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Fabrication of Semiconducting Nanocrystals: Synthesis of Cadmium Selenide (CdSe) Nanocrystals

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Abstract

Nanotechnology is a study on materials which deal with the structures in nanometer scales, typically ranging from sub-nanometers to several hundred nanometers. Nanocrystals is the material whose crystal structure is measured in nanometers and also known as quantum dots. Nanocrystals have a very interesting optical properties and characteristics where a range of samples of different radii, will emit different wavelengths when excited by the same wavelength of light. Some of the properties are the unique excellent optical properties, such as wide absorption and narrow emission spectra, large extinct, coefficients, resistance to photo-bleaching, long fluorescence lifetime, and size-tunable emission.

We studied the way to synthesize Cadmium Selenide (CdSe) quantum dots in wet chemical approach and explore characteristics of CdSe with experimental investigation through synthesis and spectroscopy. We compare the effect of ratio between Cd precursor and Se precursor by study the luminescence level of CdSe product under fluorescent light and ultra-violet (UV) light. We also studied the behavior of CdSe product to be cool down under room temperature (27°C) and under ice water (4°C), the effect of the reaction media to the growth of CdSe in term of the amount of TOPSe and the amount of TOP as well as the effect of the temperature to the CdSe growth.

In the whole studied, we can conclude that the effect of the reaction medium-TOPSe amount does affect the quality of CdSe product produced. The higher the amount of TOPSe used, the higher the quality and wider range of product. While the effect of the reaction medium-TOP, we found that is does not have an obvious different between the two sample we produced. Therefore, we conclude that the Se source will be also will capped even the TOP used is 20% lesser that the amount calculated with the Stoichiometry in our formula. In the last part of the studied, we observed there are major different in the two set of samples therefore we conclude that the temperature does affect the growth of CdSe.

We suggest in order to cold down the samples in ice water, the time for cold down must be carry on well to have a identical different. Besides that, we suggest the temperature of the CdSe product can be set to ~225°C in order to get full spectrum product in one set sample. We recommend the best volume of TOPSe to use is 2ml, to produce a better quality of CdSe product. This is because the effective attachment of TOP on the quantum dots surface at the higher concentration may result in the effective blocking of electrons and holes recombination on the nanocrystals surface or in other word, the quantum dots will have better passivation.

COLLOIDAL SYNTHESIS OF CdSe NANOCRYSTAL

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Abstract

We studied the way to synthesize Cadmium Selenide (CdSe) quantum dots in wet chemical approach and explore characteristics of CdSe with experimental investigation through synthesis and spectroscopy. We compare the effect of ratio between Cd precursor and Se precursor by studying the luminescence level of CdSe product under fluorescent light and ultra-violet (UV) light. We also studied the behavior of CdSe product to be cool down under room temperature (27°C) and under ice water (4°C), the effect of the reaction media to the growth of CdSe in term of the amount of TOPSe and the amount of TOP as well as the effect of the temperature to the CdSe growth. Besides that, we will give recommendation for the colloidal synthesis approach as well as the precaution steps during the experiment.

1.0 Introduction

Nanotechnology is a study on materials which deal with the structures in nanometer scales, typically ranging from sub-nanometers to several hundred nanometers. Nanocrystals also known as quantum dots are the material whose crystal structure is measured in nanometers. Quantum dots have the optical characteristics where a range of samples of different radii, will emit different wavelengths when excited by the same wavelength of light. Some of the properties for quantum dots are the unique excellent optical properties, such as wide absorption and narrow emission spectra, large extinct, coefficients, resistance to photo-bleaching, long fluorescence lifetime, and size-tunable emission^[1] and a smaller particle leads to a more confined electron, which in turn increases the band gap and the energies of absorbance and emission.^[2-5]

Semiconducting nanocrystals are the semiconductor compounds which is formed by the combination of two or more elements. Cadmium selenide (CdSe) is a solid, binary compound of cadmium and selenium. CdSe are highly sensitive to visible light, both semiconductors display peculiar features of photoconductivity and electrical properties. Thermal evaporation, sputtering, glow discharge deposition and chemical deposition techniques have been used for preparing II-VI compound semiconductors successfully^[6]

To synthesis nanocrystals heavy metal compound had been used. If this type of compound contamination in water, especially groundwater has been recognized as a major problem of catastrophic proportions, therefore the synthesis process as well as disposal procedure should be carry on in the better and secure area.

In our study, we produced the quantum dots ranging from 450 nm to 650 nm. Our studied include the cold down effect using ice water compare to other studies which used liquid nitrogen, and other non-coordinating reagent. We also manipulated some reaction media to see the effect to the growth of CdSe particles. The subjects we studied are the amount of TOPSe the amount of TOP as well as the temperature to the effect of the CdSe particles to growth.

2.0 Synthesis of semiconducting CdSe nanoparticles

Synthesis of CdSe semiconductor nanocrystallites reported by Murray et.al,^[7] is used as the approach here to synthesize the CdSe nanoparticles. Here, the chemical we used are Cadmium Oxide (CdO) (202894), Selenium Powder (Se) (209651), Oleic Acid (364525), 1-Octadecene (O806), and Tri-n-octylphosphine (TOP) (117854) from Sigma-Aldrich.

To study the cooling time of CdSe with the luminescence level, we prepare two sets of sample; first set of sample is cold down in the ice water (~4°C), while the second set is cold down in room temperature (~27°C). Then we have two other groups who do the product with 2 ml and 3 ml TOPSe stock.

A stock of TOP capped Se precursor is prepared ahead of time by combining 30 mg of Se and 5 ml of 1-octadecene (tech., 90%) in a 50-ml conical flask then the solution is heat up and stir with 0.4 ml of tri-n-octylphosphine (TOP) on the stirrer hot plate. The solution is warmed until the Se powders are fully dissolved in the solution. Then Cd precursor was

prepared starting by adding 13 mg of CdO, 0.6 ml of oleic acid and 10 ml of octadecene to a 50-ml conical flask. When the temperature reaches 240 °C, 1 ml of the room-temperature TOPSe is transferred to the 240 °C cadmium solution. Then the timer is started, and with a pasteur pipet, the sample is taken on the time intervals of three to four second for first five samples and ten-twenty seconds intervals for the rest fifteen samples. In the first set, the sample is cold down in the ice water (~4°C). While for the second set, the Cd precursor is prepare like first set but the sample taken is cold down in room temperature (~27°C) and the same TOPSe stock is used.

In the second study, the synthesis process in the same with first study. We manipulated the amount of TOP used to cap Se precursor in the amount show in table 1 to study the effect of the reaction media to the growth of CdSe. Besides that, we also studied the effect of the temperature to the growth by the having a set of sample growth in 200°C with a set of sample growth in 240°C with 80% of TOP volume.

The device we used to characterize the CdSe nanocrystals is the USB4000 Miniature Fiber Optic Spectrometer by ocean optics.

TOP amount (%)	Se (mg)	TOP (ml)	ODE (ml)
180%	30	0.72	5
100%	30	0.40	5
80%	30	0.32	5

Table 1: The percentages weight of TOP amount

3.0 Result and Discussion

3.1 First experiment-the effects of TOPSe to the growth of CdSe and the cold down effect with ice water

We produced the sample ranging from 400 nm to 620 nm (violet to orange) in visible light spectrum. We then compared the six sample in figure 6 and found that the luminescence level under florescence light (white light) ultra-violet light (UV) for the sample with 3 ml TOPSe stock (figure 1 [E-F]) is better compare to 1 ml and 2 ml TOPSe stock. With this we believe that the sample with 3 ml TOPSe stock have higher quantum yield compare with the sample with 1 ml and 2 ml TOPSe stock.

In our studied, we found that there are researcher who used some cold down method like the liquid

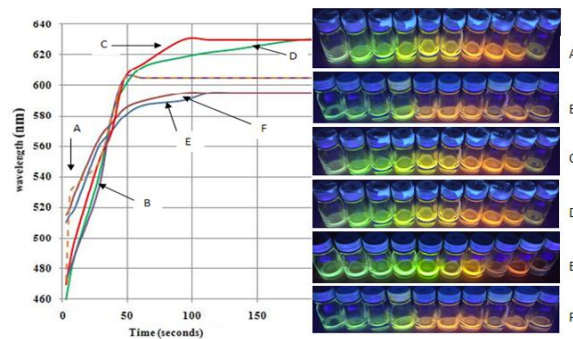


Figure 1: The comparison in term of wavelength of CdSe in different time for 1, 2, 3 ml TOPSe stock for cool in ice water (~4°C) and room temperature (~27°C) [A] 1ml TOPSe stock (w/o ice), [B] 1 ml TOPSe stock (with ice), [C] 2ml TOPSe stock (w/o ice), [D] 2 ml TOPSe stock (with ice), [E] 3 ml TOPSe stock (w/o ice), [F] 3ml TOPSe stock (with ice).

nitrogen and other non-coordinating reagent such as octadecene (ODE) and hexane as cold down medium^[8-9]. Therefore we tried to study the effect if we cold down the sample with the ice water (~4°C). We found that the sample which cold down in the ice water (~4°C) have the shorter range of wavelength when explore under UV light compare with the samples cold down in room temperature (~27°C). The problem of this method is the time of the sample cold down in ice water must be handling well. Based on our observation, if the sample which cold down in ice water being handle well the result will be give a more identical different with the sample which cold down in room temperature. On the other hand, the results may not have obvious different between the two set sample if the time does not handle properly.

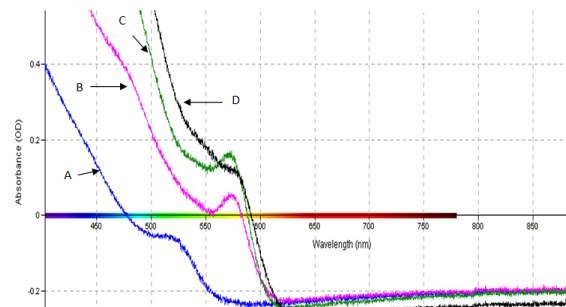


Figure 2: The comparison in term of wavelength of CdSe in different time for 1, 2 ml TOPSe stock for cool in room temperature (~27°C). [A] sixth seconds 1 ml TOPSe stock, [B] forty-eight seconds 1 ml TOPSe, [C] sixth seconds 2 ml TOPSe stock, [D] forty-eight seconds 2 ml TOPSe.

