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STRUCTURAL, MORPHOLOGICAL AND OPTICAL PROPERTIES OF Eu3+ DOPED ZnO/Zn2SiO4 FABRICATED VIA THERMAL TREATMENT METHOD

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By

SUHAIL HUZAIFA BIN JAAFAR

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

April 2021

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DEDICATIONS

To my beloved mother, Ummi Kalsom Binti Abdullah for her never-ending support, understanding, and love

To my late father, Jaafar Bin Ismail for all the sweet memories and lessons

To my siblings, family and family to be for their motivational and financial support

To my friends for making my life full of flavors and never dull

To all my lectures for the lessons and supports throughout this journey

I would like to say that this work is a gift for all of us, not only mine

I cannot thank all of you enough but still, thank you.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Master of Science

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Chairman: Mohd Hafiz Bin Mohd Zaid, PhD
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The advancement in the electronic display and optical electronics' technology has made the phosphor field one of the interesting fields to study among researchers. Hence, a lot of new synthesis techniques as well as different starting materials have been used for the fabrication of enhanced phosphor. This research work uses the thermal treatment method to synthesis the Eu^{3+} doped ZnO/Zn_2SiO_4 based composites which undergo calcination process at a temperature between 600 °C, 700 °C, and 800 °C for 2 h, 3 h and 4 h holding times. Different parameters or variables have been studied in this work including the effect of calcination temperature, holding time and Eu^{3+} concentration on the structural, morphological and optical characteristics of ZnO/Zn_2SiO_4 based composites. The XRD analysis shows the existence of two major phases which are ZnO and Zn_2SiO_4 crystals, which are supported by the finding in the FTIR. The FESEM micrograph show that progress in the calcination temperature and holding time, affects to the existence of necking-like shape particle. Absorption humps discovered through UV-Vis spectroscopy reveal that samples at the higher calcination temperature, holding time and Eu^{3+} concentration possess higher absorption intensity. Two types of band gap can be seen from the energy band gap analysis which occurs from ZnO crystal and Zn_2SiO_4 crystal progress. It is also discovered that the Zn_2SiO_4 crystal (5.345 to 4.182 eV) has a higher band gap compared to the ZnO crystal (3.217 to 3.176 eV). While, for the photoluminescence study, the emission spectra show higher calcination holding time exhibit higher emission intensity with 700 °C being the optimum temperature. The emission spectra also show higher concentration of Eu^{3+} helps for enhancing the emission intensity until 5 mol% of Eu^{3+} dopant concentration. The study findings provide a new and simple method for the fabrication of Eu^{3+} doped ZnO/Zn_2SiO_4 based composites for new potential red phosphor application in the optoelectronic fields.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk Ijazah Master Sains

