UNIVERSITI PUTRA MALAYSIA

STRUCTURAL, OPTICAL AND DIELECTRIC PROPERTIES OF PBS AND ZNS NANOPARTICLES SYNTHESIZED VIA MICROWAVE IRRADIATION

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By

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

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The solar energy harvesting technology can be achieved by using semiconductor nanomaterial including lead sulphide (PbS) and zinc sulphide (ZnS) nanoparticles. Therefore, the modification of various properties of PbS and ZnS nanoparticles can be achieved by controlling their size may provide an advantage in producing new materials with optimized properties for many applications including photovoltaic, solar cells and other photo-electronic devices. In microwave irradiation synthesis, the effect of power, irradiation time and the solvent choice can impact the nature of the reaction and are still a major problem in preparing a particular range of nanoparticle size. Hence, in this research, different particles sizes of ZnS and PbS are prepared by microwave irradiation method involving distilled water, ethylene glycol, ethylene alcohol and isopropanol as various solvents used. The concentration of the precursors of zinc and lead sources to sulphur source (1:1), the solvents quantity of 40 ml, power ($300\ W$) and the irradiation time are fixed. The particles sizes were determined using Scherrer's equation from XRD spectra and from transmission electron microscopy (TEM). The crystallite sizes of PbS synthesized in ethylene glycol, distilled water, ethylene alcohol and isopropanol ranged from 28.7 to 43.3 nm and their corresponding estimated optical band gaps ranged from 2.53 to 2.20 eV. For ZnS nanoparticles the crystallite sizes ranged from 8.2 to 13.0 nm and their corresponding optical band gaps ranged from 3.52 to 3.34 eV. The effect of irradiation time on optical and structural properties of PbS and ZnS nanoparticles was also investigated, a number of samples are obtained by varying the irradiation time from 10, 15, 45 and 50 min for both the two samples. The reactions carried out in a fixed concentration, power (200 W) and ethylene alcohol as solvent. The crystallite sizes of PbS nanoparticles ranged from 32. to 65.8 nm and their estimated optical band gaps values ranged from 2.41 to 1.60 eV. For ZnS nanoparticles, the average particle sizes in different irradiation time ranged from 7.9 to 87.01 nm and their corresponding optical band gaps ranged from 3.42 to

3.26 eV. The FESEM studies for ZnS nanoparticles showed the effect of irradiation time on morphology on each particles size of PbS and ZnS nanoparticles. The PVP/PbS and PVP/ZnS nanocomposites are synthesized in ethylene glycol solution in 10, 20 and 30 min irradiation time. The final products are characterized using FTIR, XRD and UV-VIS spectroscopy. The dielectric properties of the obtained different sizes of PVP/PbS nanocomposites (23.4, 28.7 and 52.4 nm) and PVP/ZnS (7.9, 13.0 and 64.9 nm) are analysed using impedance spectroscopy at constant temperature of 303 K and frequency range of 40 Hz to 1 MHz. The maximum value of AC conductivity of each size of PVP/PbS (52.4, 28.7, 23.4 nm) ranged from 9.56 x 10^{-6} down to 3.55 x 10^{-6} S/cm, while for PVP/ZnS (64.9, 13.0, 7.9 nm), the values ranged from 1.34 x 10^{-5} down to 7.21 x 10^{-6} S/cm. The corresponding values for the DC conductivity are also found. In this work, the values of electrical conductivities are decreased as the particle sizes of the samples reduced from 64.9 down to 7.9 nm for PVP/ZnS nanocomposites while from 52.4 down to 23.4 nm for PVP/PbS nanocomposites. The synthesized nanocomposites with particles sizes of 64.9 and 52.4 nm are considered to be the nanocomposites possessing the best particle sizes for the applications of electronic devices due to their excellent electrical conductivity as compared to other samples.