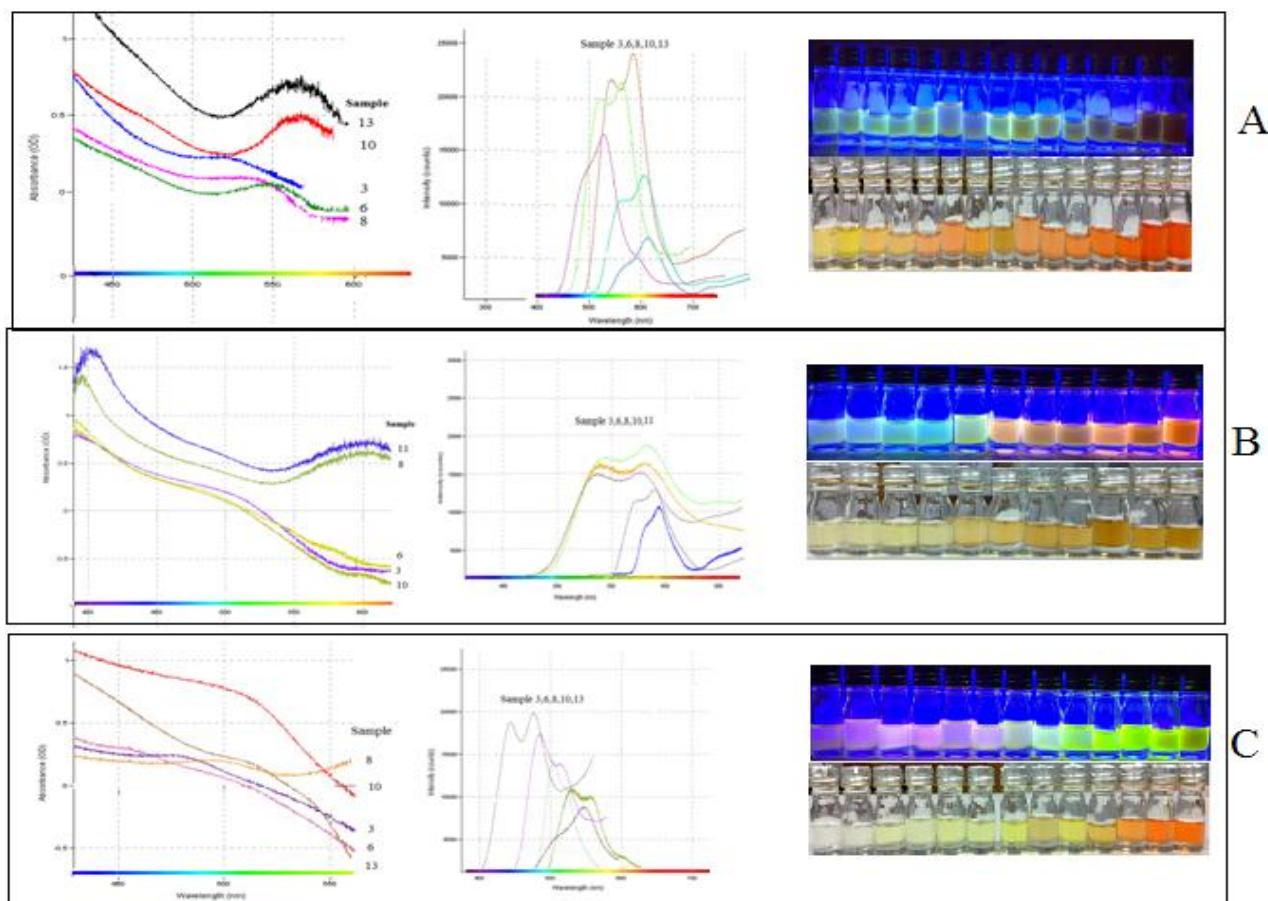


Figure 3: The absorbance and intensity spectrum of the second and third experiment. [A] The sample with 80 % more TOP stock [B] The sample with 20 % less TOP stock (growth in 240°C) [C] The sample growth in 200°C

Besides that, the volumes of TOPSe stock are also affecting the CdSe product growth time where the higher volume of TOPSe stock, the faster the growth. This mean it is more difficult to get the smaller wavelength of CdSe product in higher volume TOPSe stock. These shows in Figure 2 where the comparison was made in term of absorbance wavelength of CdSe product for 1, 2 ml TOPSe stock which cold down in room temperature (~27°C).

3.2 Second experiment-the effects of reaction media-TOP to the growth of CdSe

TOP is used to cap Se source to control the synthesis of CdSe growth. In this session, we studied the effect of the reaction media-TOP to the growth of CdSe. Figure 3A-B shows the comparison of the sample for different TOP amount, from the observation; we found that more TOP amount does not help to increase the quantum yield of the CdSe product. Our product is ranging from 495nm-650 nm (Green- Red).

We concluded that the more TOP being used would not help to increase the quality of the quantum dots but it is also a kind of waste to the chemical.

We also found that the photoluminescence intensity for the sample with 80% more TOP is higher compare to the sample with 20% less TOP. Figure 3A-B shows the absorbance and intensity graph for sample with 80% more TOP stock while Figure 10 shows the absorbance and intensity graph for sample with 20% less TOP stock. The two intensity graphs have the same trend; when the wavelength increase, the intensity is decreasing.

3.3 Third experiment-The effects of temperature to the growth of CdSe

In this session, we studied the temperature effect to the growth of CdSe product. Figure 3B-C shows the comparison of the sample for different growth temperature. We found that the sample which growth in 200°C have the wavelength range from 420 nm to 520 nm (Violet-Green) while the sample growth in

240°C have the wavelength range from 520 nm to 650 nm (Green –Red) which shows in figure 3B-C.

With this observation, we found that to get the temperature are also affecting the CdSe product growth time where the higher the temperature, the faster the growth. This mean it is more difficult to get the smaller wavelength of CdSe product in higher growth temperature. We also found that the photoluminescence intensity is decreasing, when the wavelength increase.

In the whole studied, we can conclude that the effect of the reaction medium-TOPSe amount does affect the quality of CdSe product produced. The higher the amount of TOPSe used, the higher the quality and wider range of product. While the effect of the reaction medium-TOP, we found that is does not have an obvious different between the two sample we produced. Therefore, we conclude that the Se source will be also will capped even the TOP used is 20% lesser that the amount calculated with the Stoichiometry in our formula. In the last part of the studied, we observed there are major different in the two set of samples therefore we conclude that the temperature does affect the growth of CdSe.

4.0 Suggestion and Recommendation

We suggest to cold down the samples in ice water, the time for cold down must be carry on well to have a identical different. Besides that, we suggest the temperature of the CdSe product can be set to ~225°C in order to get full spectrum product in one set sample. We recommend the best volume of TOPSe to use is 2ml, to produce a better quality of CdSe product.

5.0 Conclusion

In this studied, we observed the characteristic and the luminescence level CdSe by varying the reaction medium and temperature. We found that higher TOPSe is better for quality of CdSe product, while higher temperature increase growth rate. On the other hand, the amount of TOP used would not help to increase the quality of the CdSe product.

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Chapter 1 Introduction

1.1 Introduction

Nanotechnology is a study on materials which deal with the structures in nanometer scales, typically ranging from sub-nanometers to several hundred nanometers. Similar to quantum mechanics, on nanometers scales, materials or structures may possess new physical properties or exhibit new physical phenomena. Some of the properties are the unique excellent optical properties, such as wide absorption and narrow emission spectra, large extinct, coefficients, resistance to photo-bleaching, long fluorescence lifetime, and size-tunable emission. ^[1]

We are going to study the Cadmium Selenide (CdSe) nanocrystals in term of the synthesis, characteristic and the application of the nanocrystals. We found many literature on the synthesis of nanocrystals which also known as quantum dots. Among the studies, we are choosing the greener approach to synthesise the nanocrystals which is the colloidal synthesis. The colloidal synthesis involve either rapid injection of reagents into hot surfactant solution followed by aging at high temperature, or the mixing of reagents at a low temperature and slow heating under controlled conditions.

1.2 Objective

The objective of this paper is to illustrate how to synthesis nanocrystals by colloidal synthesis method with a proper procedures, provides a review of the literature concerning how CdSe have been characterized. We also compare the effect of ratio between Cd precursor and Se precursor by study the luminescence level of CdSe product under fluorescent light and ultra-violet (UV) light. We also study the behavior of CdSe product to be cold down under room temperature (27°C) and under ice water (4°C). Besides that, we will give recommendation for the colloidal synthesis approach as well as the precaution steps during the experiment. We will explore these properties by using the knowledge of quantum mechanics, coupled with experimental investigation through synthesis and spectroscopy. We are also tried to synthesize the nanocrystals through a facile and inexpensive synthesis process.

Chapter 2 Literature Reviews

2.1 Introduction

Nanocrystals are the material whose crystal structure is measured in nanometers. The nanocrystals are more familiar to call as quantum dots (QDs). Quantum Dots (QDs) are small particles of a semiconductor material, consisting of a few hundreds to thousands of atoms. Their small size, ranging for most of the systems from 1 nm to 10 nm, is mostly responsible for their unique optical, electrical and chemical properties. The main procedures by which QDs can be fabricated, attaining 3D confinement of the charge carriers, include diffusion controlled growth, lithography, epitaxy and colloidal chemistry.

Quantum dots have the optical characteristics where a range of samples of different radii, will emit different wavelengths when excited by the same wavelength of light. The energy of fluorescence is proportional to the specific size of the particles. This shift in the emission peaks (and absorbance peaks), is characteristic of semiconductor particles when they get small enough, and is due to a quantum mechanical effect known as quantum confinement. This is an effect where by the small volume of the particle restricts the energies of the electrons, and creates a change in the band gap. A smaller particle leads to a more confined electron, which in turn increases the band gap and the energies of absorbance and emission.^[2]

Semiconducting nanocrystals are the semiconductor compounds which is formed by the combination of two or more elements. The most common compound semiconductors are those formed by the elements of groups symmetrical in respect to the IV group, which is the group of Si, namely, the II-VI (cadmium and zinc chalcogenides), and III-V compounds (gallium and indium arsenides and antimonides). Cadmium selenide (CdSe) is a solid, binary compound of cadmium and selenium. CdSe are highly sensitive to visible light, both semiconductors display peculiar features of photoconductivity and electrical properties. Thermal evaporation, sputtering, glow discharge deposition and chemical deposition techniques have been used for preparing II-VI compound semiconductors successfully^[3]

2.2 Characterization for Nanocrystals

There are many enabling technologies that are currently used in this field. However a distinction must be made between the technologies that help produce the CdSe nanocrystals and the one that are responsible with the nanocrystals characterization. Due to the extremely small sizes of nanocrystals which ranging from a few nanometers, semi conductive CdSe nanocrystals are generally searched by means of electron microscopy. It's good to emphasize the fact that each synthesis method can produce different types of nanocrystals, hence the need for various characterization methods.

