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STRUCTURAL AND OPTICAL PROPERTIES OF GRAPHENE-BASED ZINC SELENIDE COMPOSITES PREPARED VIA MICROWAVE-ASSISTED HYDROTHERMAL METHOD FOR PHOTOVOLTAIC AND PHOTONICS APPLICATION

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By

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STRUCTURAL AND OPTICAL PROPERTIES OF GRAPHENE-BASED ZINC SELENIDE COMPOSITES PREPARED VIA MICROWAVE-ASSISTED HYDROTHERMAL METHOD FOR PHOTOVOLTAIC AND PHOTONICS APPLICATION

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April 2019

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The global energy demands will more than supply by the year 2040. In order to resolve this problem, the energy should be generated by using renewable source or reduce the energy consumption. Zinc selenide (ZnSe) has been widely used in applications such as LED, solar cell and other. There were researches on enhancing ZnSe properties by doping or mixing with other materials but there is not much reports on forming graphene-based ZnSe composite. In this research, graphene-based zinc selenide composites were synthesized via microwave-assisted hydrothermal method. Microwave-assised hydrothermal method was employed as it can greatly reduce the energy consumption, cost and time. Home made hydrothermal autoclave was used to charge the precursor and heated in the microwave oven. Through this method, the energy consumption, cost and reaction time were drastically reduced by 99%. The optimized condition to synthesis graphene-based ZnSe composite was using 0.100 mole of NaOH, 2ml of diethanolamine (surfactants) heated for 3 minutes under 700 W microwave irradiation power. The structural, morphological and optical properties of graphene-based zinc selenide composites were then characterized with X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Raman Spectroscopy, Field Emission Scanning Electron Microscope (FESEM), Atomic Force Microscope (AFM), Diffuse Reflectance Spectroscopy (DRS) and Photoluminescence (PL) Spectroscopy. For ZnSe/GO and ZnSe/GNP composites, cubic ZnSe was grown on the GO or GNP sheets which was observed in the AFM image. The spherical shape of pure ZnSe changed to nanoflakes as GO or GNP was added in as precursor. The purity of ZnSe formed was reduced from 100 % of ZnSe to 86.3 % as concentration of GO or GNP added was higher. The electron density in the sample was then determined via Fourier mapping. The electron density occupied the empty spaces within the cubic structure of ZnSe

as GO or GNP was added. From Raman spectra, it can be confirmed that ZnSe/GO and ZnSe/GNP composites were formed. The intensity ratio between D band and G band (I_d/I_g) was ~1 for ZnSe/GO composites and ~0.3 for ZnSe/GNP composites. The chemical bonding of GO and GNP was determined via FTIR technique where C=C, C-O, CH_3, C-H and $-COO^-$ bonding were found in ZnSe/GO and ZnSe/GNP composite. The optical band gap of ZnSe is 2.64 eV. The band gap changed to 2.62 eV and 3.02 eV as GO and GNP were added, respectively. The sample was excited by a wavelength of 290 nm where the PL emission peak for all samples were centered at ~466 nm. The samples were tested on the photovoltaic and photonic applications. The solar efficiency was increased from 0.19% (pure ZnSe) to 1.61 % when GO was added and 11.88 % when GNP is added. In addition, it has been proved that graphene-based ZnSe composite managed to generate femtosecond pulse of ~540 fs which can become a promising materials in photonics application such as micro- or nano-machining technology, non-linear imaging and microscopy and micro- and nano-surgery technology. Modification of ZnSe to form ZnSe/GO and ZnSe/GNP composite will change the electron density, morphology and optical properties. This research provides a fundamental knowledge to support the properties required for photovoltaic and photonics application.

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

SIFAT STRUKTUR DAN OPTIK KOMPOSIT ZINK SELENIDA BERASASKAN GRAFIN YANG DISEDIAKAN MELALUI KAEDAH HIDROTERMA DENGAN BANTUAN GELOMBANG MIKRO UNTUK APLIKASI FOTOVOLTAN DAN FOTONIK

