Formation of Quantum Dots by Aerosol Reactor

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Abstract:

Nanometer sized semiconductors, or quantum dots (QDs) are sought after for their size-dependent photoluminescent properties. The synthesis of QDs in the past has been by the hot solvent injection approach. An alternative to hot solvent injection is aerosol route formation, which has the potential to be a continuous and large scale QD production method. This study focused on; (a) the aerosol route formation of QDs, and (b) examination of the effects of the ratio between the cadmium (Cd) and selenium (Se) precursors. The first part of the study was primarily to create QDs of various sizes with emission wavelengths ranging from blue (465 nm) to red (608 nm) using an aerosol-based synthesis. The second part of the study explored the effects of the molar ratio of cadmium acetate to Se on particle growing rate and the photoluminescent properties. Various molar ratios were tested, 1:5, 1:2, 1:1, 2:1, and 5:1, of cadmium acetate to selenium.

Introduction:

Quantum dots (QDs) are desired for their photoluminescent properties. By tuning the size of the QDs, the band gap can be altered. The specific size of the band gap contributes to a certain wavelength of emission. Researches have been able to use the very specific emission wavelengths in devices such a light emitting diodes as well as in bio-imaging. QDs have been attached to certain proteins that attach to tumors and then fluoresce under ultra-violet light, which makes the tumors visible.

The most common method used to produce QDs is hot-solvent injection method. This method injects hot solvent into a heated precursor and the amount of time the solvents are left in the reaction mixture determines the size of the QD. This method is difficult to operate on a large scale. Another method for producing QDs is aerosol furnace formation. In this method, the precursors are combined into an atomizer and sprayed into a tube furnace, which cooks the QDs at a specific temperature. The QDs are then collected in a bubbler. This method allows for continuous QD production as well as scaled-up production. The purpose of this study is to further explore the aerosol furnace formation of QDs as well as the effect of the molar ratio of Cd to Se.

Experimental Procedure:

Our procedure was modified from Didenko, et al. [1]. All reagents were used as received from Sigma Aldrich. We weighed 0.104 grams cadmium acetate dihydrate ($Cd(Ac)_2$) reagent grade 98% assay and placed it into 1.92 ml oleic acid technical grade 90% assay. Then we heated the solution with a heater set to 140°C until the $Cd(Ac)_2$ totally dissolved. A 10 mL toluene chromasolv reagent 99.9% assay was added into the solution, which was then cooled to 40°C with the heater set to 30°C. In another container, we weighed 0.192 g Se 100 mesh 99.5+% into 1.6 ml trioctylphosphine (TOP) technical grade 90% assay. The solution was mixed until the Se totally dissolved.

Once the oleic acid and $Cd(Ac)_2$ solution was cooled, the second solution containing Se and TOP was added. The solution was shaken well to mix and was added to the atomizer, which had a spray with a mean diameter size of ≈ 0.33 μm. Finally, 15 ml toluene was placed into each bubbler. The temperature of the Lindberg/Blue tube furnace was set (see Figure 1). Once the furnace reached the desired temperature, the nitrogen gas tank was turned on. The pressure before the atomizer was set to 40 psi. The vacuum was turned on, and the valve was adjusted on the Magnahelic laminar flow meter so the flow rate was 0.75 lpm. The reaction was allowed to take place for two hours. The vacuum was turned off and then the nitrogen gas tank was turned off also. The sample was then collected from the bubblers.

For the second part of the study, the same procedure was followed, but the molar ratio of the precursors was altered. Four different molar ratios of Cd(Ac) to Se were tested: 1:2, 1:1, 2:1, and 5:1.

Results and Conclusions:

From our testing, various-sized QDs were produced by aerosol reactor; see Figure 2 (note the full color version on the cover). The emission wavelengths of these dots ranged from 450-608 nm (see Figure 3). QDs with the Cd-to-Se ratio of 1:1 had minimum reduction in emission intensity and wavelength as compared to those with the ratio of 1:5 (see Figure 4).

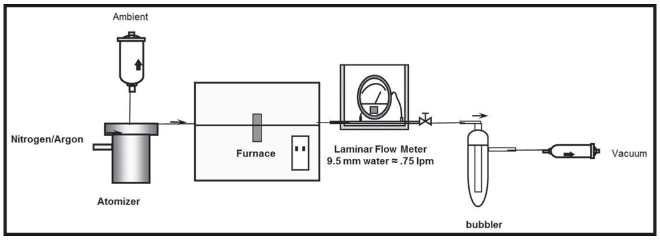

Figure 1: Experimental set-up as modified from Didenko [1].

Figure 2: Samples ranging in temperature from 220-350 under UV light (note the full color version on the cover).

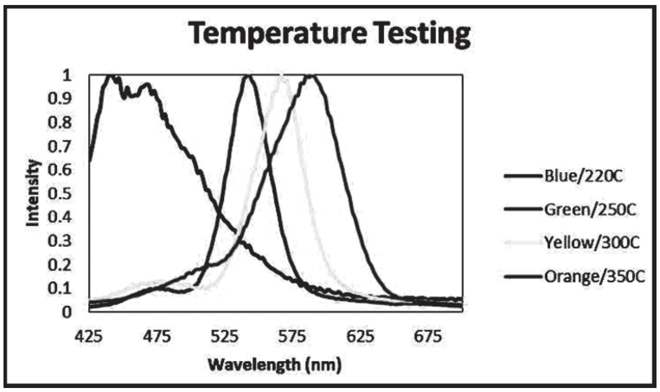

Figure 3: Normalized emission spectra of temperature testing.

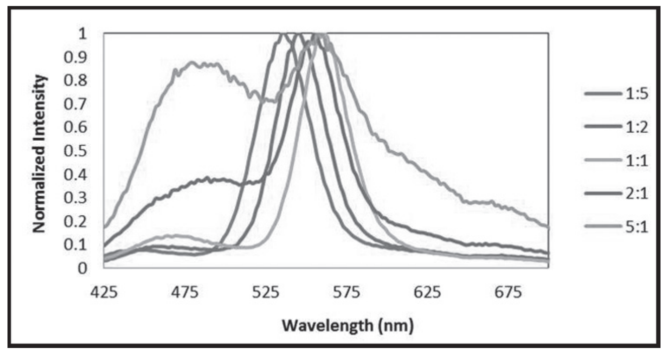

Figure 4: Normalized emission spectra of molar ratio testing.

The 1:1 ratio may prove to be a viable option for future synthesis. The reduced amount of precursor could eliminate the need for "washing" of product, which would reduce waste.

Future Work:

More development is needed to test and improve the QD collection efficiency of the system. Further testing is also needed to find environmental-friendly solvent(s) for the synthesis. At last, testing is necessary to improve the purification process to remove excess un-reacted precursors.

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References:

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