UNIVERSITI PUTRA MALAYSIA

SYNTHESIS, STRUCTURAL AND OPTICAL PROPERTIES OF ZNO-SIO_2 COMPOSITE DERIVED FROM WASTE COCONUT HUSK

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By

MUHAMMAD FAHMI BIN ANUAR

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

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SYNTHESIS, STRUCTURAL AND OPTICAL PROPERTIES OF ZnO-SiO_2 COMPOSITE DERIVED FROM WASTE COCONUT HUSK

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Chair : Yap Wing Fen, PhD
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The optical properties of ZnO-SiO_2 composite had attracted a lot of attention this past decade. However, the production cost is quite high due to huge amount of high purity silica required in the synthesis process. Thus, alternative method was required especially using waste materials for example coconut husk. In this research, low cost ZnO-SiO_2 composite were prepared by solid state reaction method using agricultural waste materials: coconut husk. The coconut husk was subjected to high temperature 500, 600, and 700 ºC to obtain coconut husk ash (CHA). CHA was then undergone chemical treatment (acid and alkali) as an attempt to increase its silica content. Thermogravimetry analysis (TGA) was carried out where it shows that most of the weight degradation of coconut husk during pyrolysis occurs at 221 ºC to 360 ºC. From X-ray fluorescence (XRF) analysis, the silica content increased from 8-11 % to more than 90 % for both acid and alkali treatment Moreover, the intensity of X-ray diffraction (XRD) peaks increased along the increased in temperature between 500 ºC to 700 ºC. The Field emission scanning electron microscopy (FESEM) images was analysed and showed rod-like structure of the CHA that was irregular in sizes and become slightly smaller after acid and alkali treatment. Besides that, broad bands were found by Fourier Transform Infrared spectroscopy (FTIR) at wavenumber 1066 cm^{-1}, 788 cm^{-1} and 432 cm^{-1} which related to Si-O-Si thus confirmed the presence of silica in CHA. The optical band gap of CHA untreated, acid treated, and alkali treated were 4.05 eV, 4.34 eV and 4.21 eV respectively. Next, ZnO-SiO_2 composite were prepared by mixing zinc oxide (ZnO) powder with silica from CHA with 1:1 ratio by solid state method sintered at 600 ºC-1000 ºC to investigate the effect of sintering temperature on the properties of ZnO-SiO_2 composite. First, XRD spectrum showed the zinc silicate was formed at sintering temperature 600

^{o}C and the peaks intensity increased along the sintering temperature. The composite shows well-distinct boundaries of rhombohedral-like particle when sintered at 1000 ºC that indicates the high crystallinity of the zinc silicate when sintered. Furthermore, the FTIR results shows the formation of Si-O-Si, ZnO_4 and SiO_4 vibration in the composite spectrum becoming narrow and stronger as the sintering temperature increased. The absorbance intensity of the ZnO-SiO_2 composite was observed to decrease with the sintering temperature and the optical band gap of the composite at room temperature is 3.22 eV. The value increased to 3.26 eV at 600 ºC and decreasing until 900 ºC before rapidly increased to 4.05 eV at 1000 ºC. In the nutshell, the CHA has great potential to be used as silica substitute in preparation of ZnO-SiO_2 composite.

