



UNIVERSITI PUTRA MALAYSIA

***MICROWAVE ASSISTED SYNTHESIS AND PHOTOCATALYTIC
ACTIVITY OF ZNS AND CDS QUANTUM DOT SYSTEMS***

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ACTIVITY OF ZNS AND CDS QUANTUM DOT SYSTEMS**

By

NAYEREH SOLTANI

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

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DEDICATION

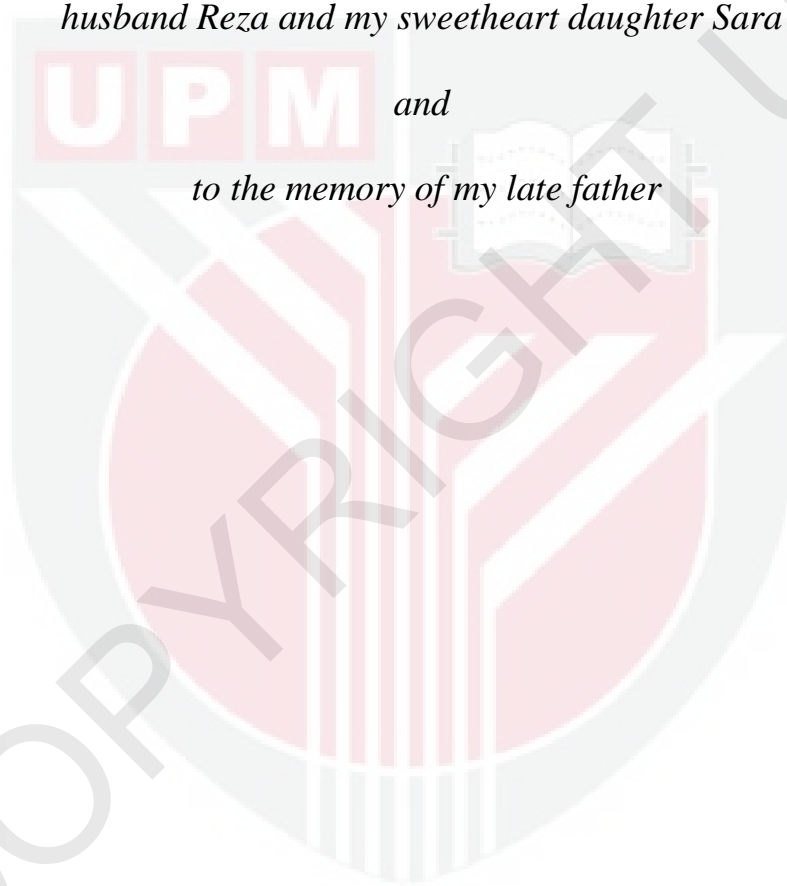
In appreciation of their love, sacrifices, faith, and eternal goodness

I would like to dedicate my thesis to my dear mother, my beloved

husband Reza and my sweetheart daughter Sara

UPM and

to the memory of my late father



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment
of the requirement for the degree of Doctor of Philosophy

**MICROWAVE ASSISTED SYNTHESIS AND PHOTOCATALYTIC
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November 2012

Chairman: Professor Elias Saion, PhD

Faculty: Science

Solar energy is an alternative sustainable energy resource that can be harvested using semiconductor quantum dots including zinc sulfide (ZnS) and cadmium sulfide (CdS). However, at present is only a small fraction of the sun's energy (< 10%) absorbed by using pure ZnS and CdS semiconductor solar cells. To increase the overall photoactivity of these nanomaterials, various selective surface modifications can be used during synthesis process such as depositing the surface with organic or inorganic materials that causes charge transfer and electronic interaction between the surface attachment and the host semiconductor.

In this work, pure ZnS and CdS quantum dots, PVP capped ZnS and CdS quantum dots, and ZnS/CdS and CdS/ZnS core shells were prepared by using the microwave irradiation technique involving water and polyol solvents. Pure ZnS and CdS semiconductor quantum dots were synthesized by both the Microwave-Hydrothermal (M-H) and Microwave-Polyol (M-P) methods, polymer-capped ZnS and CdS by the

M-H method, and ZnS/CdS and CdS/ZnS core shells by the M-P method. The concentration of precursors was adjusted by varying the molar ratio of zinc and cadmium sources to sulfur source (1:1, 1:1.25, 1:1.5, and 1:2) in a fixed irradiation time (20 min). To study the effect of microwave irradiation, the reactions were carried out in 5, 10, 15, 20, 25, 30 and 40 min irradiation time using a pulse regime of 20% power (on for 5 s, off for 20 s). The formation of ZnS and CdS nanoparticles have been observed by the change in color of samples from colorless to white for ZnS nanoparticles and to yellow for CdS nanoparticles and confirmed by powder X-ray diffraction (XRD).

