UNIVERSITI PUTRA MALAYSIA

STRUCTURAL AND OPTICAL PROPERTIES OF ZINCSELENIDE NANOCRYSTAL AND QUANTUM DOTS COLLOIDAL PREPARED VIA HYDROTHERMAL ROUTE

FAKHRURRAZI ASHARI

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By

FAKHRURRAZI ASHARI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science

March 2017

DEDICATION

To my beloved parents Hj Ashari and Hjh Ruziah

For their love and concern….

To my friends (too many)

For their wonderful encourgament and support…

To my supervisor Dr. Jospehine Liew Ying Chyi

For her guidance, advice, understanding and endless support…

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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Chair : Josephine Liew Ying Chyi, PhD
Faculty : Science

Zinc Selenide which show a potential application in electronic devices such as photovoltaic devices, light emitting devices and photodetector has been synthesized through a hydrothermal method. It has been found that the hydrothermal reaction conducted at 180 °C for 32 hours is optimum to produce NCr ZnSe, while the hydrothermal reaction at 150 °C for 8 hours is optimum for ZnSe QDs. Formation of ZnSe in nanocrystal (NCr) and quantum dots (QDs) using different source and different concentration ratio has been study in detail to optimize the production condition. The concentration effect of the precursor ($ZnCl_2$/ $Zn(O_2CCH_3)_2(H_2O)_2$) which varied from 0.25:1, 0.5:1, 1:1, 2:1, 3:1 until 4:1 has been explained in terms of its structured, morphology, composition and the optical behavior. The structural, morphology, compositional and optical properties of the nanocrystal and quantum dots were study using X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), Transmission Electron Microscopy (TEM), X-ray Fluorescence (XRF), UV-Vis NIR Spectroscopy (UV-Vis) and Photoluminescence (PL). The difference of NCr and QDs of ZnSe has been explored in detail through their structural properties, particle size, absorption and optical band gap (E_g) also emission peak. For NCr, the particle size is around 0.08 µm to 15.7 µm for both precursor ($ZnCl_2$/ $Zn(O_2CCH_3)_2(H_2O)_2$). The system structure is cubic structure ($ZnCl_2$) and sphere structure ($Zn(O_2CCH_3)_2(H_2O)_2$). While for QDs, the particle size is around 6.2 nm to 49.1 nm ($ZnCl_2$/ $Zn(O_2CCH_3)_2(H_2O)_2$) and the structure system is cubic and sphere structure. All this will affect and show the difference of optical behavior between NCr and QDs and it is related to the quantum confinement effect especially for the QDs samples. The absorption peak is at 460 nm, while the E_g value at 2.60 eV and emission peak is at 486 nm (blue region). The result is for NCr while for QDs, the absorption peak around 313 nm to 317 nm with E_g value is around 3.60 eV to 3.80 eV with emission peak at range 450 nm to 490 nm. The optical behavior will show the difference between NCr and QDs sample, when the size is smaller the color emitted light will shift from red to blue shift while the optical band gap (E_g) will increase. So, it has found that the optical behavior is strongly affected by the morphological behavior of the nanocrystal and quantum dots