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Permintaan tenaga secara global akan melebihi bekalan tenaga pada tahun 2040. Untuk menyelesaikan masalah ini, tenega harus dihasilkan dengan sumber boleh diperbaharui atau mengurangkan penggunaan tenaga. Zink selenida (ZnSe) digunakan dalam applikasi seperti LED, panel solar dan lain-lain. Terdapat penyelidikan unuk meningkatkan sifat-sifat ZnSe dengan mengedopkan atau memcampurkan bahan lain, tetapi jarang ada kajian untuk membentukkan komposit ZnSe berdasaskan grafin. Dalam kajian ini, komposit zink selenida berasaskan grafin telah disediakan dengan kaedah hidroterma dengan bantuan gelombang mikro. Autoklaf hidroterma khas telah digunakan untuk menempatkan pelopor dan dibakar dalam ketuhar gelombang mikro. Dengan menggunakan kaedah ini, pengunaan kuasa, kos dan masa tindak balas telah dikurangkan secara drastik iaitu 99 %. Keadaan optimum untuk sintesis komposit ZnSe berasaskan grafin adalah dengan menggunakan 0.100 mol NaOH, 2 ml dietanolamina (surfaktan) yang dibakar 3 minit di bawah kuasa gelombang mikro 700 W. Sifat-sifat struktur, morfologi dan optikal komposit ZnSe berasaskan grafin telah dikaji dengan Pembelauan Sinar-X, Spektroskopi transformasi Fourier inframerah, Raman spektroskopi, mikroskop elektron imbasan pancaran medan, mikroskop daya atom, spektroskopi reflektansi meresap dan spektroskopi fotoluminesen. Untuk komposit ZnSe/GO dan ZnSe/GNP, ZnSe kubik telah tumbuh di atas lapisan GO atau GNP dimana ia boleh diperhatikan dalam imej AFM. ZnSe berbentuk sfera telah bertukar menjadi serpihan-nano apabila kepekatan GO atau GNP ditambahkan sebagai pelopor. Ketulenan ZnSe telah berkurang dari 100 % ke 86.3 % apabila kepekatan GO atau GNP bertambah. Ketumpatan elektron dalam sampel telah dikaji dengan pemetaan Fourier. Ketumpatan elektron telah menduduki ruang kosong dalam struktur kubik ZnSe apabila GO atau GNP ditambahkan. Daripada spektrum Raman, ia dapat mengesahkan terbentuknya komposit ZnSe/GO dan ZnSe/GNP. Nisbah keamatan antara jalur D dan jalur G (I_d/I_g)

bagi komposit ZnSe/GO adalah ~1 dan komposit ZnSe/GNP adalah ~0.3. Jadi, ini dapat memastikan GO dan GNP ada dalam komposit. Ikatan organik bagi GO dan GNP ditentukan dengan teknik FTIR dimana ikatan C=C, C-O, CH_3, C-H and $–COO^-$ ditemui dalam komposit ZnSe/GO dan ZnSe/GNP. Jurang jalur optik ZnSe adalah 2.64 eV. Jurang jalur bertukar ke 2.62 eV dan 3.02 eV apabila GO dan GNP ditembahkan masing-masing. Sampel telah dirangsangkan dengan panjang gelombang 290 nm dan puncak pelepasan PL untuk semua sampel berpusat di ~466 nm. Komposit grafin berasaskan ZnSe telah dikaji untuk aplikasi fotovoltan dan fotonik. Kecekapan suria meningkat daripada 0.19 % (ZnSe tulen) kepada 1.61 % apabila GO ditambah dan 11.88 % apabila GNP ditambah. Tambahan pula, ia telah dibuktikan bahawa komposit ZnSe berasaskan grafin mampu menjana nadi saat-femto ~540 fs dimana ia boleh dijadikan bahan yang menjanjikan aplikasi fotonik seperti teknologi pemesinan mikro dan nano, imej tidak linear dan mikroskopi, teknologi pembedahan mikro dan nano. Modifikasi ZnSe untuk menjadi komposit ZnSe/GO dan ZnSe/GNP akan mengubah sifat-sifat ketumpatan elektron, morfologi dan optik. Kajian ini menyediakan pengetahuan asas untuk menyokong sifat-sifat yang diperlukan untuk aplikasi fotovoltan dan fotonik.

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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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

~	Approximately
AFM	Atomic force microscope
ASE	Amplified spontaneous emission
DSR	Diffuse Reflectance Spectroscopy
EDF	Erbium-doped fibre
E_g	Band gap
FESEM	Field emission scanning electron microscope
FTIR	Fourier transform infrared
GNP	Graphene nanoplatelet
GO	Graphene oxide
hkl	Miller indices
LO	Longitudinal optical
M.D.	Modulation depth
ML	Mode locked
NP	Nanoparticle
OSA	Optical spectrum analyzer
PC	Polarization controller
PEEK	Polyether ether ketone
PL	Photoluminescence
QD	Quantum dot
rpm	Rotation per minute
SA	Saturable absorber
THF	Tetrahydrofurane
TO	Transverse optical
ULTEM	Polyetherimid
UV-Vis	Ultraviolet - visible
WDM	Wavelength division multiplexer
XRD	X-ray Diffration
ZnSe/GNP	Zinc selenide/ graphene nanplatelet
ZnSe/GO	Zinc selenide / graphene oxide