The characterization machines that widely used are the Transmission Electron Microscopy (TEM), High Resolution Transmission Electron Microscopy (HRTEM), X-ray diffraction (XRD) and optical spectroscopy. Transmission Electron Microscopy (TEM) and High Resolution Transmission Electron Microscopy (HRTEM) were used to approximate the size of the particle ^[4-6]. Figure 1 show the sample TEM image for nanocrystals. In order to get a good description of the type of crystalline structure the nanocrystals are characterized by X-ray diffraction (XRD) ^[5]. Equally important is the elemental composition of this binary compound that can be easily done with the help of X-ray fluorescence (XRF) and X-ray energy dispersive analysis (EDX) ^[7]. Other diagnostic method that is widely used to search the optical properties of the CdSe nanocrystals is absorption spectroscopy or optical spectroscopy. ^[8, 9]

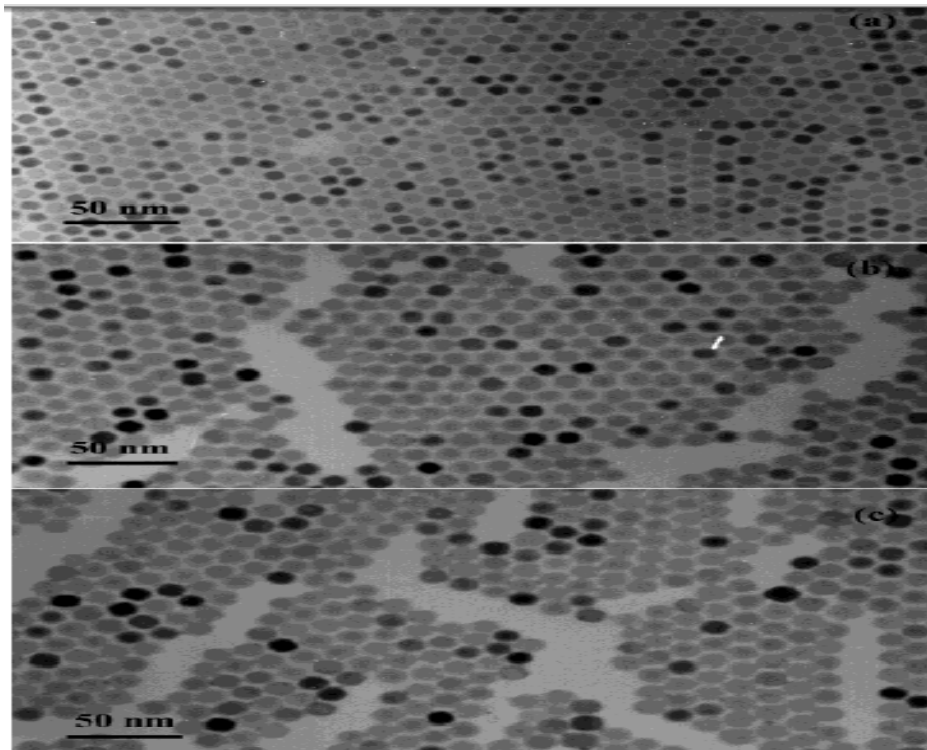


Figure 1: TEM images of (a, top) 6 nm, (b, middle) 8 nm, and (c, bottom), 9 nm sized nanocrystals. ^[10]

Optical Spectroscopy had been widely used for the characterization of nanomaterials and the techniques can be generally categorized into two group, absorption and emission spectroscopy and vibrational spectroscopy. Absorption and transmission spectroscopy determines the electronic structures of atoms , ions, molecules or crystals through exciting electron from the ground to excited states (absorption) and relaxing from the excited to ground stage to ground stages (emission). Figure 2 shows optical absorption spectrum of CdSe nanocrystals with varying the diameter and reveals the increased band gap as indicated by a blue shift in the absorption edge and discrete electronic transitions as the nanocrystals get smaller ^[11]. This is an observed in the absorption and emission spectra of nearly isolated atoms and ions due to the transitions between quantum levels are extremely sharp. Therefore the wavelength and photon energies can be determined with great accuracy.

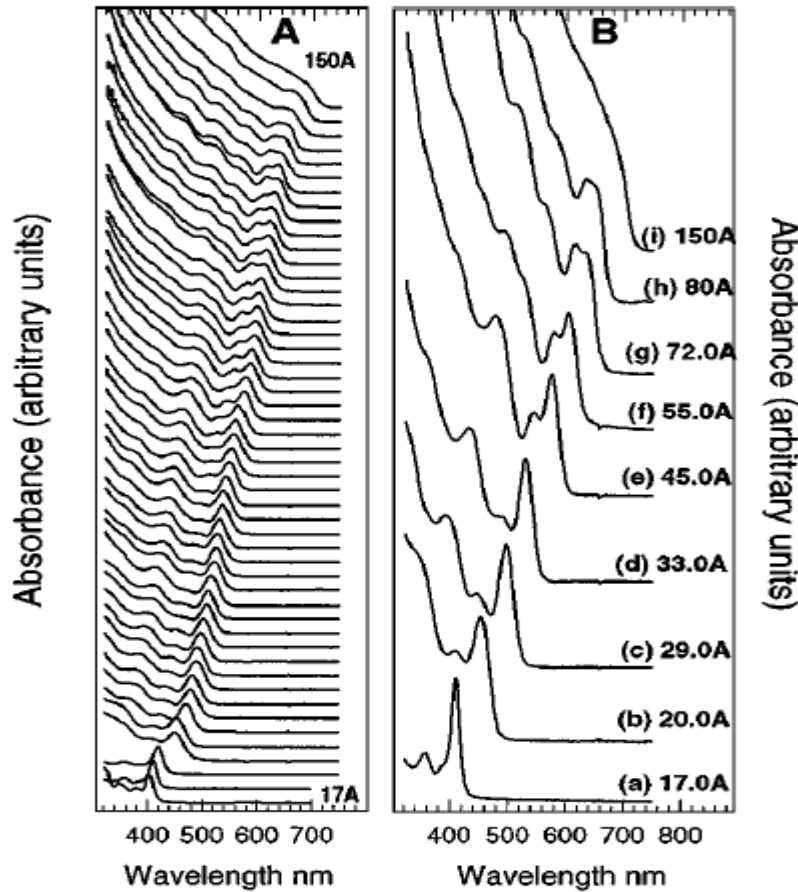


Figure 2: Optical absorption spectra of CdSe nanocrystals with varying diameters
 (A) This is seen spectroscopically as a blue shift in the absorption edge and a larger separation between electronic transitions for a homologous size series of CdSe nanocrystals dispersions, collected at RT. (B) Observation of discrete electronic transitions in optical absorption. ^[11]

With the TEM we can also study the size and shape of nanocrystals. Size and shape selected nanocrystals behave like molecular matter that can be used as fundamental building blocks for constructing nanocrystals assembled superlattices. These nanocrystals form a new class of materials that have orders in both atomistic and nanocrystals length scales. The nanocrystals are passivated with organic molecules (called thiolates) that not only protect them from coalescence but act as the molecular bonds for forming the superlattice structure. The interparticle distance is adjustable, possibly resulting in tunable electric, optical and transport properties. Figure 3 shows the Atomic structural model of truncated octahedral nanocrystals and a schematic model illustrating the ordered self-assembling.^[12-16]

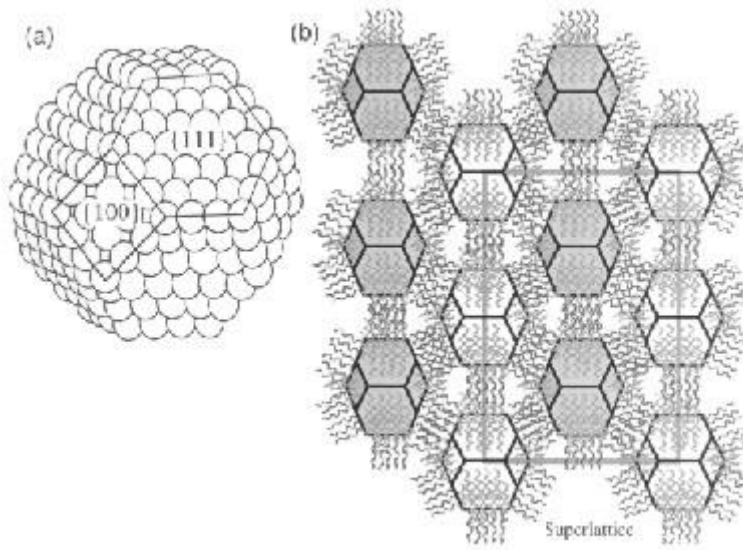


Figure 3: (a) Atomic structural model of truncated octahedral nanocrystals
(b) A schematic model illustrating the ordered self-assembling^[12]

Luminescence is study how electrons in excited state drop to lower levels by emitting photon. Photoluminescence (PL) is the re-emission of light after absorbing a photon. The physical processes involved in photoluminescence are more complicated than those in absorption because it is tied up with the emission relaxation mechanism in the material. For direct band gap materials, the excited electrons and holes will relax rapidly to the lowest energy states within their respective bands, and the photons are emitted when electrons at the bottom of the conduction band recombine with holes at the top of the valence band. This optical transition of direct band gap semiconductors is dipole-allowed and has large matrix elements. The luminescence efficiency is therefore expected to be high. It is noteworthy that the emission and absorption spectra are not the same. The band gap corresponds to the threshold for optical absorption, not to the energy of optical emission. For indirect band gap semiconductors, the conduction band minimum and valence band maximum are at different points in the Brillouin zones Conservation of momentum requires that a phonon must be involved when the photon is emitted. This

makes it a second-order process, with a relatively low transition probability.^[17-20] For example CdSe nanocrystals can be used as single photon sources which can generate single photons under pulsed excitation.^[21-25]

Some study had introduced a novel 8-dimensional microscope. The microscope has been proved to be able to perform non-invasive real time in situ measurements on nanomaterials, and on the structural and functioning parameters of single intact cells with high sensitivity and spatial resolution, and really an 8-dimensional one: 3 dimensions in space, one in time, one in wavelength, and the 6th to 8th one are about the light from the sample: transmitted light, scattered light, and fluorescence.^[26]

While some literature study on the temperature effect on the band gap of CdSe nanocrystals. Figure 4 shows the temperature dependence of absorption spectra of 4.0 nm and 2.5 nm CdSe nanocrystals; temperature-induced variation of band gap absorption peak positions of 4.0 nm and 2.5 nm CdSe nanocrystals. When the temperature increases, the absorption spectra of both the 4.0 nm and 2.5 nm CdSe nanocrystal samples exhibit a redshift^[27].