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

SINTESIS, SIFAT STRUKTUR DAN OPTIKAL ZnO-SiO_2 KOMPOSIT DIHASILKAN DARIPADA BAHAN TERBUANG SABUT KELAPA

Oleh

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Ciri-ciri optik ZnO-SiO_2 komposit telah menarik banyak perhatian pada dekad ini. Namun, kos penghasilannya adalah tinggi disebabkan oleh proses sintesis yang memerlukan penggunaan silika ketulenan tinggi dalam jumlah yang besar. Oleh itu, kaedah yang baru diperlukan terutamanya menggunakan bahan sisa seperti sabut kepala. Dalam kajian ini, komposit ZnO-SiO_2 kos rendah disediakan melalui teknik tindak balas keadaan pepejal dengan menggunakan bahan sisa pertanian iaitu sabut kepala. Sabut kelapa telah dikenakan haba pada suhu 500, 600, dan 700 ºC untuk menghasilkan abu sabut kelapa (ASK). ASK kemudiannya telah dirawat (asid dan alkali) sebagai percubaan untuk meningkatkan kandungan silika. Analisis termogravimetri (TGA) telah dilakukan dan menunjukkan hampir keseluruhan penurunan berat bahan sabut kelapa semasa proses pirolisis berlaku pada suhu 221 ºC sehingga 360 ºC. Menurut analisis pendarflour sinar-x (XRF), kandungan silika meningkat dari 8-11 % menjadi lebih 90 % untuk kedua-dua rawatan asid dan alkali. Keamatan puncak belauan sinar-x (XRD) meningkat mengikuti peningkatan suhu dalam julat 500 ºC sehingga 700 ºC. Mikroskopi pengimbasan elektron pelepasan medan (FESEM) analisis menunjukkan ASK mempunyai bentuk seperti rod dan tidak teratur serta menjadi kecil selepas rawatan asid dan alkali. Selain itu, jurang besar yang ditemui pada spektrum spektroskopi inframerah transformasi Fourier (FTIR) pada 1066 cm^{-1}, 788 cm^{-1}, and 432 cm^{-1} mendedahkan kehadiran silika didalam ASK. Jurang jalur optikal ASK telah diukur 4.05 eV, 4.34 dan 4.21 eV mewakili ASK yang tidak dirawat, melalui rawatan asid, dan rawatan alkali. Kemudian, komposit ZnO-SiO_2 disediakan dengan mencampur serbuk zink oksida (ZnO) dan silika dari ASK berjulat 1:1 menggunakan teknik keadaan pepejal dan kemudian di sinter pada suhu 600-1000 ºC untuk mengenal pasti kesan suhu terhadap ciri-ciri komposit ZnO-SiO_2. Pertama, spectra XRD menunjukkan zink silikat terbentuk pada suhu sinter 600 ºC dan keamatan puncak belauan meningkat mengikut suhu. Komposit juga menunjukkan

sempadan yang jelas seperti zarah berbentuk rombohedron apabila suhu sinter meningkat kepada 1000 ºC yang mengesahkan sifat kristal yang tinggi pada zink silikat. Seterusnya, analisis FTIR menunjukkan kehadiran formasi getaran Si-O-Si, ZnO_4 dan SiO_4 dan menjadi kecil dan kuat apabila suhu meningkat. Keamatan penyerapan komposit ZnO-SiO_2 dilihat menurun apabila suhu sinter meningkat dan jurang jalur optikal komposit pada suhu bilik ialah 3.22 eV. Nilai jurang jalur optikal meningkat kepada 3.26 eV pada suhu 600 ºC dan berkurangan apabila suhu meningkat sehingga 900 ^oC tetapi meningkat secara mendadak menjadi 4.05 eV pada suhu 1000 ºC. Secara ringkasnya, ASK mempunyai potensi yang besar untuk digunakan sebagai pengganti silika dalam proses sintesis ZnO-SiO_2 komposit.

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I certify that a Thesis Examination Committee has met on 11 June 2020 to conduct the final examination of Muhammmad Fahmi bin Anuar on his thesis entitled "Synthesis, Structural and Optical Properties of ZnO-SiO_2 Composite Derived from Waste Coconut Husk" 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.

Members of the Thesis Examination Committee were as follows:

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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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I hereby confirm that:

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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Name of Member of Supervisory Committee: Dr. Mohd Hafiz bin Mohd Zaid

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LIST OF ABBREVIATIONS

ASCII	American Standard Code for Information Interchange
ASK	Abu Sabut Kelapa
CHA	Coconut Husk Ash
CS	Coconut Shell
CSA	Coconut Shell Ash
EDX	Energy Dispersive X-Ray
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared
GFN	Grain Fineness Number
HCL	Hydrochoric Acid
HF	Hydrofluoric acid
JCPDS	Joint Committee on Powder Diffraction Standards
KOH	Potassium Oxide
LDD1	Microbial community
NaOH	Sodium Hyroxide
NBO	Non-bridging Oxygen
PEG	Polyethylene Glycol
PL	Photoluminescence
PVA	Polyvinyl Alcohol
SEM	Scanning Electron Microscopy
SiC	Silicon Carbide
SiO_2	Silicon Dioxide
SLS	Soda Lime Silicate
TEM	Transmission Electron Microscope
TEOS	Tetraethyl Orthosilicate
TGA/DTG	Thermogravimetry analysis/Derivative Thermogravimetry
UV-Vis	Ultraviolet- Visible Light
WRHA	White Rice Husk Ash
wt. %	Weight Percentage
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
Zn_2SiO_4	Zinc Silicate
ZnO	Zinc Oxide