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

SIFAT STRUKTUR, OPTIK DAN DIELEKTRIK NANOZARAH PBS DAN ZNS YANG DISINTESIS MELALUI PENYINARAN I MIKROGELOMBANG

Oleh

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Teknologi penuaian tenaga solar boleh dicapai dengan menggunakan nanobahan semikonduktor termasuk nanopartikel sulfida plumbum (PbS) dan sulfida zink (ZnS). Oleh itu, pengubahsuaian terhadap pelbagai sifat nanozarah PbS dan ZnS yang boleh dicapai dengan mengawal saiznya dapat memberi kelebihan dalam menghasilkan bahan-bahan baru yang mempunyai sifat yang dioptimumkan untuk pelbagai aplikasi termasuk fotovoltaik, sel suria dan alat peranti foto-elektronik yang lain. Dalam sintesis penyinaran mikrogelombang, kesan kuasa, masa penyinaran dan pemilihan pelarut dapat memberikan impak pada keadaan reaksi dan masih merupakan masalah utama dalam menyediakan pelbagai saiz nanopartikel tertentu. Oleh itu, dalam penyelidikan ini, saiz partikel yang berbeza bagi ZnS dan PbS telah disediakan menggunakan kaedah penyinaran mikrogelombang yang melibatkan air suling, etilena glikol, etilena alkohol dan isopropanol sebagai penggunaan pelbagai pelarut. Konsentrasi prekursor bagi sumber zink dan plumbum pada sumber sulfur (1:1), kuantiti pelarut (40 ml), kuasa (300 W) dan masa penyinaran , telah ditetapkan. Saiz partikel telah ditentukan menggunakan persamaan Scherrer dari spektra XRD dan dari mikroskop elektron penghantaran (TEM). Jangkaan purata saiz bagi PbS dalam etilena glikol, air suling, etilena alkohol dan isopropanol adalah dalam julat antara 28.7 hingga 43.3 nm dan anggaran kesepadanan jurang jalur optikal mereka adalah dalam julat dari 2.53 hingga 2.20 eV. Bagi nanozarah ZnS pula , purata saiz zarah dalam etilena glikol,air suling, etilena alkohol dan isopropanol adalah dalam julat dari 8.2 hingga 13.0 nm dan kesepadanan jurang jalur optikal adalah dalam julat dari 3.52 hingga 3.34 eV. Kesan masa penyinaran ke atas sifat optikal dan struktural nanozarah PbS dan ZnS juga diselidiki; sejumlah sampel telah diperoleh dengan mempelbagaikan masa penyinaran daripada 10, 15, 45 dan 50 minit bagi kedua-dua sampel. Reaksi telah dijalankan dalam konsentrasi, kuasa dan pelarut yang tetap. Purata saiz zarah bagi nanozarah Pbs adalah dalam julat dari 32.3 hingga 65.8 nm dan

anggaran jurang jalur optikal adalah dalam julat dari 2.41 hingga 1.60 eV. Bagi nanozarah ZnS, purata saiz zarah dalam masa penyinaran yang berbeza, masing-masing adalah dalam julat dari 7.9 hingga 87.01 nm, dan jurang jalur optikal adalah dalam julat dari 3.42 hingga 3.26 eV. Kajian FESEM bagi nanozarah ZnS menunjukkan kesan masa penyinaran ke atas morfologi setiap saiz zarah bagi nanozarah PbS dan ZnS. Nanokomposit PVP/PbS dan PVP/ZnS telah disintesiskan dalam larutan glikol etilena , dalam masa penyinaran 10, 20 dan 30 minit. Produk akhir telah dicirikan dengan menggunakan spektroskopi FTIR, XRD dan UV-VIS. Sifat dieletrik bagi pelbagai saiz yang diperoleh bagi nanokomposit PVP/PbS (23.4, 28.7 dan 52.4 nm) dan PVP/ZnS (7.9, 13.0 dan 64.9 nm) telah dianalisis menggunakan spektroskopi impedans pada suhu yang malar, iaitu 303 K dan julat kekerapan, dari 40 Hz hingga 1 MHz . Nilai maksimum konduktiviti AC bagi setiap saiz PVP/PbS (52.4, 28.7, 23.4 nm) adalah dalam julat dari 8.96 x 10^{-6} turun kepada 4.59 x 10^{-6} S/cm. Bagi nanokomposit PVP/ZnS (64.9, 13.0, 7.9 nm) nilai adalah dalam julat dari 6.06 x 10^{-5} turun kepada 8.79 x 10^{-5} S/cm. Nilai kesepadanan bagi konduktiviti DC juga masing-masing diperoleh. Dalam kajian ini, nilai konduktiviti eletrik masing-masing telah diturunkan ketika saiz zarah bagi sampel dikurangkan dari 64.9 turun kepada 7.9 nm bagi nanokomposit PVP/ZnS, manakala dari 52.4 turun kepada 23.4 nm bagi nanokomposit PVP/PbS. Nanokomposit yang telah disintesis dengan saiz zarah 64.9 dan 52.4 nm dianggap sebagai nanokomposit yang mempunyai saiz zarah terbaik untuk aplikasi peranti elektronik kerana kekonduksian elektriknya yang sangat baik berbanding dengan sampel lain.