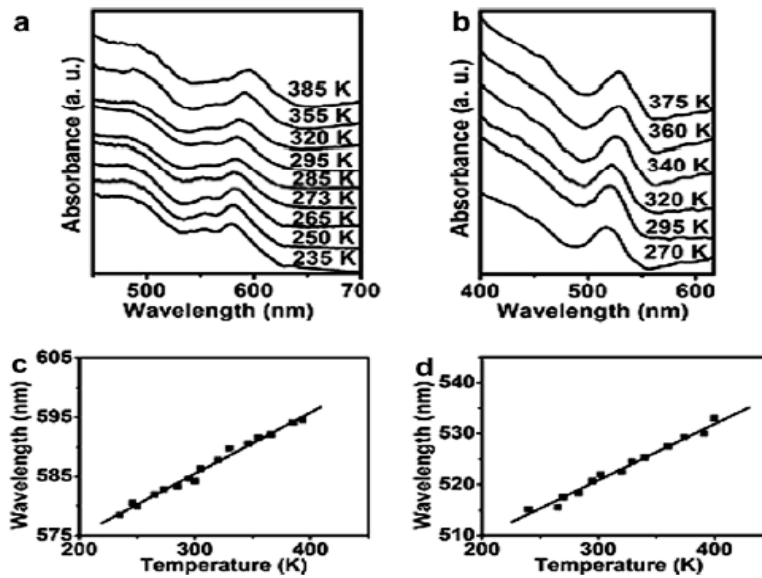


Figure 4: Temperature dependence of absorption spectra of 4.0 nm (a) and 2.5 nm (b) CdSe nanocrystals; temperature-induced variation of band gap absorption peak positions of 4.0 nm (c) and 2.5 nm (d) CdSe nanocrystals.^[27]

By using Atomic Number Contrast Scanning Electron Microscopy (Z-STEM), the core/shell structures at the atomic level can be studied. The purpose of the shell material is to passivate surface trap sites and to energetically confine the electron and hole. Ideally, for every photon that creates an electron and hole pair, one photon is emitted. To ensure radiative electron and hole recombination, a material with a wider band gap than the core is used to coat the surface. In the case of CdSe is typically used as the shell material due to its wide band gap.^[28, 29]

2.3 Synthesis of semiconductor nanoparticles

The evolution of a solid from a vapour, liquid, or solid phase involves two fundamental steps: nucleation and growth. As the concentration of the building blocks of a solid becomes sufficiently high, they aggregate into small cluster (or nuclei) through homogeneous nucleation^[30]. Synthesis of the non-oxide semiconductor is commonly synthesized by pyrolysis of organometallic precursor(s) dissolved in anhydrous solvents at elevated temperatures in an airless environment in the presence of polymer stabilizer or capping materials. Preparation of Nanocrystals samples that are uniform in composition, size, shape, internal structure, and surface chemistry is essential to successfully mapping their size dependent materials properties. High-temperature solution-phase synthesis provides a method of preparing such uniform nanocrystals samples for a variety of metals^[31] and semiconductors. Each nanocrystal in a sample consists of an inorganic crystalline core surrounded by an organic monolayer.

Colloidal Synthesis is the synthesis of nanocrystals using wet chemical approach. Typically, these approaches involve either rapid injection of reagents into hot surfactant solution followed by aging at high temperature, or the mixing of reagents at a low temperature and slow heating under controlled conditions^[32]. The synthesis of nanocrystals by the pyrolysis of organometallic reagents, first developed by Murray et al. is the most common chemical procedure for producing large quantities of II-VI and III-V quantum dots with a narrow size distribution^[33]. The reaction performed in inert atmosphere, which is a major drawback of this reaction.

The procedure to prepare CdSe nanocrystals with colloidal synthesis is briefly outlined below. First CdO is used as Cd source, and the mixed tri-n-octylphosphine (TOP) and tri-n-octylphosphine oxide (TOPO) solution were used as solvents and capping materials, also known as coordinating solvents while trioctylphosphine selenide (TOPSe) were used as Se precursors. Fifty grams of TOPO is dried and degassed in the reaction vessel by heating to ~ 200°C at ~ 1 torr for ~20 min, flushing periodically with argon. The temperature of the reaction flask is then stabilized at ~300°C under ~1 atm of argon. Cd source is added to TOP in the dry box and TOPSe stock solution is added to TOP solution. Two solutions are then combined and loaded into a syringe in the dry box. The heat is removed from the reaction vessel. The syringe containing the reagent mixture is quickly removed from the dry box and its content delivered to the vigorously stirring reaction flask in the single injection through a rubber septum. The rapid introduction of the reagent mixture produces a deep yellow/orange solution with an absorption feature at 400-460 nm. Depending on the aging time, CdSe nanoparticles is prepared^[31]. The sizes of CdSe nanocrystals depend on the growth time. In order to observe the color variation, it is critical to remove and quench the samples at frequent time intervals, as quickly as possible in the beginning.^[34-38] However, there are several ways to improve the nanocrystals nucleation and growth (increase Quantum Yield and Quantum Efficiency). For example, coating CdSe with ZnS or adding TOP/TOPO with HDA.^[39-50]

The synthesis of semiconductor nanocrystals and the synthesis of CdSe nanocrystals in particular is a cross-disciplinary science. Since it addresses the making of a chemical compound, one might infer that the nanocrystals production is chemistry related discipline. This is partly true because the motivation that drives the nanotechnology people resides in the need for novel optical devices that can be used, for example, in optical physics and biology.

As in the growth of spherical CdSe nanocrystals in hot TOP solution, the surfactants dynamically adsorb to the growing crystallites, allowing atoms to add and subtract for high crystallinity. This enables the growing crystallites to anneal, resulting in good crystallinity, while suppressing particle aggregation. It is important to note that the growth mode of the nanocrystals depends strongly upon monomer concentration.^[4]

Besides organometallic method to synthesize nanocrystals that being introduce above, some literature had synthesis the nanocrystals in different method and handling manner, they are synthesis using pulse electrode position technique^[51], modified hydrothermal method^[52], non-organometallic method^[53], synthesis under micro-flow-reactor^[54,55],

2.4 Application of Nanocrystals

There many possible applications and potential applications of nanomaterials have been studied from electronics, optical communications and biological systems. In electronics, there are devices in made by the nanomaterials, for example the quantum dot devices have been used in lasers and detectors. When the crystallites size is in the same order of magnitude of the Bohr radius of the bulk crystal exciton, the binary semiconductor CdSe exhibit both linear and nonlinear optical properties that can be applied in optoelectronic devices the implementation of nanocrystals in optoelectronic devices are like solar cells, sensors, light emitting diode and etc ^[56]. While some nanoparticles is are also implemented into nanoelectronic devices due to the semiconducting behavior of different materials. Examples of nanoelectronic devices include single-electron transistors, field effect transistor (FET), and sensors. One of the attractive applications is the application of nanocrystals in the practice of medicine. With the nanoparticles and nanocircuits, the nanoscales devices are implemented for improved therapy and diagnostics.

2.4.1 Photovoltaic / Solar Cells

With the crystal growth kinetics, a better structure of nanocrystals will well suit the application for photovoltaic system. Scientists and researchers had discovered with fabrication of non-core heterostructure, the optical properties of the photovoltaic have higher efficiency compare to the previous approach. ^[57-59]

2.4.2 Sensor/ Nanosensor

Another application for nanocrystals is the implementation on the optical devices like sensor. One of the techniques to produce the sensor is the synthesis of nano-structured materials by laser ablation. With this technique nanocrystals were grown with nanoparticles generated by laser ablating. While dielectrophoresis techniques fabricated nanostructured materials sensors create a photosensor with better detection sensitivity as well as the response time ^[60-62]. There are also several types of nanostructured metal oxide semiconductor materials have been successfully synthesized for the sensing of both ethanol and oxygen gases. ^[63] Nanosensor is having more superior properties over exciting technique because it has higher sensitivity, can provide rapid reading, and low cost on-field detection. ^[64] The application of quantum dots of II/VI materials as sensing elements while immobilized in a solid host which can be used in combination with optical fiber or integrated optics sensing technology. ^[65]

2.4.3 Laser (Quantum Dot Laser)

Quantum dot laser with ultralow-threshold current densities and low sensitivity to temperature variations have been demonstrated. A research has been done to analyze room temperature lasers based on single crystals of chromium-doped CdSe due to their broad band tunability beyond spectral region. This type of laser has a fast room temperature emission lifetime values. ^[66]

2.4.4 Fluorescence probes

Fluorescence probes are commonly used to label biological molecules and very useful practice in fields of chemistry, biology and medical science. Compare to the most uses techniques-Organic dyes; Fluorescence probes have lesser significant limitations, including photo-bleaching and low signal intensities. Furthermore, most of them have narrow excitation spectra, and often exhibit broad emission bands with red tailing, which makes it difficult for simultaneous quantitative evaluation of relative amounts of two or more sensors, presenting in the same sample because of spectral overlap. The new fluorescence probes for tracing allow ultrasensitive quantitative detection as it can be applied in medical treatment, biological engineering, and especially in food antisepsis to replace chemically synthesized ones ^[67-71]. There also application of quantum dots in non-invasive Imaging of biological study like the study on mice. ^[72, 73]

2.4.5 Light-Emitting diodes (LEDs)

There are also light-emitting diodes (LEDs) have been developed based on nanostructures of wide-band gaps semiconductor materials. This type of devices takes the advantages of quantum well configuration and direct energy band gap to achieve high internal radiative efficiency. Thin nanoparticle films have been characterized by conventional absorption and luminescence measurements and were used for fabrication of LEDs. ^[74-76]

2.4.6 Quantum-Dot Memory

There some study of memory effects in the electronic transport in colloidal undoped CdSe nanocrystals quantum dot arrays. The memory effects in the electronic transport in CdSe nanocrystals quantum-dot arrays have been observed and characterized. The studied also show switching of the conduction in CdSe nanocrystals arrays and found the behaviour to be highly sensitive to the value and duration of the laser and voltage pulses. ^[77]