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Nanotechnology and nanoscience research is getting more attention with new technology era. The term "nano" means it is in a very small unit which is one – billionth of a unit. When it is applied into materials, it will drastically change properties such as physical, chemical, mechanical, morphological, optical, electrical and other properties. This is due to the smaller particles with bigger surface area to volume ratio. Thus, it will improve the absorption or even electron transfer of the material when it is in nano-size. When it is applied in semiconductors, it exists in 0D (quantum dots), 1D (quantum wires), 2D (quantum wells) and 3D (nanoparticles) (Grimes et al., 2008). When it exists in different dimension, the carriers in the materials (electrons and holes) will form discrete levels between the conduction and valance bands in the semiconductors (Grimes et al., 2008).

Other than that, the concerns regarding the impact of the current technologies to synthesize semiconductors which will harm the environment are getting more attention as well. Therefore a environmental friendly synhesis technology should be developed for a better future. The synthesis technology should be highlighted on the reduction on energy consumption, manufacturing cost and the reaction time when producing the semiconductors. Worldwide research should be focused on using cleaner or greener method to fabricate semiconductors for future generation. When energy consumption is high, it favors the formation of greenhouse gaseous and air pollutants which lead to air pollution, global warming and climate change (Zhang and Zhao, 2009). Moreover, the total current power produced by the world is 15 TW and it is expected to increase by 50% which is 30 TW by the year of 2050 (Cheng et al., 2009). Therefore, renewable resources to replace those non-renewable resources or minimize the wastage of power has been proposed by Petinrin & Shaaban (2015). In conjuction of this, microwave-assisted synthesize method should be implemented. By using microwave synthesis method, it favors the reaction which improve and increase the yield rapidly as compared to conventional synthesis method (Barnhardt et al., 2006). Hence microwave-assisted synthesize method is widely applied in the organic synthesis nowadays (Biswas et al., 2017; Kuik et al., 2017).

Semiconductor plays an essential role in the modern era. The conductivity of semiconductor is in between conductors and insulator, where this properties made it become fundamental material which used as optoelectronics such as solar panel, light-emitting, windows, switch, amplifiers, modern electronics and others. ZnSe is selected in this research because it is II-VI semiconductor which can be applied in light emitting diodes, solar cell, photodetectors, laser diode and other optoelectronic devices (Kowalik et al., 2008). ZnSe, itself, is a wide band gap materials. The band gap is around 2.70 eV. It can be synthesized via sol-gel process (Wang et al., 2004), precipitate reaction (Andrade et al., 2009), chemical solution method (Song et al., 2015) and hydrothermal method (Peng et al., 2010) and other.

There are lots of research has been done to improve the properties of ZnSe by increasing or decreasing the size of ZnSe or forming metal-doped ZnSe such as copper-doped ZnSe (Panda et al., 2011), nickel-doped ZnSe (Yadav et al., 2015), manganese-doped ZnSe (Zhou et al., 2017), iron-doped ZnSe (Gavrishchuk et al., 2018) and others. However, there is very less reports on mixing ZnSe with carbon-based materials to improve the properties on ZnSe. Thus, it caught the attention to produce cabon-based ZnSe composite in order to further investigate what happened when carbon-based material such as graphene, graphene oxide, carbon nanotubes and others mixed with ZnSe. There are only few methods that had been reported on synthesizing ZnSe or carbon-incorporated ZnSe using microwave-assisted hydrothermal method. This is because microwave-assisted synthesis method is mainly used in organic synthesis rather than inorganic synthesis.

In this work, graphene-based zinc selenide composite is synthesized via microwave-assisted hydrothermal method. The structural, morphological and optical properties of the sample is characterized to study the effects of graphene nanoplatelet and graphene oxide on the properties as compared to ZnSe.

1.2 Problem statement

There are various methods in synthesizing ZnSe nanoparticles as mentioned at the previous section. These methods included sol-gel process (Jiang et al., 2004), sonochemical method (Zhu et al., 2000), hydrothermal method (Cao et al., 2008). However the conventional synthesizing methods are energy and time consuming which lead to higher cost. Therefore, a greener method should be developed and implemented to minimize the energy consumption, synthesizing time and cost effectiveness.