CHAPTER 1

INTRODUCTION

1.1 Silica

Silica (SiO_2) is one of the valuable raw materials on Earth and white color is preferred for silica in the optoelectronic application. It can exist in different state such as gel, crystalline and amorphous state (Todkar *et al.*, 2016). Silica can be prepared by using vapour-phase reaction, sol-gel, and thermo-decomposition methods (Bogush *et al.*, 1988; Grandgirard *et al.*, 2002; Tomozawa *et al.*; 2001; Wu *et al.*, 2000). Silica is commonly been used as a precursor for variety of materials in synthetic chemistry application such as catalysts, thin film or coating for electronic and optical materials. It is also used in other fields such as ceramics, chromatography, anticorrosion agent and in catalysts. Uses of high purity silica in the industrial application are highly cost due to the high melting point of 1700 ºC. Thus, the extraction of silica from agricultural waste is one way of researchers nowadays trying to reduce the cost of using high purity silica.

Nowadays, numerous researches were carried out to utilize waste products as a renewable source of silica and use them as substitute materials to reduce the cost. In addition, SiO_2 can be found on agriculture waste such as rice husk, sugarcane bagasse, palm ash, and coconut husk ash (Sinyoung *et al*., 2017). Although the production of SiO_2 from agriculture waste products was not much efficient yet compared to the commercialized silica, it still can be used as a material in studies or research purposes. The use of renewable resources for synthesizing high-value materials is becoming very important for eco-friend nanoscience and technology (Sankar *et al.*, 2018). Waste products-derived silica is economic in terms of money and energy and can help greatly in mitigating environmental issues and at the same time help to increase the interests of many researchers to get involved in the green synthesis method.

1.2 Silica from agriculture waste

Agricultural industry is one of the most important industries in the world especially to a tropical country such as Malaysia. In Malaysia, the major crops are palm oil and rubber, followed by paddy and cocoa production. There are also few numbers of crops grown for domestic purposes such as bananas, coconuts, durians, and pineapples. Major agricultural products can be broadly categorized into foods, fibers, fuel, and raw materials. There are huge numbers of agricultural products such as cotton, wool, dye, resin, drugs, perfume, biofuels, cosmetic products, and silk (Ahmad *et al*., 2007; Baqueiro-Peña and Guerrero-Beltrán, 2017; Ho *et al*., 2005; Nurjanah *et al*., 2016). The process to produce these

products will also produce the byproduct that also known as agricultural waste. These types of waste eventually will be discarded in various ways by the industries.

One of the most popular agricultural products in Malaysia is coconuts. Coconut (Cocos Nucifera) are popular for its versatility and often use in traditional ways, as consumable products (foods) and even used in cosmetic products. In some Africa's countries, the coconut waste was burnt as fuel and become the source of energy-biofuel when the cost of fuel oil, natural gas, and electricity increased (Madakson *et al.*, 2012; Vadery *et al.*, 2014). Today, researchers give more focus on using coconut husk as activated carbon which has potential as good absorbent materials that can solve the wastewater treatment problems in the world (Samanta *et al*., 2018; Tan *et al.*, 2008). At the same time, numerous researchers have been carried out to extract silica from agriculture waste such as rice husk, palm waste, corn cob ash and sugarcane bagasse (Khaidir *et al*., 2020; Faizul *et al.*, 2013; Okoronkwo *et al*., 2016; Norsuraya *et al.*, 2016).

It is assumed that coconut husk also contains silica and has great potential as an alternative silica source. A large portion of 33–35% of the coconut is husk and it acts as the mesocarp of the coconut fruit (Rodiah *et al.*, 2018). To date, coconut husks are used as a source of fuels for coconut processing, domestic fuel, and as fiber sources for the production of ropes and mats.

For example, rice husk contains huge amount of carbon and silica at pre-combustion process (Stochero *et al*., 2017) but white rice husk ash (WRHA) can be obtained after burning rice husks at certain temperatures. It is white and contains about 90% SiO_2 and other components such as lignin, cellulose, hemicellulose, and metallic oxides. Using rice husk is relatively cheap rather than using conventional silica (Leenakul *et al.*, 2016). Therefore, coconut husk is expected to have the same properties and advantages as rice husk.