SIFAT STRUKTUR, MORFOLOGI DAN OPTIK ZnO/Zn_2SiO_4 DIDOPKAN Eu^{3+} DIFABRIKASI MELALUI KAEDAH RAWATAN HABA

Oleh

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April 2021

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Kemajuan dalam teknologi paparan elektronik dan elektronik optik telah menjadikan bidang fosfor sebagai salah satu bidang yang menarik untuk dikaji di kalangan penyelidik. Disebabkan itu, banyak teknik sintesis baru dan bahan permulaan yang berbeza telah dicipta dan digunakan untuk pembuatan fosfor yang lebih baik. Kerja penyelidikan ini menggunakan kaedah rawatan haba untuk mensintesis komposit berasaskan ZnO/Zn_2SiO_4 didopkan Eu^{3+} yang menjalani proses kalsinasi pada suhu antara 600 °C, 700 °C dan 800 °C selama 2 jam, 3 jam dan 4 jam masa tahan. Parameter atau pemboleh ubah yang berbeza telah dikaji dalam penyelidikan ini termasuk pengaruh suhu kalsinasi, masa tahan dan jumlah kandungan Eu^{3+} terhadap sifat struktur, morfologi dan optik komposit ZnO/Zn_2SiO_4. Analisis XRD menunjukkan kewujudan dua fasa utama iaitu fasa ZnO dan Zn_2SiO_4, yang disokong oleh penemuan dalam FTIR. Mikrograf FESEM mengesahkan kenaikan suhu kalsinasi dan masa tahan mempengaruhi kewujudan zarah berbentuk leher. Bonggol penyerapan yang terlihat melalui spektroskopi UV-Vis menunjukkan sampel pada suhu kalsinasi, masa tahan dan juga kepekatan dopan yang lebih tinggi memiliki intensiti penyerapan yang lebih tinggi. Dua jenis jurang jalur dapat dilihat daripada analisis jurang jalur tenaga yang terhasil daripada kristal ZnO dan kristal Zn_2SiO_4. Juga didapati bahawa kristal Zn_2SiO_4 (5.345 hingga 4.182 eV) mempunyai jurang jalur yang lebih tinggi berbanding dengan kristal ZnO (3.217 hingga 3.176 eV). Sementara itu, untuk kajian fotoluminesen, spectrum-specktrum pelepasan menunjukkan masa tahan kalsinasi yang lebih tinggi memberikan intensiti pelepasan yang lebih tinggi dengan 700 °C menjadi suhu optimum. Spektrum-spektrum pelepasan juga menunjukkan kepekatan Eu^{3+} yang lebih tinggi sehingga 5 mol% dopan membantu meningkatkan intensiti pelepasan menjadi lebih tinggi. Penemuan kajian memberikan kaedah baru dan mudah untuk pembuatan komposit berasaskan ZnO/Zn_2SiO_4 doped Eu^{3+} untuk aplikasi fosfor merah yang berpotensi dalam bidang optoelektronik.

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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 Master of Science. The members of the Supervisory Committee were as follows:

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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LIST OF ABBREVIATIONS AND SYMBOLS

ZnO	Zinc oxide
Zn_2SiO_4	Zinc silicate
Eu^{3+}	Europium (III) ion
SiO_2	Silicon dioxide
PVP	Polyvinyl pyrrolidone
XRD	X-ray diffraction
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy
UV-Vis	Ultraviolet-visible spectroscopy
PL	Photoluminescence
Mn^{2+}	Manganese (II) ion
Ti	Titanium
ZnSe	Zinc selenide
h	Hour(s)
g	Gram
°C	Degree Celcius
Min(s)	Minute(s)
mmol	Millimol
mL	Milliliter
UV	Ultraviolet
eV	Electronvolt
mol	Mole
SLS	Soda-lime silica

CHAPTER 1

INTRODUCTION

1.1 Introduction

Recently, phosphors have been attracting numerous interests due to the advancement in electronic display and optical technology. Widely used as a light source for the most advanced televisions, X-ray screens, scintillators, display devices and coating applications, phosphors have been considered as the future in lighting technology due to the ability and capability to be used as an alternative for the conventional fluorescent lamps and incandescent light bulb with lower energy consumption (Yanagita & Arimoto, 2013; Nazarov, 2016). It seems the journey to enhance and synthesize phosphors material with better performance shall never end as the demand in the new technology display for optoelectronics devices such as plasma displays is increasing every single day (Tsukatani & Wataya, 2015; Ali et al., 2018).

A lot of research regarding the enhancement of the phosphor materials is being carried including developing and modifying new fabrication methods, much simpler synthesis techniques as well as controlling the production of fine and uniform material (Ali et al., 2018). Among the numerous studies, zinc oxide (ZnO) and zinc silicate (Zn_2SiO_4) based phosphor materials have been widely used in the optoelectronic industry due to its optical ability to become an outstanding host matrix for rare earth and transition metal dopants (Effendy et al., 2016; Khaidir et al., 2020). The ZnO/Zn_2SiO_4 based phosphor materials are also being globally studied because of their physical and chemical stabilities besides being less expensive and high quantum efficiency (Alibe et al., 2018).