Graphene-based materials are getting attentions as it can be applied as drug deliver materials, supercapacitors and other. However, graphene itself is very conducting which made it hardly to be applied as some of the applications such as photovoltaic. Thus, graphene-based composite had been growing in the latest research. Graphene-based material can formed composite with metal, metal oxide, metal-organic framework and other materials. It was proven that by forming the composites, it will enhance the electrical, optical and mechanical properties of materials. Therefore, this research will synthesized graphene-based ZnSe composite in order to investigate the structural, morphological and optical properties of the composite to understand the impact of this approaches via fundamental investigation.

Thus, this research will tackle the research questions as following:

1. How to mix graphene-based materials with ZnSe using greener method that can drastically reduce the energy consumption, reaction time and cost effectively?
2. What is the optimum condition to synthesize the graphene-based ZnSe composite by implement this greener synthesis method?
3. Will graphene-based material in ZnSe influence the structural, morphology, and optical properties of the composite?
4. What is the potential application of graphene-based ZnSe composite?

1.3 Objectives and hypotheses of the study

Based on the research questions, it is interesting to discover a new and greener environment method which can greatly reduce the energy and time consumption as well as the production cost. Therefore, microwave-assisted hydrothermal method is implemented to synthesize graphene-based ZnSe composite. There are various parameters that need to be tuned in order to obtain the best synthesizing condition. The sample obtained is characterized to study the structural, morphological and optical properties. The sample is also tested for solar cell and photonics applications. The objectives throughout this research are:

1. to synthesize and optimize the synthesis condition of graphene-based ZnSe via microwave-assisted hydrothermal method.
2. to characterize the structural, morphological, and optical properties of graphene-based ZnSe composite.
3. to evaluate the potential application of the synthesized graphene-based ZnSe composite.

By referring to the above objectives, this study is hypothesized as below:

1. Microwave-assisted hydrothermal method will reduce the time and energy consumption and cost effectively. The rapid heating via microwave synthesis increases the net rate of the process. Thus, microwave synthesis generated intense heating with inverse temperature gradient effect where his minimized the problem of inhomogeneties when heating the sample. This lead to the molecules to covered by microwave-absorbing polar solvent which changed he surface temperature and produced a homogenous local overheating. The microwave irradiation will directly transfer to solvent and heat up the solvent evenly and thus the reaction can occur homogenously. In conventional method, the heat is transfer from one medium to another medium. Thus, there is energy loses and the reaction will become slower and require more energy as compare to microwave-assisted hydrothermal method.

2. It is believed that microwave irradiation power, reaction time, concentration of sodium hydroxide, type and volume of surfactants, concentration of graphene oxide and concentration of graphene nanoplatelet will affects the reaction during synthesis process. Thus, optimum condition in syhthesizing graphene-based ZnSe composite should be determined by varying these parameters.
3. Introducing other material such as graphene nanoplatelet and graphene oxide into ZnSe is believed to change the properties of the stand-alone ZnSe by enhancing the carriers in ZnSe.
4. As graphene nanoplatelet and graphene oxide is mixed with ZnSe to form composite, it is believed that introducing graphene nanoplatelet and graphene will influence the performance as solar cell and saturable absorber would be improved or decreased.

1.4 Scope of study

This study will emphasis on the synthesizing graphene-based ZnSe via microwave-asissted hydrothermal method. The parameters such as microwave irradiation power, reaction time, concentration of sodium hydroxide, type and concentration of surfactants, and concentration of graphene nanoplatelet and graphene oxide are studied. The structural, morphological and optical properties of the sample have been studied through X-ray diffraction, Raman spectroscopy, Fourier ransform infrared spectroscopy, field emission scanning electron microscope, atomic force spectroscopy, UV-Visible spectroscopy and photoluminescence spectroscopy.. Finally, the sample was tested on photovoltaic (solar cell) and photonics application by looking at the solar efficiency, open circuit voltage, short circuit current, fill factor that obtained from solar testing and pulse duration for saturable absorber testing.

1.5 Thesis outline

This thesis consisted of six chapters. Chapter one briefly describes the general introduction of zinc selenide and microwave-assisted synthesis method, background of the study, and objectives of the research. In chapter two, it is related with the literature reviews about previous studies on synthesizing ZnSe and carbon-based ZnSe composites. Synthesis of ZnSe via microwave-assised methods will be discussed as well. Chapter three will explain the theory of microwave synthesis method and carrier recombination process. Chapter four focuses on how the samples were prepared. In addition, equipment that used for measurement and characterization have been discussed in this chapter. Chapter five presenting the obtained data and results of current research work, followed by discussion on the result obtained. Finally, chapter six concludes the research findings and future research recommendations.

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