Furthermore, silica can be extracted from agricultural waste by acid leaching and mixing alkaline treatment followed by precipitation with acid. In general, the properties and structure of silica from agriculture waste are sensitive to the method used to synthesize the silica (Shen, 2017). These different occasions utilization of agricultural waste products so far have proved to be economically viable or commercially feasible. So far, there are lacks information on using coconut husk as a potential source of silica. Hence, this study explores the properties of silica extracted from the coconut husk as new potential alternative sources for optical applications.

1.3 $ZnO\text{-}SiO_2$ composite

Recently, researchers have shown increasing interest in wide band gap semiconductor zinc oxide (ZnO). Generally, ZnO can emit three luminescence bands in UV, green, and yellow spectral range (He *et al.*, 2003). ZnO based materials also caught the attention of researchers because of its non-toxicity, environment-friendly and low-cost materials that causing it to be highly valued by industry and researcher.

Various researches have shown that composites of ZnO and SiO_2 show promising results for electroluminescent application in several aspects such as flat-panel displays, photoelectronic integrated devices, and semiconductor lasers. Besides that, green-yellow emissions were also enhanced in ZnO-based nanocomposites and it is known that its promising features can be modified by doping with different types of metals (Meyer and Damonte, 2015). Furthermore, the optical properties of ZnO/SiO_2 can also be influenced by modifying the formation of Zn-O-Si cross-linking bonds between ZnO and the pore wall of SiO_2 (Fu *et al.*, 2003).

Many researchers used $ZnO\text{-}SiO_2$ to synthesize a glass-ceramics component of zinc silicate. Zinc silicate was investigated because of its unique luminescence properties and was used as a phosphors materials for fluorescence or neon lamp and several more displays application back in the days. Nowadays, it is being used in various technologies including laser, sensors, and more optoelectronic devices (Takesue *et al.*, 2009).

1.4 Problem Statements

As of today, numerous studies had been conducted on zinc oxide composite especially to studies about the optical properties. The growing interest towards the $ZnO\text{-}SiO_2$ composites is due to the optical properties of the materials as it shows great potential to be used in various optoelectronic applications. Even though it is a very simple material, the production of $ZnO\text{-}SiO_2$ composites is expensive due to the huge amount of SiO_2 required and the high cost of pure SiO_2 powder. To fix this problem, alternative ways are required to potentially reduce the overall cost of production. Thus, this study is to provide alternative ways to synthesize $ZnO\text{-}SiO_2$ composites derived from waste materials.

Several past studies have proven that silica can be extracted from various waste materials. Among them, there are only a few published analyses related to coconut as source of silica. Generally, they are consumed as food, drink, or other types of products. Around 90 % of coconut (empty fruit bunch, fibers, fronds, trunks, and shells) were discarded as a waste product and either burned in open air or left to settle in waste pond that causing damage to the land and

environmental pollution (Bamgboye *et al.*, 2006). This action contributes significantly to the emission of CO_2 and methane gas to the environment (greenhouse effects). Without proper treatment or disposal methods, these waste materials especially the husk will result in various environmental problems that can lead to a multitude of health issues. Further researches are needed to be carried out to utilize these waste products and prevent from further harming the Earth. Therefore, coconut husks were used as a source of silica in this study for synthesis of ZnO-SiO_2 composite.

Acid and alkali treatments are generally used to extract silica from waste agricultural ash. These different methods so far have proved to be economically and commercially feasible. However, there is lack of information especially about the comparison between the properties of silica obtained by using both methods. Hence, this study helps to explore the differences between the properties of silica obtained by acid and alkali treatment from waste coconut husks.

Lately, many studies have been conducted to synthesized Zn_2SiO_4. A few methods had been developed such as hydrothermal process (Gabás *et al.*, (2009), J. Yang *et al.*, (2011); An *et al.* (2010); Lin and Hsi, (1995), sol-gel method (Rasdi *et al*., 2017), solid state method (Omar *et al.*, 2016) and spray pyrolysis method (Nath *et al.*, 2013). Instead of using other methods to synthesize $ZnSiO_4$, the method by using solid state reaction is much simpler and can be reproduced in large quantities compared to the chemical methods. The other disadvantages of using the chemical method are the high equipment cost, long preparation periods and very complicated steps while some of the chemicals itself considered harmful substances to the environment.