Various methods and techniques for the fabrication of ZnO/Zn_2SiO_4 based phosphor materials have been conducted by many researchers. Generally, ZnO/Zn_2SiO_4 is synthesized using solid-state reaction method by applying a conventional calcination process over a long period at a specified temperature, usually high to a combination of ZnO and silicon dioxide (SiO_2) powder (Yoshizawa et al., 2012). Depending on the fabrication technique and starting materials used, the characteristics of the synthesized product can be different and certainly, the type of silica precursors used may influence the final properties of the fabricated silicate-based phosphor.

Furthermore, the sol-gel method, hydrothermal method, solvothermal method, supercritical water method and vapor method are different methods reported for synthesizing ZnO/Zn_2SiO_4 based phosphor materials (Takesue et al., 2009). However, most of the methods are difficult to be employed due to high production costs with complex fabrication steps and usage of high reaction temperatures which contribute to high energy consumption (Lee et al., 2015; Baqer et al.,

2018). Hence, a simple method called thermal treatment method was applied to fabricate ZnO/Zn_2SiO_4 based composites due to the material handling simplicity, low energy consumption, and environmentally friendly (Salem et al., 2017; Alibe et al., 2018).

Recently, thermal treatment method has been utilized to fabricate metal oxide particles and metal ferrite particles (Al-Hada et al., 2019). By dissolving zinc nitrate, the metal precursor and polyvinyl pyrrolidone (PVP) as the binding agent in deionized water, pure ZnO particles was fabricated after the drying, grinding and calcination process (Al-Hada et al., 2014; Lee et al., 2015). While, Naseri et al. (2011) presented the fabrication of nickel ferrite particles by utilizing iron nitrate and nickel nitrate as metal nitrate reagents, added with poly(vinyl alcohol) (PVA) as the capping agent and deionized water as the solvent. The fabrication of copper ferrite particles was also successfully synthesized by changing copper nitrate as the metal nitrate reagent, with the addition of iron nitrate, PVA, and deionized water, (Naseri et al., 2013). Likewise, zinc selenide had also fabricated using the thermal treatment method by utilizing zinc nitrate ($Zn(NO_3)_2$), selenium powder, and PVP while deionized water and ethylenediamine were used as the solvents (Salem et al., 2017).

In this study, zinc acetate ($Zn(CH_3CO_2)_2$) and silicon tetraacetate ($Si(OOCCH_3)_4$) is used as a starting material, followed by PVP as a capping agent, europium acetate ($Eu(CH_3CO_2)_3$) as a dopant and deionized water as the solvent to fabricate and synthesize Eu^{3+} doped ZnO/Zn_2SiO_4 based composites. To analyze, measure and study the structural, morphological and optical characteristics of the fabricated composite, XRD analysis, FTIR analysis, FESEM, UV-Vis spectroscopy, optical band gap analysis and photoluminescence analysis has been conducted. The final product may have several potential applications including phosphor in LEDs, lamps, optical fiber, and many more.

1.2 Problem Statement

ZnO/Zn_2SiO_4 can be synthesized by miscellaneous methods such as the famous conventional solid-state reaction method (Ramakrishna et al., 2014; Essalah et al., 2020) and sol-gel method (Elhadi et al., 2019). Besides that, hydrothermal method (Qiao et al., 2018), co-precipitation method (Ntwaeaborwa et al, 2017), sonochemical method (Masjedi-Arani and Salavati-Niasari, 2016), spray pyrolysis method (Lee et al., 2011), super-critical water method (Toyama et al., 2015) and solvothermal method (Nagornov et al., 2020) were also being utilized for the production of ZnO/Zn_2SiO_4 (Omar et al., 2016). However, due to the complicated procedures, the requirement of high reaction temperature and longer time of reaction affects those methods to facing difficulty in large scale production. Thus, in this study, a much simpler technique called thermal treatment method was proposed to tackle some of the undesired problems during fabrication such as high energy consumption, complicated procedures and long reaction time (Salem et al., 2017; Alibe et al., 2018;).