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

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LIST OF ABBREVIATIONS

CO_2	Carbon dioxide
CB	Conduction band
CBM	Conduction band maximum
CTEM	Conventional transmission microscopy
DOS	Density of states
EDX	Energy dispersive x-ray
EG	Ethelene glycol
EtOH	Ethelene alcohol
eV	Electron volt
EIS	Electrochemical impedance spectroscopy
EMA	Effective mass approximation
EHF	Extremely high frequency
FTIR	Fourier Transform Infrared Spectroscopy
FWHM	Full width at half maximum
FESEM	Field emission scaning microscopy
.ΔE	Blue-shift energy
GeTe	Germanium tellurides
GeSe	Germanium Monoselenide
H_2O	Distilled water
HRTEM	High resolution transmission electron microscopy
ISO	Isopropanol
IS	Impedance spectroscopy
LED	Light emitting diode
MC	Metal chalcogenide

NaCl	Sodium chloride
PV	Photovoltaic
PVP	Polyvinylpyrrolidone
PbSe	Lead selenide
PbTe	Lead telluride
PbS	Lead sulfide
QDs	Quantum dots
Sn	Tin
S	Sulfur
SnSe	Tin selenide
SnS	Tin sulfide
SEM	Scaning electron microscopy
SHF	Super-high frequency
TEM	Transmission electron microscopy
UV-Vis	Ultraviolet and visible
UHF	Ultra-high frequency
VBM	Valence band maximum
VB	Valence band
XRD	X-ray diffraction
K	Kelvin
Ω	Ohms
nm	Nanometer(10^{-9})

CHAPTER 1

INTRODUCTION

1.1 Background of study

Renewable energy sources offer our planet a chance to lower carbon emissions, provide a clean air and place our civilization on a more sustainable footing. Recently there has been an essential interest in renewable sources of energy (Balamurugan *et al.*, 2011). This has been driven by an increase on non-renewable energy prices globaly due to economic and geopolitical influence and the regular burden associated with global warming that is intensify by the emission of green gases as a result of conventional means (Solangi *et al.*, 2011). Although several advanced technologies have been designed to supplement non-renewable energy as a fundamental energy source, while renewable energy sources are considered as the key to long-term weaning of the industrialize economics from perfect dependence on oil, natural gas, and coal. These consist of wind, solar cell, geothermal, biofuels and many more. Among those sources of energy, solar energy remains ultimately as a sustainable choice in terms of its availability and its vast potential (Jacobson and Delucchi, 2011). The amount of incident solar energy on earth's surface at any given time is more than 8200 times the available amount of energy from the sun annually (Islam and Morimoto, 2018). Modern silicon photovoltaic cells can have a power conversion efficiency of about 15-20% range. Although they are significantly better than biofuel production, they are still about 10 times more costly than using fossil fuels. Interest in semiconductor quantum dots for applications in the solar cell has been extensive for the last 5-10 years as a result of their inherent band gap energy tenability through the entire solar spectrum (Chu *et al.*, 2017)

Quantum dot based solar cells can potentially access the sun with theoretical power conversion efficiency of 66% (ie an infinite layered multi-junction cell) (Razykov *et al.*, 2011). Current single bandgap solar cells have a theoretical efficiency limit of 31%. Quantum dot based solar cells could improve the overall cost of solar energy conversion through higher efficiency cells based on material that is inexpensive to produce compared with single crystal silicon devices (Snaith, 2013).

1.2 Solar energy and Photovoltaic devices

The sun provides energy to the earth surface as a radiation distributed across the electromagnetic spectrum from infrared to ultraviolet wavelengths (Diffey, 2002). The amount of solar energy is accessible at the earth's surface in the direction of the sun is consistently 1000 W/m^2. In the 21st centuary, the energy from the sun is anticipated to provide a critical input to the global energy demand because it has more advantages over conventional energy (Hoffert *et al.*, 2002). Energy from the sun can be a source of heat at homes and industries and can also be applied into different aspects such as crop drying, outdoor and indoor pools, preheating boiler feed water et cetera (Mekhilef *et al.*, 2011). This solar energy minimizes cost and provides clean energy to the environment. The adaptability and safety of solar energy have expanded adequetly, composing it more pleasant options in either home or in business use.