2.4.7 Nanomedicine

Nanomedicine is a medical application of nanotechnology. The application of nanotechnology on medicine field ranges from medical use of nanomaterials, nanoscale electronic biosensors to molecular nanotechnology ^[78]. Recently, the field of nanomedicine-based contrast agents offers a great opportunity to develop highly sophisticated devices that can overcome many traditional hurdles of contrast agents including solubility, cell-specific targeting, toxicities, and immunological responses. This type of application helps to reduce the time of identifying the syndrome of illness ^[79]. Besides that, the optical properties of nanocrystals are also believed can help to detect early stage of cancer ^[80, 81]. However some problems need to be overcome before nanomedicine can fully implement, they are the toxicity and stability of nanocrystals. ^[82]

2.4.8 Treatment on Pollution

As a result of larger specific surface area nanoparticles are significantly and more active than larger particles of the same material. This property has been exploited in the use of iron oxide nanoparticles for removing arsenic from groundwater. Iron oxide nanoparticles are able to bind irreversibly arsenic five to ten times more effectively than micron-sized particles and, because of their superparamagnetic properties, can be separated from the purified water by the application of a magnetic field. The nanoparticles can afterwards be retrieved by deactivating the magnetic field with negligible risk of their being released into the environment. ^[83]

Although there are many applications for nanocrystals, the most important concern is the radiative lifetime, which the average time interval between two consecutive emitting photons from the single-photon source consequently affect the speed of optoelectronic devices. Compared with the typical 1-ns radiative lifetime in bulk materials, the radiative lifetime of CdSe nanocrystals usually ranges from several to tens of nanoseconds. One interesting feature of the photoluminescence (PL) decay of colloidal QDs is the almost universal occurrence of biexponential time distribution in the radiative lifetime. Typically, a shorter lifetime is on the time scale of several nanoseconds, and a longer one is tens of nanoseconds. ^[9]

2.5 Hazardous of nanocrystals

Nanotechnology is a multidisciplinary field that covers a vast and diverse array of devices derived from engineering, physics, chemistry, and biology. The burgeoning new field of nanotechnology, opened up by rapid advances in science and technology, creates myriad new opportunities for advancing medical science and disease treatment in human health care. To synthesis nanocrystals heavy metal compound had been used. This type of compound contamination in water, especially groundwater, has been recognized as a major problem of catastrophic proportions. The toxicology and health hazard also has been reported for many years. Because of the recognition that heavy metal at low concentrations in drinking water causes severe health effects, the technique to handle the materials have become increasing important.

Applications of nanotechnology to medicine and physiology imply materials and devices designed to interact with the body at molecular scales with a high degree of specificity. This can be potentially translated into targeted cellular and tissue-specific clinical applications designed to achieve maximal therapeutic efficacy with minimal side effects.^[84]

Semiconductor nanoparticles such as CdSe/ZnS nanoparticles have been utilized heavily for bioimaging applications^[85]. The biggest challenge with this type of material is the potential of high toxicological effect caused by heavy metal dissociation^[86-88]. Some studies shown smaller nanoparticles nanocrystals can cause increased cytotoxicity compared to larger particles molecules. The lack of interaction of larger particles with DNA is suggested to be due to steric hindrance. While nanocrystals may be very effective cancer treatments, healthy cells would also be affected potentially causing toxicity^[88-90].

The rapid recent rise in nanotechnology has led to exciting developments in optical, electronic and medical devices. Most of these rely on techniques that manipulate nanoparticles so that they can bypass the human body's defence mechanisms, which might imply that less desirable nanoparticles could also penetrate into cells or cross natural barriers. Therefore the regulatory frameworks and trial safety procedures is important and sufficient to cover the use of nanotechnologies especially in medicines and medical devices.

Chapter 3-Research Methodology

We started our study by find and read the literature on the synthesis, characteristic and application of the CdSe nanocrystals. Then we choose the wet-chemical approach to synthesis our CdSe nanocrystals, which is the colloidal synthesis. Our study on the literature show that typically the synthesis involve either rapid injection of reagents into hot surfactant solution followed by aging at high temperature, or the mixing of reagents at a low temperature and slow heating under controlled conditions ^[32]. The synthesis of nanocrystals by the pyrolysis of organometallic reagents, first developed by Murray et al. is the most common chemical procedure for producing large quantities of II-VI and III-V quantum dots with a narrow size distribution ^[33]. The reaction performed in inert atmosphere, which is a major drawback of this reaction. In this paper, we studied the behavior of CdSe product to be cool down under room temperature (27°C) and under ice water (4°C), the effect of the reaction media to the growth of CdSe in term of the amount of TOPSe and the amount of TOP, as well as the temperature effect on the CdSe growth.

The chemical we used to synthesis our CdSe product are Cadmium Oxide (CdO) (202894), Selenium Powder (Se) (209651), Oleic Acid (364525), 1-Octadecene (O806), and Tri-n-octylphosphine (TOP) (117854) from Sigma-Aldrich. In the first experiment, we studied the effect reaction media-TOPSe to the growth of the CdSe nanocrystals and the effect of the cold down operation by ice water (~4°C). A stock solution of Se precursor is prepared ahead of time by combining 30 mg of Se and 5 ml of 1-octadecene (tech., 90%) in a 50-ml conical flask then the solution is heat up and stir with 0.4 ml of tri-n-octylphosphine (TOP) on the stirrer hot plate. The solution is warmed under the Se powder are fully dissolve in water.

Then Cd precursor was prepared starting by adding 13 mg of CdO, 0.6 ml of oleic acid and 10 ml of octadecene to a 50-ml conical flask. The temperature is taken from time to time and recorded in Table 1 and 2. When the temperature reaches 240 °C, 1 ml of the room-temperature TOPSe is transferred to the 240 °C cadmium solution. Then the timer is started, and with a pasteur pipet, the sample is taken on the time intervals of three to four second for first five samples and ten-twenty seconds intervals for the rest fifteen samples. In the first set, the sample is cold down in the ice water (~4°C). While for the second set, the Cd precursor is prepare like first set but the sample taken is cold down in room temperature (~27°C).

We then compared our result with the other two groups who are prepared CdSe with the 2 ml TOPSe and 3 ml TOPSe. Figure 5 shows the comparison in term of wavelength of CdSe in different time for 1, 2, 3 ml TOPSe stock for cool in ice water (~4°C) and room temperature(~27°C). While figure 6 shows the comparison of the sample for 1, 2, 3 ml

TOPSe stock for cool in ice water ($\sim 4^{\circ}\text{C}$) and room temperature ($\sim 27^{\circ}\text{C}$) under ambient light and UV light.

In the second experiment, we change the TOP weight in the TOPSe solution to study its effect to the CdSe growth. The steps to prepare the CdSe product are the same as the first experiment but the weight of TOP is change according to the data shows in table 1. In the first set, we prepared the product with 80% more of TOP and second set we prepared the product with the TOP amount with 20% less.

In the third experiment, we studied the temperature effect to the CdSe growth. We prepared the CdSe sample in 200°C with the 20% less of TOP amount to compare with the sample in second experiment.

TOP amount (%)	Se (mg)	TOP (ml)	ODE (ml)
180%	30	0.72	5
100%	30	0.40	5
80%	30	0.32	5

Table 1: The percentages weight of TOP amount

Chapter 4-Results and Discussions

4.1 Results

4.1.1 First experiment-the effects of TOPSe to the growth of CdSe and the cold down effect with ice water

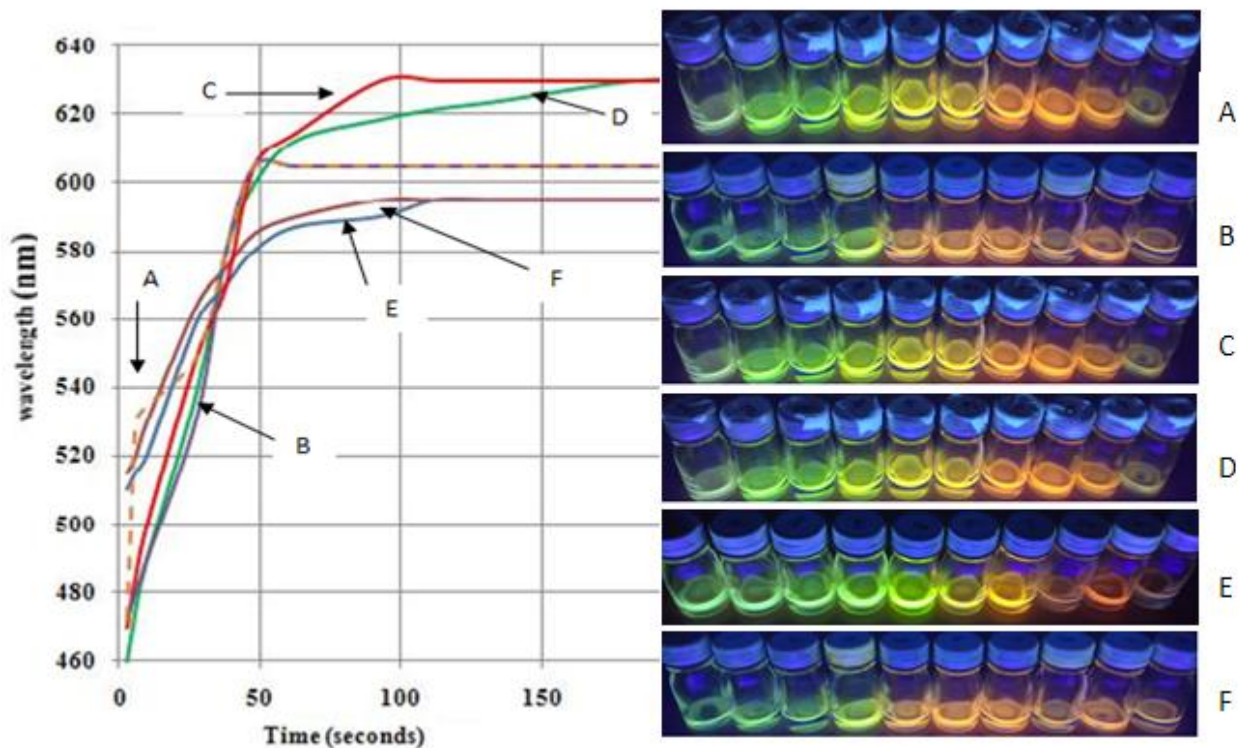


Figure 5: The comparison in term of wavelength of CdSe in different time for 1, 2, 3 ml TOPSe stock for cool in ice water ($\sim 4^{\circ}\text{C}$) and room temperature ($\sim 27^{\circ}\text{C}$) [A] 1ml TOPSe stock (w/o ice), [B] 1 ml TOPSe stock (with ice), [C] 2ml TOPSe stock (w/o ice) , [D] 2 ml TOPSe stock (with ice), [E] 3 ml TOPSe stock (w/o ice), [F] 3ml TOPSe stock (with ice).