The crystal structure of ZnS and CdS nanocrystals synthesized in both the M-H and M-P method are cubic and hexagonal, respectively. The particle size of nanocrystals was determined using Scherrer's equation from XRD spectra and transmission electron microscopy (TEM). The estimated average sizes in the M-H method are between 3.3 and 4.8 nm for ZnS and between 9.7 and 12.5 nm for CdS nanoparticles depending on the irradiation time. In the M-P method, the average sizes are between 3.2 and 4.9 nm for ZnS and between 8.9 and 11.4 nm for CdS. The size of nanoparticles was also calculated by the Brus formula according to the UV-Visible spectrum which agrees fairly well with those determined from the XRD spectra and TEM images.

The optical band gap of ZnS and CdS nanoparticles was calculated from Tauc plot using UV-Visible spectra. The estimated band gaps of the M-H method samples are between 4.24 and 4.30 eV for ZnS and between 2.61 and 2.66 eV for CdS nanoparticles. For the M-P method, the optical band gaps are between 4.00 and 4.42

eV for ZnS, and between 2.62 and 2.67 eV for CdS. The blue shift of the absorption edge compared to that of bulk clearly explained the quantum confinement effect. Photoluminescence spectra of the samples all exhibited two individual peaks corresponding to zinc or cadmium vacancies and sulfur vacancies.

The size of PVP-capped ZnS and CdS nanoparticles slightly increased compared to the bare ones. Consequently the absorption peaks shifted to higher wavelengths. The average particle sizes and estimated optical band gaps of synthesized nanoparticles in appropriate PVP concentration (5%) were 5.1 nm and 4.07 eV for ZnS and 18.3 nm and 2.53 eV for CdS. PL spectra of PVP-capped ZnS nanoparticles de-convoluted into three different bands which were attributed to zinc and sulfur vacancies, and surface trap states. PL spectra of PVP-capped CdS showed two green emission peaks related to cadmium and sulfur vacancies.

The XRD spectra of ZnS/CdS and CdS/ZnS core shell nanoparticles were similar to the XRD pattern of the core materials. The estimated average sizes of ZnS/CdS core shell nanoparticles were 11.2 and 14.6 nm corresponding to shell thickness of 3.4 and 5.1 nm, respectively. For CdS/ZnS core shell nanoparticles the average particle sizes were 17.0 and 20.7 nm corresponding to shell thickness of 3.4 and 5.3 nm. The optical properties of ZnS/CdS and CdS/ZnS core shell nanoparticles in the visible region are dominated by CdS since its band gap is smaller than that of ZnS and both the conduction band and the valence band of CdS are located within the energy gap of ZnS. PL spectra of core shell nanoparticles de-convoluted into four different bands that could be attributed to zinc, cadmium and sulfur vacancies, and the radiative recombination of carriers at interfaces.

The photocatalytic activity of the prepared nanoparticles was examined using dye degradation and water splitting for hydrogen production under illumination by a halogen lamp as a visible light source. To obtain the best condition of photocatalytic activity, samples were categorized in three groups. In the first group, pure ZnS and CdS nanoparticles were mixed together with ZnS:CdS weight ratios of (1:0), (0:1), (1:2), (2:1), (1:3), (3:1), (1:4) and (4:1). In the second group, PVP capped ZnS and CdS were mixed together with the same weight ratios of the first group and in the third one, photocatalytic activity of ZnS/CdS and CdS/ZnS core shells were examined. The results show that photocatalytic activity of samples in the first and second groups depends on the weight ratio of mixture. Using a typical 0.2 mg weight of photocatalyst quantum dots, the highest photocatalytic dye degradation rate after 120 min illumination in the three groups were 4.82×10^{-3} , 9.15×10^{-3} and 7.56×10^{-3} min^{-1} , respectively. In the water splitting reaction the maximum amount of hydrogen evolution after 6 hours in the three groups were 629, 736 and 1560 μmol respectively.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**SINTESIS BANTUAN MIKROGELOMBANG DAN AKTIVITI
FOTOPEMANGKINAN ZNS DAN CDS BINTIK KUANTUM SYSTEMS**