Abstrak thesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Sarjana Sains

PENCIRIAN STRUKTUR DAN OPTIK ZINK SELENIDA NANOHABLUR DAN TITIK KUANTUM KOLOID DISEDIAKAN MELALUI KAEDAH HIDROTERMA

Oleh

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Zinc selenide menunjukkan potensi yang bagus dalam peranti elektronik seperti peranti photovoltaic, peranti pemancar cahaya dan pengesan foto setelah disintesis melalui kaedah hidroterma. Ia telah mendapati bahawa tindak balas hidroterma yang dijalankan pada suhu 180°C selama 32 jam adalah optimum untuk menghasilkan NCr ZnSe, manakala tindak balas hidroterma pada suhu 150°C selama 8 jam adalah optimum untuk ZnSe QDs. Pembentukan ZnSe dalam nanocrystal (NCr) dan titik kuantum (QDs) menggunakan sumber yang berbeza dan nisbah kepekatan yang berbeza telah di kaji secara terperinci untuk mengoptimumkan jumlah pengeluaran. Kesan kepekatan pelopor ($ZnCl_2/Zn(O_2CCH_3)_2(H_2O_2)$) yang dibezakan daripada 0.25:1, 0.5:1, 1:1, 2:1, 3:1 sehingga 4:1 telah dijelaskan dari segi struktur, morfologi, komposisi dan tingkah laku optik. Struktur, morfologi, komposisi dan sifat-sifat optik nano hablur dan titik kuantum telah dikaji menggunakan pembelauan sinar X (XRD), Field Pelepasan Pengimbasan Elektron Mikroskopi (FESEM), Transmisi Elektron Mikroskopi (TEM), Pendarfluor sinar X(XRF), UV-Vis NIR Spektroskopi (UV-Vis) dan Photoluminescence (PL). Perbezaan NCr dan QDs daripada ZnSe telah diterokai secara terperinci melalui sifat-sifat struktur, saiz zarah, penyerapan, jurang jalur optik (E_g) dan juga pelepasan puncak. Untuk NCr, saiz zarah adalah sekitar 0.08 mikron sehingga 15.7 mikron untuk kedua-dua pelopor ($ZnCl_2/Zn(O_2CCH_3)_2(H2O)_2$). Struktur sistem ialah struktur kubus ($ZnCl_2$) dan struktur sfera ($Zn(O_2CCH_3)_2(H_2O)_2$). Manakala bagi QDs, saiz zarah adalah sekitar 6.2 nm sehingga 49.1 nm ($ZnCl_2$/ $Zn(O_2CCH_3)_2(H_2O_2)$) dan sistem struktur adalah struktur kubus dan sfera. Semua ini akan memberi kesan dan menunjukkan perbezaan ciri optik antara NCr dan QDs dan ia berkaitan dengan kesan pantang kuantum terutamanya bagi sampel QDs. Puncak penyerapan ialah pada 460 nm, manakala nilai E_g pada 2.60 eV dan pelepasan puncak adalah pada 486 nm (kawasan biru). Hasilnya ialah untuk NCR manakala bagi QDs, puncak penyerapan sekitar 313 nm ke 317 nm dengan nilai Eg adalah sekitar 3.60 eV 3.80 eV dengan puncak pelepasan pada jarak 450 nm kepada 490 nm. Kelakuan optik akan menunjukkan perbezaan antara sampel NCR dan QDs, apabila saiz yang lebih kecil cahaya warna yang dipancarkan akan beralih dari merah ke biru manakala jurang jalur optik (E_g) akan meningkat. Jadi, ia telah di dapati bahawa tingkah laku optik amat dipengaruhi oleh tingkah laku morfologi nanocrystal dan kuantum titik-titik.

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I certify that a Thesis Examination Committee has met on 16 March 2017 to conduct the final examination of Fakhrurrazi bin Ashari on his thesis entitled "Structural and Optical Properties of Zinc Selenide Nanocrystal and Quantum Dots Colloidal Prepared via Hydrothermal Route" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Masters of Science. The members of the Supervisory Committee were as follows:

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α	absorption coefficient
°C	degree of Celsius
d	interatomic spacing
e	electron
E_g	Energy band gap
hv	photon energy
n	electronic transitions
V	voltage
Θ	angle of incident
λ	wavelength
XRD	X-Ray diffraction
UV-Vis	Ultraviolet- visible
FESEM	Field emission scanning electron microscopy
PL	Photoluminescence
JCPDS	Joint committee on powder diffraction standards
TEM	Transmission electron microscopy
HRTEM	High resolution transmission electron microscopy
XRF	X-ray fluorescence
EDX	Energy dispersive X-ray

CHAPTER 1

INTRODUCTION

1.1 Introduction

Semiconductor is a material which has conductivities between conductors and insulator. It plays an importance role for the fundamental part of computer, switching, amplification and modern electronics. As reported by Yang et al. (2010), semiconductor is widely used in optoelectronic devices such as diode laser, thin film solar panel and light emitting diode (LEDs). Cadmium telluride (CdTe), Tin selenide (SnSe), Zinc sulfide (ZnS) and zinc selenide (ZnSe) are some examples of semiconductor materials. An increased knowledge of semiconductor materials and fabrication processes, modern scientist need the understanding of quantum physics to explain the movement of electrons and holes, electrical, thermal and optical properties of semiconductor inside a lattice. It will help to resolve the complexity of integrated semiconductor devices. Therefore, there are several techniques used to synthesis semiconductor material especially zinc selenide (ZnSe) where are precipitate reaction (Andrade et al. 2009), sol-gel process (Wang et al., 2004), chemical solution method (Song et al., 2010) and hydrothermal method (Peng et al., 2010)

In this study, zinc selenide (ZnSe) was selected because it is well-known as II–VI semiconductor which is suitable for light emitting devices, and window material in thin film solar cells due to its wide energy band gap, i.e. 2.7 eV (Kowalik et al., 2008).