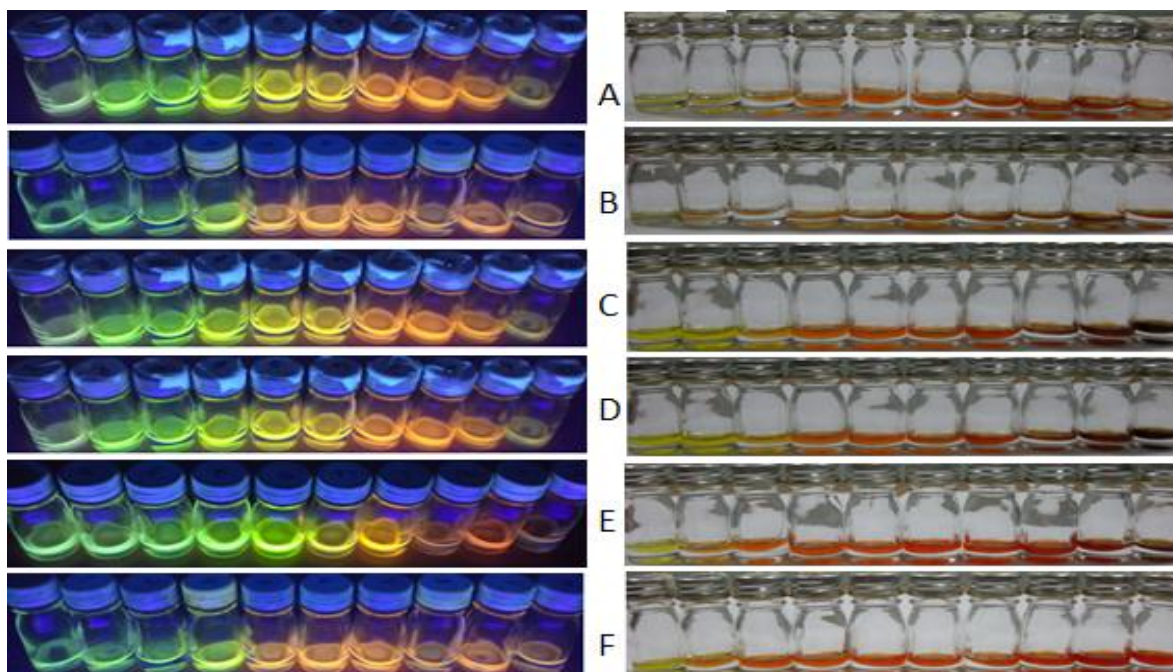


Figure 6: The comparison of the sample for 1, 2, 3 ml TOPSe stock for cool in ice water ($\sim 4^{\circ}\text{C}$) and room temperature ($\sim 27^{\circ}\text{C}$) under ambient light and UV light. [A] 1ml TOPSe stock (w/o ice), [B] 1 ml TOPSe stock (with ice), [C] 2ml TOPSe stock (w/o ice) , [D] 2 ml TOPSe stock (with ice), [E] 3 ml TOPSe stock (w/o ice), [F] 3ml TOPSe stock (with ice).

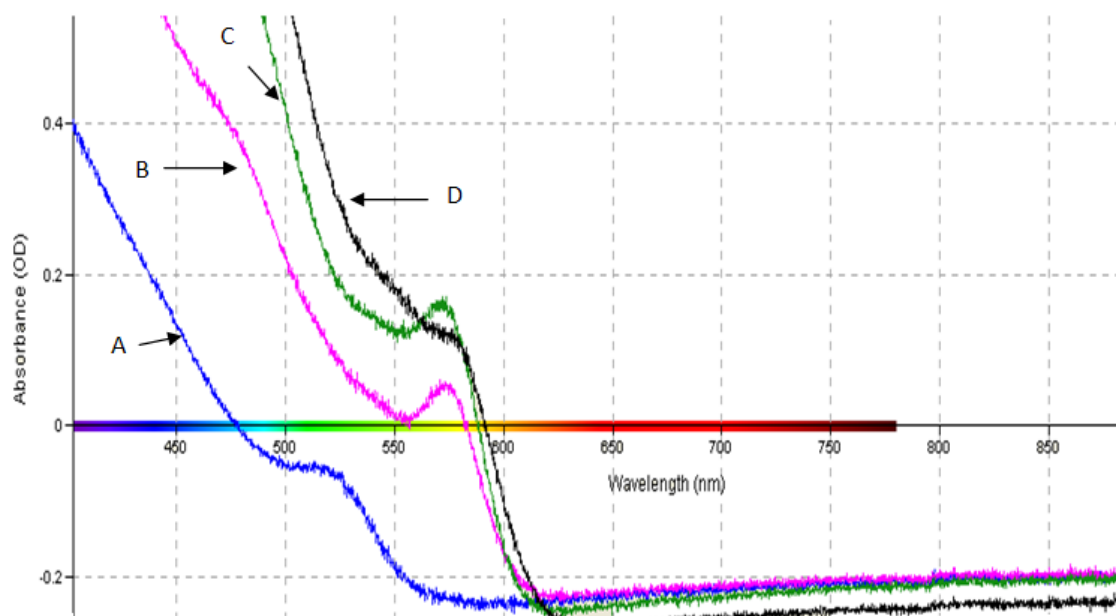


Figure 7: The comparison in term of wavelength of CdSe in different time for 1, 2 ml TOPSe stock for cool in room temperature ($\sim 27^{\circ}\text{C}$). [A] sixth seconds 1 ml TOPSe stock, [B] forty-eight seconds 1 ml TOPSe, [C] sixth seconds 2 ml TOPSe stock, [D] forty-eight seconds 2 ml TOPSe.

4.1.2 Second experiment-the effects of reaction media-TOP to the growth of CdSe

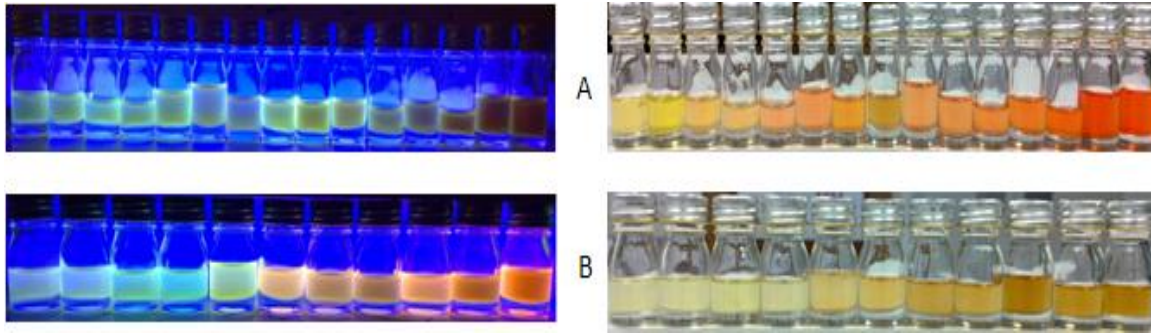


Figure 8: The comparison of the sample for different TOP amount. [A] 80 % more TOP stock
[B] 20% less TOP stock