Oleh

NAYEREH SOLTANI

November 2012

Pengerusi: Profesor Elias Saion, PhD

Fakulti: Sains

Tenaga suria adalah sumber tenaga alternatif yang mampan yang boleh dituai menggunakan semikonduktor bintik kuantum termasuk zink sulfida (ZnS) dan kadmium sulfida (CdS). Walau bagaimanapun, pada masa ini hanya pecahan kecil daripada tenaga suria (<10%) diserap dengan menggunakan sel solar semikonduktor ZnS dan CdS tulen. Untuk meningkatkan fotoaktif keseluruhan bahan-bahan nano ini, pelbagai jenis pengubahsuaian permukaan terpilih boleh digunakan semasa proses sintesis seperti mendepositkan permukaan dengan bahan organik dan bukan organik untuk pemindahan caj dan tindak balas elektronik diantara lampiran permukaan dan semikonduktor tuan rumah.

Dalam penyelidikan ini, bintik kuantum tulen ZnS dan CdS, bintik kuantum ZnS dan CdS bersalut PVP, dan tempurung teras ZnS/CdS dan CdS/ZnS telah disediakan dengan menggunakan teknik sinaran mikrogelombang dalam pelarut air dan pelarut polioliol. Bintik kuantum semikonduktor tulen ZnS dan CdS telah disintesis dengan

kedua-dua kaedah Mikrogelombang-Hidroterma (M-H) dan Mikrogelombang-polyol (M-P), ZnS dan CdS bersalut PVP dengan menggunakan kaedah M-H dan tempurung teras ZnS/CdS dan CdS/ZnS dengan menggunakan kaedah M-P. Kepekatan prekursor telah diubah dengan mengubah nisbah molar sumber zink dan kadmium kepada sumber sulfur (1:1, 1:1.25, 1:1.5, dan 1:2) dalam masa sinaran yang tetap (20 minit). Untuk mengkaji kesan sinaran nanogelombang, tindak balas itu telah dijalankan pada 5, 10, 15, 20, 25, 30 dan 40 minit masa sinaran menggunakan denyut rejim pada tenaga 20% (buka pada 5 s, tutup pada 20 s). Pembentukan nanozarah ZnS dan CdS telah dipatuhi melalui perubahan pada warna sampel daripada tidak berwarna kepada putih untuk nanozarah ZnS dan daripada tidak berwarna kepada kuning untuk nanozarah CdS dan disahkan melalui pembelauan serbuk X-ray (XRD).

Struktur kristal nanokristal ZnS dan CdS yang telah disintesis dalam kedua-dua kaedah M-H dan M-P adalah masing-masing berbentuk kubik dan heksagon. Saiz zarah untuk nanokristal telah ditentukan melalui persamaan Scherrer daripada spektrum XRD dan mikroskopi transmisi elektron (TEM). Anggaran purata saiz dalam kaedah M-H adalah di antara 3.3 dan 4.8 nm untuk nanozarah ZnS dan di antara 9.7 dan 12.5 nm untuk nanozarah CdS bergantung kepada masa sinaran. Dalam kaedah M-P, saiz purata adalah di antara 3.2 dan 4.9 nm untuk nanozarah ZnS dan di antara 8.9 dan 11.4 nm untuk nanozarah CdS. Saiz nanozarah telah dikira dengan menggunakan formula Brus berdasarkan kepada spektrum UV-tampak dimana secara langsung adalah setuju dengan yang ditentukan daripada spektrum XRD dan imej TEM. Jurang jalur optikal untuk nanozarah ZnS dan CdS telah dikira daripada plot Tauc menggunakan spektrum UV-tampak. Anggaran jurang jalur

melalui kaedah M-H adalah di antara 4.24 dan 4.30 eV untuk nanozarah ZnS dan di antara 2.61 dan 2,66 eV untuk nanozarah CdS. Dalam kaedah M-P, jurang jalur optikal adalah di antara 4.00 dan 4.42 eV untuk nanozarah ZnS dan di antara 2.62 dan 2.67 eV untuk nanozarah CdS. Peralihan biru pinggir penyerapan berbanding pukal telah dijelaskan melalui kesan pengurangan kuantum. Spektrum fotoluminesens (PL) sampel mempamerkan dua puncak berbeza yang sepadan dengan kekosongan zink atau kadmium dan kekosongan sulfur.