Photovoltaic (PV) devices are generally typically consist set of thin layers semiconductor materials arranged to convert energy from the sun to direct-current (electricity) (Razykov *et al.*, 2011). When the device is exposed to sunlight energy, a cell from this device produces an electric current which is directly proportional to the magnitude of light energy it received. Photovoltaic cells can provide a boundless dependence on dwindling oil reserves and alleviate unfriendly effects to the environment (Chu and Majumdar, 2012). Presently, crystalline silica-based solar cells are the available form of Photovoltaic device, providing high solar conversion performance, simple of construction, defiance of degradation and plenty of siliceous initial materials. However, for ambitious alternative energy, the cost of photovoltaic needs to be decrease to at least 20% or less compare to the present cost (Dusonchet and Telaretti, 2010). For this purpose, there is a need to investigate an advanced, economical and capable photovoltaic material.

1.3 Blue-shift and the size dependent optical properties

The optical properties of semiconductors nanomaterial vary according to their particle sizes. A simple example is the gradual blue-shift in the absorption edge of nanoparticles as their size decreases (Xu *et al.*, 2015). The blue-shift correlation with particle size can be clearly explain from particle-in-a-box problem, which started the increase in energy level spacing when dimension of the box is reduced as a result of the quantum confinement. In this condition, the energy of the particles E, is directly proportional to $\frac{1}{a}$, where a reffered to the radius of the box. This explained that, as the box become smaller the energy of the particle increases (blue-shift). The most understood effect of quantum conferment is the blue shift of the first absorption feature with decreasing size of the nanoparticle. In addition, it can be understood using simple effective mass theory which considered parabolic conduction and valence bands with bulk effective masses for the electron and hole (Kiprotich *et al.*, 2018). Both electron and hole can be considered as particles within a sphere bond to the surface of nanoparticles by an infinite potential.

The width of the surface plasmon absorption and the frequency depend on the shape and the size of the metal nanoparticles, it also depends on the surrounding medium and the dielectric constant (Scholl *et al.*, 2012). When the nanoparticles size increases, the layer band which can be observed due to the plasmon absorption band shifted to red (Dorfs *et al.*, 2011). This phenomena can be explained by the Mie's theory, which solves Maxwell's equation and accounts for the scattering of electromagnetic radiation by any homogeneous and nonmagnetic spherical particle (Biju *et al.*, 2008).

In addition, the surface of nanoparticles is also a related factor that enormously affected the optical properties. Since the surface-to-volume ratio of nanomaterial increases as the size decreases, then the surface characteristics can also be affected (Ip *et al.*, 2012). For a spherical form of nanoparticles, its radius is inversely proportional to the its surface-to-volume ratio. This surface plays a significant role in the essential properties of nanoparticles. More dangling bonds and less adjacent coordinated atoms are very much on the surface of the atoms. These defects can cause additional electronic states within the band gap which become as traps for electron and hole (Voznyy *et al.*, 2013). These traps can be a source of reduction in the observed transition energy and a red-shifted emission band. Hence, the surface of the nanoparticles become more important when the size reduced (Wang *et al.*, 2012b).

1.4 Significance of the study

The manufacturing of materials at the nanosized has gained a much interest as it fills the gap between the bulk and atoms or molecules, thus improving our understanding of fundamental properties and providing new physical effects. This has been one of the exciting areas of research in recent years (Suresh, 2013). Owing to the quantum size effect of the semiconductor nanoparticles, the PbS and ZnS semiconductor nanoparticles in particular, exhibit size-dependent optical properties which are of great importance for potential applications, such as solar cells (Mastronardi *et al.*, 2011), light emitting diodes (Chang *et al.*, 2012), biological labels (Frigerio *et al.*, 2012) and optoelectronic devices (Baugher *et al.*, 2014). Therefore, modifying of the properties of these nanoparticles by controlling their size may provide an advantage in producing new materials with optimized properties for many applications. Most of the optical or electrical properties exhibited by these nanoparticles are due to their crystallite sizes.