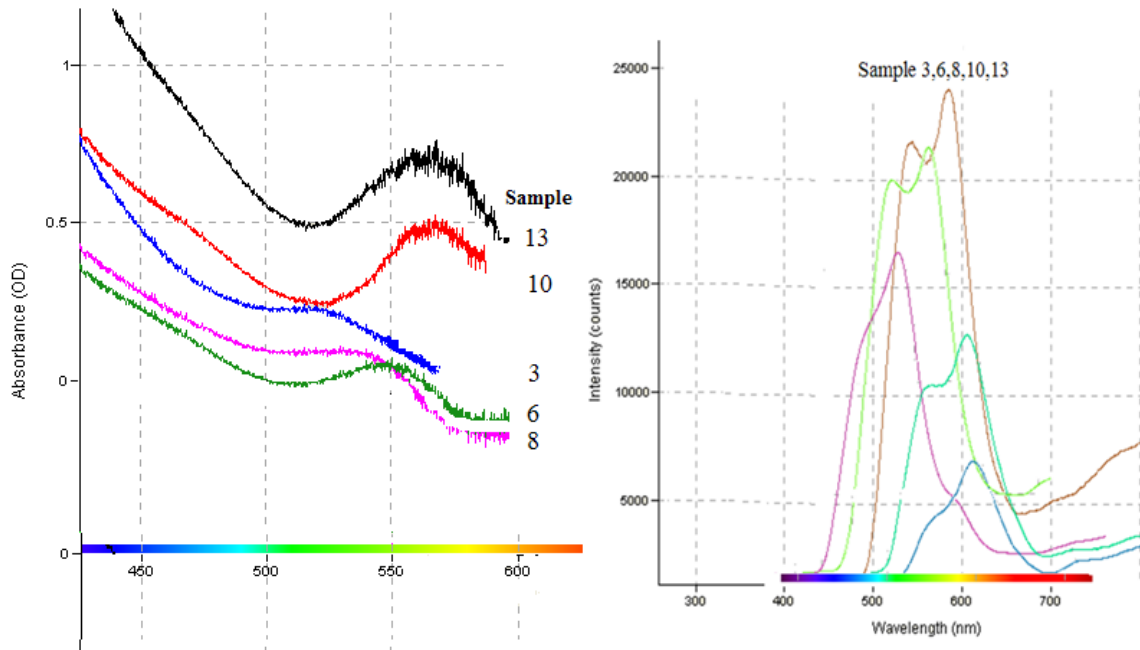


Figure 9: The absorbance and intensity graph for sample with 80% more TOP stock

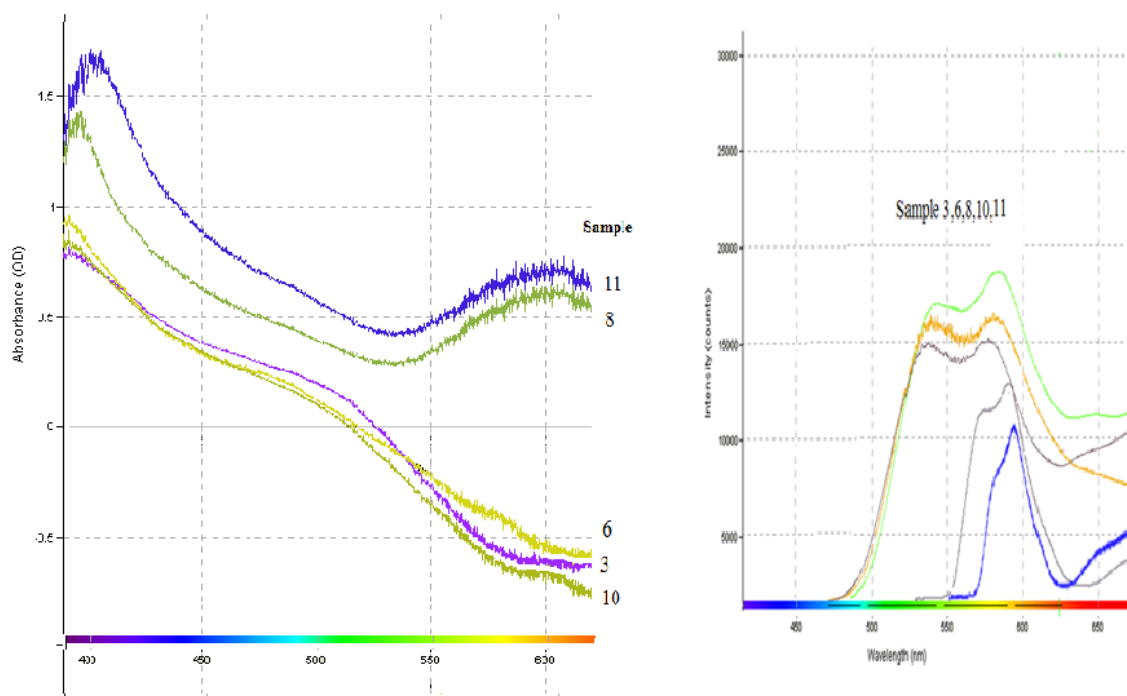


Figure 10: The absorbance and intensity graph for sample with 20% less TOP stock

4.1.3 Third experiment-the effects of temperature to the growth of CdSe

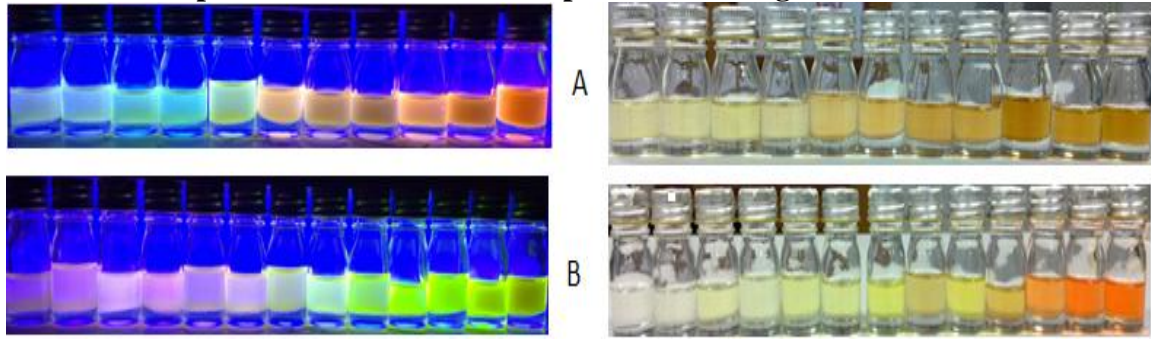


Figure 11: The comparison of the sample for different growth temperature. [A] 240°C CdSe product [B] 200°C CdSe product

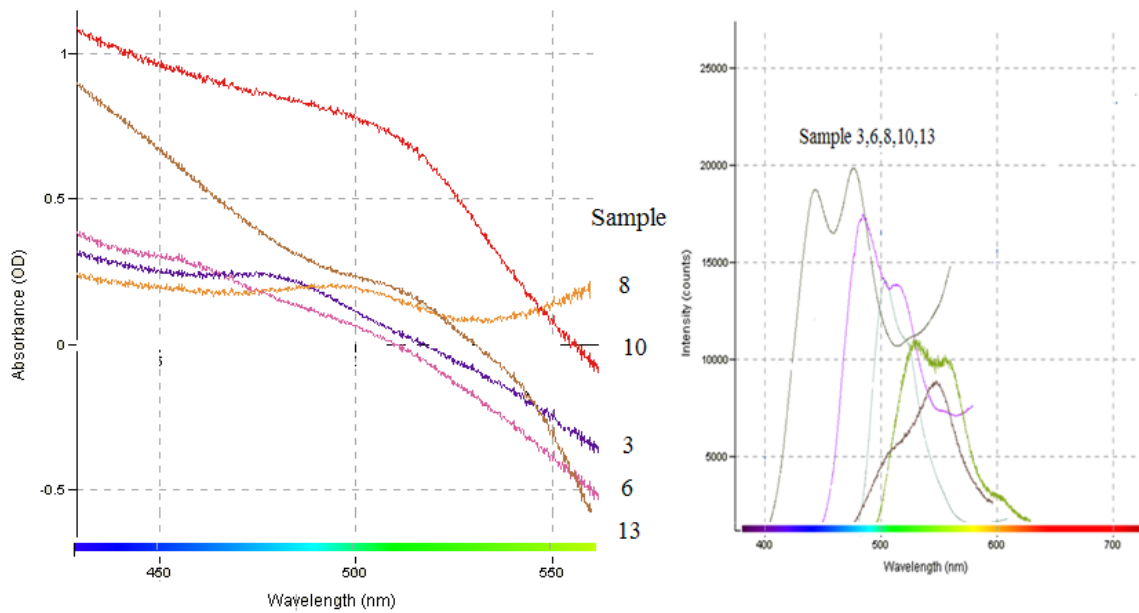


Figure 12: The absorbance and intensity graph for sample growth in 200°C

4.2 Discussions

During the experiment process, we observe the trend and reaction of CdO and octadecene when is heat together, and we found that the solution is brownish colour in room temperature until temperature reach $\sim 120^{\circ}\text{C}$. From $\sim 120^{\circ}\text{C}$ to $\sim 180^{\circ}\text{C}$, the solution turn to transparent colour, at this stage we can see the CdO powder is fully dissolve in the octadecene and oleic acid. After $\sim 180^{\circ}\text{C}$, the solution is yellowish. For Se precursor, the Se powder dissolves after heat for 7 minutes at final temperature of 42°C . The stock solution then is stored to let it cold down to room temperature.

4.2.1 First experiment-the effects of TOPSe to the growth of CdSe and the cold down effect with ice water

We produced the sample ranging from 400 nm to 620 nm (violet to orange) in visible light spectrum. We then compared the six sample in figure 6 and found that the luminescence level under florescence light (white light) ultra-violet light (UV) for the sample with 3 ml TOPSe stock (figure 6 [E-F]) is better compare to 1 ml and 2 ml TOPSe stock. With this we believe that the sample with 3 ml TOPSe stock have higher quantum yield compare with the sample with 1 ml and 2 ml TOPSe stock.

In our studied, we found that there are researcher who used some cold down method like the liquid nitrogen and other non-coordinating reagent such as octadecene (ODE) and hexane as cold down medium ^[91-92]. Therefore we tried to study the effect if we cold down the sample with the ice water ($\sim 4^{\circ}\text{C}$). We found that the sample which cold down in the ice water ($\sim 4^{\circ}\text{C}$) have the shorter range of wavelength when explore under UV light compare with the samples cold down in room temperature ($\sim 27^{\circ}\text{C}$). The problem of this method is the time of the sample cold down in ice water must be handling well. Based on our observation, if the sample which cold down in ice water being handle well the result will be give a more identical different with the sample which cold down in room temperature. On the other hand, the results may not have obvious different between the two set sample if the time does not handle properly.

Besides that, the volumes of TOPSe stock are also affecting the CdSe product growth time where the higher volume of TOPSe stock, the faster the growth. This mean it is more difficult to get the smaller wavelength of CdSe product in higher volume TOPSe stock. These shows in Figure 7 where the comparison was made in term of absorbance wavelength of CdSe product for 1, 2 ml TOPSe stock which cold down in room temperature ($\sim 27^{\circ}\text{C}$).

4.2.2 Second experiment-the effects of reaction media-TOP to the growth of CdSe

TOP is used to cap Se source to control the synthesis of CdSe growth. In this session, we studied the effect of the reaction media-TOP to the growth of CdSe. Figure 8 shows the comparison of the sample for different TOP amount, from the observation; we found that more TOP amount does not help to increase the quantum yield of the CdSe product. Our product is ranging from 495nm-650 nm (Green- Red). We concluded that the more TOP being used would not help to increase the quality of the quantum dots but it is also a kind of waste to the chemical.

We also found that the photoluminescence intensity for the sample with 80% more TOP is higher compare to the sample with 20% less TOP. Figure 9 shows the absorbance and intensity graph for sample with 80% more TOP stock while Figure 10 shows the absorbance and intensity graph for sample with 20% less TOP stock. The two intensity graphs have the same trend; when the wavelength increase, the intensity is decreasing.

4.2.3 Third experiment-the effects of temperature to the growth of CdSe

In this session, we studied the temperature effect to the growth of CdSe product. Figure 11 shows the comparison of the sample for different growth temperature. We found that the sample which growth in 200°C have the wavelength range from 420 nm to 520 nm (Violet-Green) while the sample growth in 240°C have the wavelength range from 520 nm to 650 nm (Green –Red) which shows in figure 10 and 12. With this observation, we found that to get the temperature are also affecting the CdSe product growth time where the higher the temperature, the faster the growth. This mean it is more difficult to get the smaller wavelength of CdSe product in higher growth temperature. We also found that the photoluminescence intensity is decreasing, when the wavelength increase.

We observed the samples synthesized under 200°C the sample colours reverse back. This is because in an ensemble of nanocrystals in solution, if the monomer concentration in the solution is higher than the solubility of all nanocrystals, all nanocrystals will grow. If nucleation is stopped at this moment by varying the reaction conditions, such as reducing the monomer concentration and/or temperature, the size distribution of the ensemble will be reduced rapidly.

In the whole studied, we can conclude that the effect of the reaction medium-TOPSe amount does affect the quality of CdSe product produced. The higher the amount of TOPSe used, the higher the quality and wider range of product. While the effect of the reaction medium-TOP, we found that it does not have an obvious different between the

two sample we produced. Therefore, we conclude that the Se source will be also will capped even the TOP used is 20% lesser than the amount calculated with the Stoichiometry in our formula. In the last part of the studied, we observed there are major different in the two set of samples therefore we conclude that the temperature does affect the growth of CdSe.

