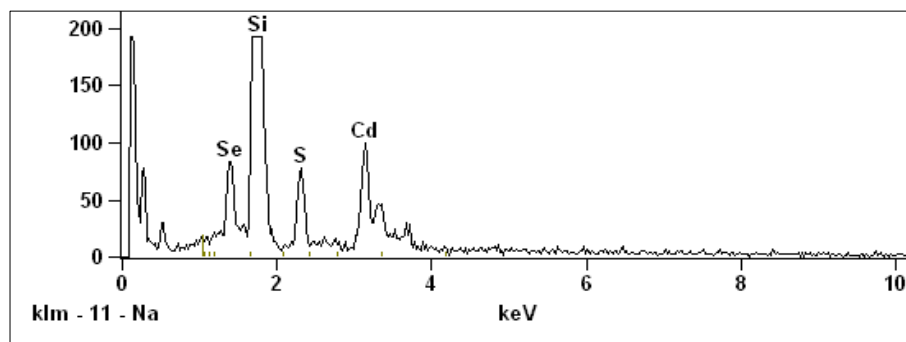


Fig. 4: EDS of CdSe QDs.

Table 1 shows the elemental analysis of the synthesized sample.

Table 1: Elemental Analysis of CdSe Quantum Dots

	KSi	S	Se	Cd
Weight %	61.39	8.03	8.67	21.92
Weight % error	+/- 0.67	+/- 0.31	+/- 2.42	+/- 0.97
Atom %	79.74	9.14	4.01	7.11
Atom % error	+/- 0.87	+/- 0.35	+/- 1.12	+/- 0.31

Energy dispersive X-ray spectrometry shows quantitative and qualitative elemental composition of material. From Table 1, we can say that Cd, Se, S are present in quantum dot samples. Silicon peak is due to the substrate.

Conclusions

We have described a seed-mediated approach for producing high-quality CdSe QDs using thioglycolic acid as a stabilizer. Different synthetic parameters, such as the initial Cd²⁺/NaHSe/TGA ratio, the concentration of precursors, and the nucleation temperature were found to affect the growth of CdSe QDs. As the reaction time is increased, there is a shift in longer wavelength side showing narrowing of peak due to multiplexing. This is an environment-friendly and quick approach and could be further used in biomedical imaging therapies.

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