Saiz nanozarah ZnS dan CdS bersalut PVP telah meningkat sedikit berbanding dengan yang tulen. Dengan itu, puncak penyerapan beralih kepada ke arah panjang gelombang yang lebih tinggi. Purata saiz zarah dan anggaran jurang jalur optik untuk nanozarah yang disintesis dengan kepekatan yang sesuai PVP (5%) masing-masing adalah 5.1 nm dan 4.07 eV untuk nanozarah ZnS dan 18.3 nm dan 2.53 eV untuk nanozarah CdS. Spektrum PL untuk nanozarah ZnS bersalut PVP dikonvolusi kepada tiga puncak yang berbeza disebabkan oleh kekosongan zink dan sulfur, dan keadaan perangkap permukaan. Spektrum PL untuk nanozarah CdS bersalut PVP menunjukkan dua pemancaran puncak sinaran hijau yang berkaitan dengan kekosongan kadmium dan sulfur.

Dua sampel disediakan setiap nanozarah tempurung teras ZnS/CdS dan CdS/ZnS yang didapati mempunyai corak XRD yang sama seperti bahan teras. Purata saiz anggaran untuk nanozarah tempurung teras ZnS/CdS adalah 11.2 dan 14.6 nm dengan ketebalan masing-masing 3.4 dan 5.1 nm. Untuk nanozarah tempurung teras CdS/ZnS anggaran purata saiz partikel adalah 17.0 dan 20.7 nm dengan ketebalan masing-masing 3.4 dan 5.3 nm. Sifat-sifat optik nanozarah tempurung teras ZnS/CdS

dan CdS/ZnS dalam rantau cahaya tampak didominasi oleh CdS kerana jurang jalurnya lebih kecil berbanding ZnS dan kedua-dua jalur konduksi dan jalur valens CdS adalah berada dalam lingkungan jurang tenaga ZnS. Spektrum PL untuk nanozarah tempurung teras dikonvolusi kepada empat puncak berbeza yang mungkin disebabkan oleh kekosongan zink, kadmium dan sulfur, dan rekombinasi pengangkut sinaran di antaramuka.

Aktiviti fotopemangkinan untuk nanozarah yang disediakan telah diselidik menggunakan penurunan pewarna dan pemecah air untuk penghasilan hidrogen di bawah pencahayaan lampu halogen sebagai sumber cahaya tampak. Untuk mendapatkan keadaan terbaik aktiviti fotopemangkinan, sampel telah dikategorikan kepada tiga kumpulan. Dalam kumpulan pertama nanozarah tulen ZnS dan CdS di campur bersama dengan dengan nisbah berat ZnS: CdS (1:0), (0:1), (1:2), (2:1), (1:3), (3:1), (1:4) dan (4:1). Dalam kumpulan kedua, ZnS dan CdS bersalut PVP di campur bersama dengan nisbah berat yang sama seperti kumpulan pertama dan dalam kumpulan ketiga, aktiviti fotopemangkinan untuk tempurung teras ZnS/CdS dan CdS/ZnS telah diselidik. Keputusan menunjukkan bahawa aktiviti fotopemangkinan sampel daripada kumpulan pertama dan kedua adalah bergantung kepada nisbah berat daripada campuran tersebut. Sebagai contoh bagi 0.2 mg berat bintik kuantum fotopemangkin yang digunakan kadar tertinggi untuk penurunan pewarna fotopemangkinan selepas 120 minit pencahayaan dalam tiga kumpulan masing-masing adalah 4.82×10^{-3} , 9.15×10^{-3} dan $7.56 \times 10^{-3} \text{ min}^{-1}$. Untuk tindak balas pemecah air untuk penghasilan hydrogen, jumlah tertinggi penghasilan hidrogen selepas 6 jam dalam ketiga-tiga kumpulan masing-masing adalah 629, 736 dan 1560 μmol .