Besides, the luminescence properties of the existing phosphor materials in the industries are quite low and require high energy for the display, especially for red phosphor application. Dash et al. (2019) reported that from the photoluminescence analysis conducted, the obtained luminescence intensity of pure ZnO fabricated via the soft-solution method is low. Effendy et al. (2019) reported that the fabricated Zn_2SiO_4 using the melt and quench method possessed low luminescence intensity when there are no rare-earth ions or transition metals involved in the system. Furthermore, Ramakrishna et al. (2014) revealed that the fabricated ZnO/Zn_2SiO_4 with no dopant emitted the lowest luminescence intensity compared to the doped one. However, previous studies reported the usage of Eu^{3+} as dopant for red phosphor to enhance the photoluminescence properties of the material. Introduction of Eu^{3+} in ZnO and Zn_2SiO_4 had affected the materials to possess much higher luminescence spectra in red region (Armelao et al., 2008; Omar et al., 2016). Hence, in this study, Eu^{3+} was deemed to be a suitable dopant for the ZnO/Zn_2SiO_4 system to obtain red phosphor with enhanced luminescence trait.

1.3 Objectives of the study

The objectives of this study are:

1. To fabricate and synthesize $ZnO/Zn_2SiO_4:Eu^{3+}$ based composites using thermal treatment method.
2. To investigate the effect of different mol percentage of Eu^{3+} content on the structural, morphological and optical properties of $ZnO/Zn_2SiO_4:Eu^{3+}$ based composites.
3. To study the effect of different calcination temperatures and holding time on the structural, morphological as well as optical properties of $ZnO/Zn_2SiO_4:Eu^{3+}$ based composites.

1.4 Scope of the study

The scopes of the study are:

1. The mol ratio between zinc acetate and silicon tetraacetate which is 1:1 and the percentage of europium (III) acetate added which are 0 mol%, 1 mol%, 3 mol%, 5 mol%, and 10 mol%.
2. The calcination temperatures used which are 600 °C, 700 °C, and 800 °C with holding time of 2 h, 3 h and 4 h.
3. The properties of the synthesized ZnO/Zn_2SiO_4 studied are structural, morphological, and optical properties.

1.5 Significance of the study

Recently, Zn_2SiO_4 based phosphor material has received quite a high amount of attention as a candidate for optoelectronic application to replace the historical use of the green display panel in the cathode ray tube. Subsequently, the growing number of applications has increased the demand for better performance phosphor. The requirements of current devices to be mobile, compact, and high-quality display are from device developers. Despite the rapid development in device technology, the element of green technology, cost-effectiveness, and simplicity in the synthesizing processes are also taken into consideration. Under these circumstances, the fabrication method and starting materials for this study were specifically chosen to study those factors. This study was focused on synthesizing $ZnO/Zn_2SiO_4:Eu^{3+}$ composites using the thermal treatment method. This method applied zinc acetate as the zinc source, silicon tetraacetate as silica source, and PVP as capping or binding agent into the deionized water with the addition of europium acetate, acting as the dopant. It was expected that the properties of $ZnO/Zn_2SiO_4:Eu^{3+}$ based composites, fabricated using the thermal treatment method and could contribute as new knowledge to synthesize phosphor materials.

1.6 Thesis outline

In this thesis, the main feature of evaluation is the study of structural, morphological and optical properties of $ZnO/Zn_2SiO_4:Eu^{3+}$ fabricated via thermal treatment method. Summary of usage of phosphor and fabrication methods added with the problem statement, objectives, scope and significance of the study were stated in Chapter 1. A brief discussion of phosphor materials, ZnO, Zn_2SiO_4, ZnO/Zn_2SiO_4 composites, europium and established method for preparation were discussed in Chapter 2. While in Chapter 3, a detail clarification of methodology and procedures involved in fabricating $ZnO/Zn_2SiO_4:Eu^{3+}$ using thermal treatment method were discussed. The result and discussion for properties' study such as XRD, FTIR, FESEM, UV-Vis, calculated band gap and PL were explained in the Chapter 4. Lastly, the conclusions of this research study in addition to the recommendations for future research were given in Chapter 5. List of references, biodata of student as well as list of publications were covered in the last part of the thesis.

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