Chapter 5: Conclusion and Recommendation

5.1 Conclusion

In this studied, we observed the characteristic and the luminescence level CdSe by varying the reaction medium and temperature. We found that higher TOPSe is better for quality of CdSe product, while higher temperature increase growth rate. On the other hand, the amount of TOP used would not help to increase the quality of the CdSe product. Two important considerations which should be take care while fabricating CdSe quantum dots are the luminescence of the quantum dots, and the colour of the quantum dots. For the luminescence of the quantum dots, it is closely related to quantum yield of the CdSe. Increasing the quantum yield will increase the degree of luminescence as the quantum yield means the total amount of energy being absorb or emit by the quantum dots and the energy will carry by photon thus light will be seen. We recommended that the cooling process should be included in the fabrication procedure because it will stop the growth of the quantum dots and be able to obtain more precise sample.

5.2 Recommendation

We suggest in order to cold down the samples in ice water, the time for cold down must be carry on well to have a identical different. Besides that, we suggest the temperature of the CdSe product can be set to $\sim 225^{\circ}\text{C}$ in order to get full spectrum product in one set sample. We recommend the best volume of TOPSe to use is 2ml, to produce a better quality of CdSe product. This is because the effective attachment of TOP on the quantum dots surface at the higher concentration may result in the effective blocking of electrons and holes recombination on the nanocrystals surface or in other word, the quantum dots will have better passivation.

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Appendix A: The total apparatus and chemical used

Apparatus

- 1) Analytical balance (0.0001 gram)
- 2) 1 timer
- 3) 1 stirrer hotplate
- 4) 1 pc of metal spatulas
- 5) 1 pc of 10 ml beaker
- 6) 3 pc of 1 ml syringe with needle
- 7) 1 pc of 10 ml syringe without needle
- 8) 1 pc of magnetic stir bar
- 9) 2 pc of 50 ml conical flask
- 10) 1 pc of pasteur pipet with bulb
- 11) 1 pc of 300°C thermometer
- 12) 40 pcs of test tubes
- 13) Retort stand and test tubes holder
- 14) Tissue Papers

Chemical (1 sets of sample)

- 1) 13 mg of Cadmium Oxide (CdO) (Product number: Sigma-Aldrich 202894)
- 2) 30 mg of Selenium Powder (Se) (Product number: Sigma-Aldrich 209651)
- 3) 0.6 ml of Oleic Acid (Product number: Sigma-Aldrich 364525)
- 1) 15 ml of 1-Octadecene (Product number: Sigma-Aldrich O806)
- 2) 0.4 ml of Trioctylphosphine (TOP) (Product number: Sigma-Aldrich 117854)

Appendix B: The Lab Manual

WARNING: Read the safety precaution before start the procedure and always wear the personal protective equipment (PPE) throughout the experiment.

- 1) First label a 50 ml conical flask as flask A, a 50 ml conical flask label as flask B, a 50 ml conical flask label as flask D, and a 10 ml beaker is label as beaker C. Then fill beaker C with water which is prepare to clean the apparatus.

- 2) Put flask A into the analytical balance, press “Reset to zero” button in order to eliminate the weight of the flask and to take the actual amount of the Se powder.

- 3) With a spatula, add Se powder in the flask A which inside the analytical balance exactly 30 mg. After that, remove the flask A from the analytical balance.

WARNING: Weight the Se powder carefully to avoid dusty environment in the analytical balance.

- 4) With a 10 ml syringe, add 5 ml Octadecene into a flask A. Then, with a 1 ml syringe, add 0.4 ml Trioctylphosphine (TOP) into the flask A.

- 5) Add the magnetic stir bar into flask A and put the flask on the hot plate to heat and stir the solution. Heat and stir the solution until the Se powder is fully dissolve the solution. Then let the solution cool to room temperature. (Move the flask to the retort stand with a clamp for cooling down process)

WARNING: Make sure the magnetic stir bar diameter is less than the diameter of the conical flask, to avoid the magnetic stir bar to break flask.

WARNING: The speed of the magnetic stir bar must well control to avoid the solution spill out

- 6) Put flask B into the analytical balance, press “Reset to zero” button in order to eliminate the weight of the flask and to take the actual amount of the CdO powder.

- 7) With a spatula, add CdO powder in the flask B which inside the analytical balance exactly 13 mg. After that, remove the flask B from the analytical balance.

WARNING: Weight the CdO powder carefully to avoid dusty environment in the analytical balance.

- 8) With a 10 ml syringe, add 10 ml Octadecene into a flask B. Then, with a 1 ml syringe, add 0.6 ml Oleic acid into the flask B.

- 9) Add the magnetic stir bar into flask B and put the flask on the hot plate to heat and stir the solution. Heat and stir the solution with the hot plate until the temperature reaches 225°C. Maintain the solution temperature at 225°C.

WARNING: Make sure the magnetic stir bar diameter is less than the diameter of the conical flask, to avoid the magnetic stir bar to break flask.

WARNING: The speed of the magnetic stir bar must well control to avoid the solution spill out

- 10) With 1 ml the syringe, transfer the 1ml of Se solution which prepared in flask A to the flask B which contains the cadmium solution. Then start timing.

- 11) With the pipet, an approximately 1 ml sample is taken out from the solution in flask B at the intervals of time and put it into the test tubes. The first sample is taken at the time of 6th seconds, and the following four samples were taken at 10 seconds interval from the first sample and 20 seconds interval after the fifth sample.

- 12) The samples are cold down in ice water (~4°C) immediately about 5 seconds.

- 13) Repeat step 6 to 11 in flask D and the sample is cold down under room temperature.

Appendix C: Fume hood layout

Suggested Fume Hood Layout

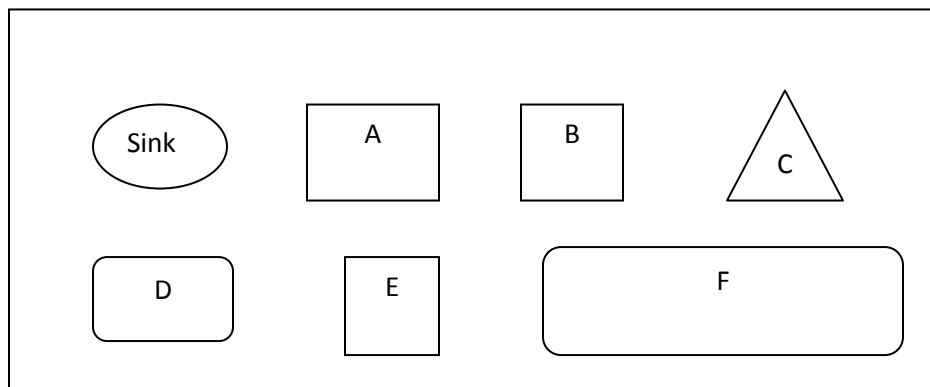


Figure C-1: Fume Hood Layout

A= Retort Stand (place for the Se solution to cool down to room temperature)

B= Analytical Balance

C=Se powder, CdO, TOP, 1-Octadecene, Oleic Acid

D= Beaker C (Cleaning Apparatus), Tissue paper

E= Hot plate

F= Test tubes with Test tubes stand, Pipet, thermometer, Syringe

Appendix D: Safety Precaution Step during the study

- 1) Avoid physical contact with cadmium oxide, selenium and cadmium selenide as they are carcinogens. **(WARNING: DO NOT PRODUCE DUSTY ENVIRONMENT, INHALATION, BY ABSORPTION THROUGH SKIN AND EFFECT OF INGESTION ARE CUMULATIVE)**
- 2) Read the Chemical Safety Information. A Material Safety Data Sheet (MSDS) should be available for every chemical you use in lab. Read these and follow the recommendations for safe use and disposal of the material.
- 3) Se, Octadecene vapor and CdO is an inhalation hazard and this operation should be done in a fume hood.
- 4) Trioctylphosphine (TOP) is corrosive and causes burns; it should be transferred exclusively by syringe from its Sure-Seal bottle.
- 5) Oleic acid is air, light and heat sensitive and irritating to eyes, respiratory system and skin. Make sure it is refrigerated after used.
- 6) Leftover CdO, Se, CdSe and Octadecene should be collected in a waste container for proper disposal.
- 7) Glassware must be in sterile condition.
- 8) While using the thermometer, do not measure the heat continuously, on the other hand the temperature must be taken from time to time because the thermometer may break and cause mercury leakage. Do not stir the mixture using thermometer.
- 9) Make sure the magnetic stir bar diameter is less than the diameter of the conical flask, to avoid the magnetic stir bar to break the flask.
- 10) Identify all the safety equipments and know how to use them as some people (possibly you) will need them. Know the locations of the fire blanket, extinguishers, eyewash, and shower.
- 11) Don't Eat or Drink in Lab

Appendix E: Clean up Method

How to Dispose Chemical

- Solvents should be put into the solvent waste bin.
- Solvents should NEVER be put down the sink.
- Acids should NEVER be put into the solvent container.
- Chemicals containing cadmium and selenium cannot go down drain and need to be put in a container. Environmental health & safety must be called to pick up.

Cleaning Up Fume Hood/Glass ware (General)

Fume Hood

- After you had disposed the chemicals properly, wipe off the counter space where you were working.
- Dispose the gloves in their proper location.
- Remove all PPE.
- Never leave bottles on the floor or in the fume hood.
- Turn off hot plate when you are doing using it.

Glassware

- Clean thoroughly and rinse 3 times all glass and plastic ware and put on the pegs to dry.
Note: It is inadvisable to dry glassware with a paper towel or forced air since this can introduce fibers or impurities that can contaminate the solution. Normally you can allow glassware to air dry on the shelf. Otherwise, if you are adding water to the glassware, it is fine to leave it wet (unless it will affect the concentration of the final solution).
- Pipets and Volumetric Flasks, in some cases, you may need to soak the glassware overnight in soapy water. Clean pipets and volumetric flasks using warm soapy water. The glassware may require scrubbing with a brush. Rinse with tap water followed by 3-4 rinses with deionized water.

Note: Much of the time, detergent and tap water are neither required nor desirable. You can rinse the glassware with the proper solvent, and then finish up with a couple of rinses with distilled water, followed by final rinses with deionized water. Cleaning Up (Chemical Spill)

- If you spill a chemical, ensure that you or others have not been exposed to any of the spilled chemical.
 - If someone has been exposed, rinse the exposed area in the sink or shower for 15 minutes minimum, and stay with the individual for the entire time they are rinsing. (Point out where shower and eye wash stations are located.)
 - Contact clean room staff or the supervisor
 - Seek medical attention.
 - Refer to Emergency Instructions for more details.
- Make sure you have the proper personal protective equipment on.
- For a small spill: wipe up with a tex wipe and throw the wipe in the trash.
- For a large spill: clear out the area and inform the supervisor. DO NOT try to clean a large spill yourself