1.5 Problem statement

It is well known that the carbon-rich source such as oil, coal and the natural gas are not the proper solution for increasing energy demand due to their negative impact to the environment including CO_2 emission, and their finite natural abundance. Solar energy, on the other hand, is the most sustainable and clean energy source that provides the earth with about 120,000 Terawatts of radiation, thus making it a very attractive source for green, sustainable and efficient energy conversion devices. Solar energy can be achieved by using semiconductor nanomaterial including lead sulphide (PbS) and zinc sulphide (ZnS) nanoparticles. Small sized nanoparticles allow the study of relevant surface properties due to the high surface to bulk ratio which can bring out new and enhanced physical and chemical properties which are different with a large-scale counterpart. Therefore, modifying of the properties of PbS and ZnS nanoparticles by controlling their size may provide an advantage in producing new materials with optimized properties for many applications including photovoltaic, solar cells and other photo-electronic devices. Most of the optical or electrical properties exhibited by these nanoparticles are due to their particle sizes.

To enhance the photoconductivity of PbS and ZnS semiconductors materials, various selective modifications can be considered during synthesis processes including organic or inorganic materials that cause charge transfer and electronic interaction between the surface attachment and the host semiconductor. In microwave irradiation synthesis, the solvent choice, irradiation time and the microwave power can impact the nature of the reaction and change the nature of the final product. Thus, studying the influence of such parameters can develop this method for producing required sizes and shapes for suitable applications. In most cases, capping of nanoparticles with polymers such as PVP gives rise to have the structures with increased thermal stability and decreased reactivity and tendency of agglomeration. However, this research desired to study the electrical transport of different particle sizes of PVP-capped PbS and ZnS nanoparticles synthesized by microwave irradiation route at low cost.

1.6 Objectives

To make contributions to the knowledge, this work involves fundamental research into the effect of various solvents to the particle sizes, the effect of different nanoparticles size on dielectric properties of nanomaterial, AC and DC conductivity. Furthermore, this study attempted to synthesize high purity of ZnS and PbS using different irradiation time via microwave irradiation method. The study objectives are summarized below;

1. To synthesize ZnS and PbS nanoparticles via microwave irradiation method with various solvents including distilled water (H_2O), ethylene glycol ($C_2H_6O_2$), ethylene alcohol (C_2H_5OH) and isopropanol (C_3H_8O) and also to determine the effect of each solvent on particle sizes structure and their optical band gap.
2. To determine the effect of microwave irradiation time on particle size, distribution and the band gap energy of PbS and ZnS nanoparticles.
3. To synthesize PVP/ZnS and PVP/ PbS nanocomposites using microwave irradiation method and to determine the effect of PVP on particle sizes and band gaps energy of the nanocomposites.
4. To investigate the effect of various particle size on dielectric properties, AC and DC conductivity of PVP/PbS and PVP/ZnS nanocomposites.

1.7 Thesis Outline

This thesis consists of six chapters. Chapter 1 begins with the overview of research background, problem statement and the research objectives. Chapter 2 reports literature review; the general introduction of Semiconductor materials, PbS and ZnS semiconductors, semiconductors nanoparticles and the underlying literature in view of the synthesis methods of semiconductors nanoparticles. Chapter 3 is focused on the optical and electronic structure of semiconductors, the theories of dimensional semiconductors structure, and microwave irradiation. Chapter 4 gives a brief description of the experimental methodology, techniques used to design, synthesize and characterize PbS and ZnS nanoparticles. The microwave irradiation method used to synthesize the nanoparticles is discussed in detail. A summary of the various characterization techniques is also given. This includes a description of the operation of each of the techniques including XRD, FTIR, UV-Vis, EDX, FESEM and the Impedance Spectroscopy. Chapter 5 reports the major part of this research, in which the experimental results are presented, analyzed and discussed in details. Finally, Chapter 6 gives a summary of the result of this work and suggestions for future work.

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LIST OF PUBLICATIONS

Influence of Solvents and Irradiation time on Structural and Optical Properties of Cubic PbS Nanoparticles: Published at International Journal of ELECTROCHEMICAL SCIENCE (© 2018 The Authors. Published by ESG : www.electrochemsci.org).

The effect of solvents on particle size of ZnS Nanoparticles Synthesized by Microwave Irradiation Route. A paper presented on 9th August, 2016 at Fundamental Science Congress 2016, Universiti Putra Malaysia.

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