Moreover, Zinc Selenide (ZnSe) is widely used in light-emitting diodes (LED), diode lasers, and infrared optical material. In daily life, it can be found as the entrance optic in the new range of "in-ear" clinical thermometers or as a small yellow window.

Lately, researchers are interested to know the changes on the fundamental properties of quantum dot to enhance the optical properties based on the size. Quantum dots is a nanocrystal material whose diameter is smaller than its exciton Bohr radius that are enough to lead to quantum confinement effect and exhibit quantum mechanical properties (Murray et al., 2000). The electrical and optical properties will be highly tunable if the size is small enough and dominate to the quantum confinement effect (less than 10 nm). Yang et al. (2007) reported, ZnSe QDs was successfully synthesis through hydrothermal route with the average particle size around 4.8 nm with strong emission peak at 458 nm. Other that, the absorption of ZnSe QDs is shifted to higher energy (380 nm) compared with to the bulk (460nm) and emission peak at 470 nm (blue region) (Andrade et al., 2009). Quantum dot is closely related to the size and shape of particle. The size of quantum dot is inversely related to the optical band gap energy (E_g), which determine the frequency range of emitted light. Therefore, color of the emitted light shift from red to blue when the sizes of the quantum dot is different even the quantum dots is the same material.

Quantum dots are particularly significant for optical applications due to their high extinction coefficient. This is due to the high ability of quantum dots to absorb light at a given wavelength and related to the light absorption. The potential applications are in electronic such as photovoltaic devices, light emitting devices and photodetector devices.

1.2 Problem Statements

Amongst the semiconductor clusters, quantum dots have been great attention over the past of two decade especially colloidal quantum dots. Researchers are interested to know the changes on the fundamental properties of quantum dot and interested to improve the low performance of material. Following its discovery, interest in the Zinc Selenide QDs was focused towards its electrical, optical, morphology and the application in industrial area. A number of literatures have been reporting on the preparing of colloidal ZnSe QDs with several techniques such as sol-gel process, chemical solution method and precipitation method.

In addition, many researches have been studying on the optical absorption, optical band gap energy (E_g) and the photoluminescence of ZnSe QDs. There is a limitation of producing the semiconductor colloidal quantum such as the high temperature, the time limitation and the amount of final product. Therefore, a detailed investigation of the structural, morphological and optical properties for the production of colloidal ZnSe QDs through hydrothermal method is presented in this report.

1.3 Objective of Research

The objectives of this study are:

- To optimize the optimum parameter on synthesis ZnSe powder and colloidal ZnSe quantum dots through the hydrothermal route.
- To investigate the quality of product using different precursor concentration ($ZnCl_2$ and $Zn(O_2CCH_3)_2(H_2O)_2$) on the structural and optical properties of the ZnSe powder and colloidal ZnSe quantum dots formed.
- To analyze the optical phenomena such as the energy band gap (E_g) and emission peak of ZnSe powder and colloidal ZnSe quantum dots through photoluminescence (PL) and UV-Vis spectroscopy.

1.4 Thesis Outline

The thesis is organized into 5 chapters starting from introduction (Chapter 1) until conclusion and recommendation (Chapter 5). Chapter 1 covered brief introduction about semiconductor materials, quantum dots and background of Zinc Selenide (ZnSe). The objectives of the project were described in detail in chapter 1.

Chapter 2 provides brief literature review on semiconductor material which covered the previous works on ZnSe QDs. This includes the reports on characterization of the structure, optical properties and the application. This chapter also reviews the work which related to the preparation technique of ZnSe and colloidal ZnSe QDs in detail.

Chapter 3 is about methodology, which explains about the raw materials used for preparation of both powder and colloidal form of ZnSe powder and colloidal ZnSe QDs. The experimental procedure and preparation of ZnSe powder and colloidal ZnSe QDs conducted in laboratory are given in this chapter. Lastly, the structural and optical characterization method such as XRD, XRF, EDX, FESEM, HRTEM, UV-Vis spectrometer and Photoluminescence (PL) has been studies.

Results and discussion for each sample ratio from 0.25 until 4.0 of ZnSe powder and colloidal ZnSe QDs for the structural and optical properties characterization were documented in Chapter 4. All the data of the experiment were listed, analyzed and interpreted in the form of graphs, table, charts, diagrams as well as pictures.

Finally, the last chapter (chapter 5) summarizes all results obtained from the project as conclusion. Recommendations and suggestion are provided for future studies.

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