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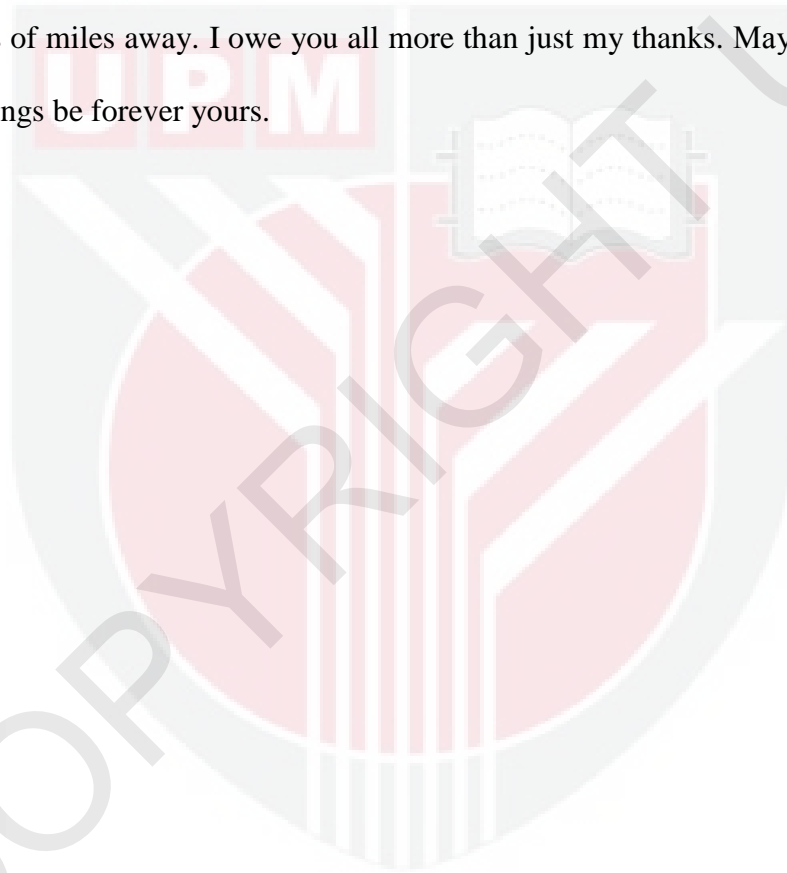
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I certify that a Thesis Examination Committee has met on 2 November 2012 to conduct the final examination of Nayereh Soltani on her thesis entitled “Microwave assisted synthesis and photocatalytic activity of II-VI semiconductor quantum dots” in accordance 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 Doctor of Philosophy.

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

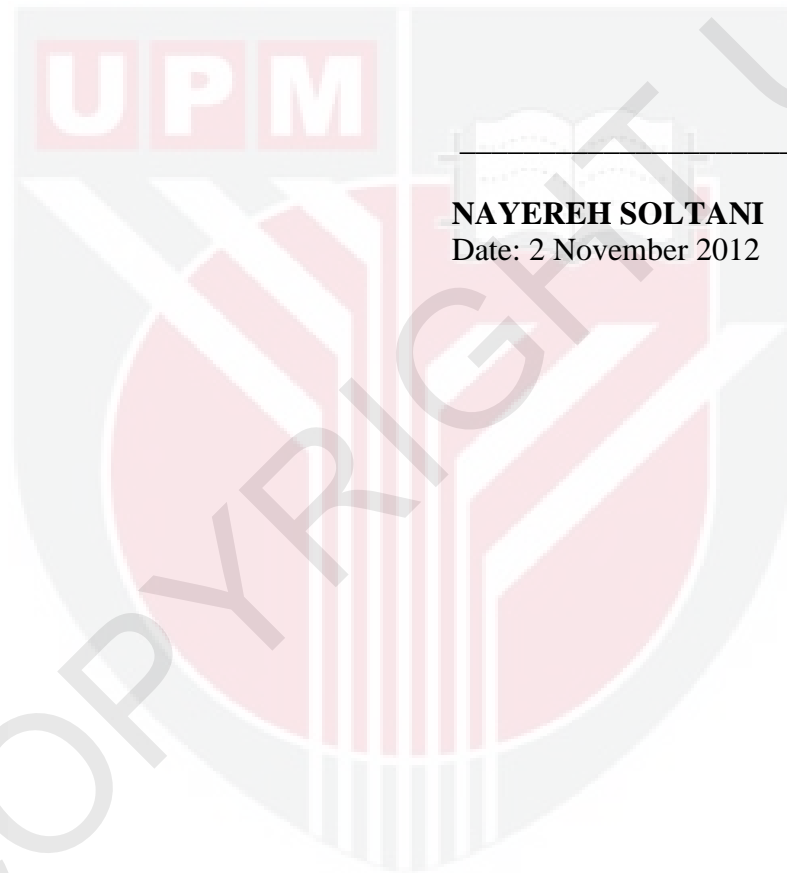


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