Materials for multilayer and multi-color displays required high optical transmittance in the visible wavelength regions. So, the optical studies of the composite specifically the optical band gap, are essential for the potential production of phosphors materials. However, due to the lack of researches, the optical knowledge of the composite was still lacking. Thus, the optical properties of the composite derived from waste coconut husks were studied for potential phosphors materials. In this study, ZnO-SiO_2 composite was synthesized from waste coconut husk by solid state reaction method to investigate their structural and optical properties for potential phosphors materials.

1.5 Objectives of Study

The objectives of this research are as followed:

1) To examine the properties of the coconut husk ash derived from the pyrolysis of coconut husk waste at different temperatures.
2) To study the effect of acid and alkaline treatment on the purity of silica.
3) To investigate the structural and optical properties of the ZnO-SiO_2 composite sintered at various temperatures.

1.6 Scopes of Study

Scopes of studies are as stated:

1) Coconut husk was turned into coconut husk ash via pyrolysis technique at temperatures of 500, 600 and 700 °C.
2) Acid and alkaline treatment were used to extract high purity SiO_2 from coconut husk ash.
3) ZnO-SiO_2 composite was synthesized by adding ZnO powder with coconut husk ash by using solid state method.
4) ZnO-SiO_2 composite were sintered at temperature range from 600-1000 ºC.
5) The structural properties of the CHA and ZnO-SiO_2 composite derived from waste coconut ash were characterized by using TGA/DTG, XRF, XRD, FESEM, and FTIR.
6) The optical properties of the CHA and ZnO-SiO_2 composite derived from waste coconut husk were characterized by using UV-Vis spectroscopy.

1.7 Thesis Outline

Chapter 1, introduces the content of the thesis including the objectives and problem statements. The previous and current research which are similar or related to the studies were reviewed on Chapter 2. Chapter 3 contained detailed explanation of the methodology of the studies including materials and apparatus, flow chart of the experiment, and characterization methods used to determine the structural and properties of ZnO-SiO_2 composite. Results and finding were elaborately explained in chapter 4. The structural and optical properties of ZnO-SiO_2 composite derived from waste coconut husk will also be discussed in this chapter. Finally, last chapter of the thesis will be the conclusion of the study and recommendation for the future work.

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BIODATA OF STUDENT

The student was born on 17th December 1993 on Kuala Kangsar, Perak. He attended primary school at Sekolah Kebangsaan Seri Tasik and secondary school at Sekolah Menengah Kebangsaan Seri Tasik, Kuala Lumpur. He went into Sekolah Menengah Sains Seremban (SASER) on 2008 after being offered to study there. In 2011, he continued his studies in Pusat Asasi Sains Universiti Malaya (PASUM). After that, he furthered his studies in Bachelor degree in Materials Science Universiti Putra Malaysia and successfully graduated in year 2016.

LIST OF PUBLICATIONS

Muhammad Fahmi Anuar, Yap Wing Fen, Mohd Hafiz Mohd Zaid, Khamirul Amin Matori, and Rahayu Emilia Mohamed Khaidir. "Synthesis and structural properties of coconut husk as potential silica source." *Results in Physics* 11 (2018): 1-4

Muhammad Fahmi Anuar, Yap Wing Fen, Mohd Hafiz Mohd Zaid, Khamirul Amin Matori, and Rahayu Emilia Mohamed Khaidir. "The physical and optical studies of crystalline silica derived from the green synthesis of coconut husk ash." *Applied Sciences* 10 (2020): 1-11.

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Khaidir, Rahayu Emilia Mohamed, Yap Wing Fen, Mohd Hafiz Mohd Zaid, Khamirul Amin Matori, Nur Alia Sheh Omar, Muhammad Fahmi Anuar, Siti Aisyah Abdul Wahab, and Aisyah Zakiah Khirel Azman. "Exploring Eu^{3+}-doped ZnO-SiO_2 glass derived by recycling renewable source of waste rice husk for white-LEDs application." *Results in Physics* 15 (2019): 102596.

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Conferences

Oral presenter at 2nd International Symposium on Advanced Materials and Nanotechnology (i-SAMN), Malaysia held from 15-16th August 2018, Everly Hotel Putrajaya.

Participant in "Projek Penyelidikan Inovasi Nanoteknologi 2018 (PIN 18') on 8-11th October 2018 at Technology Park Malaysia, Bukit Jalil. Kuala Lumpur.

Poster presenter at 10th International Fundamental Science Congress on 23-24 October 2019 at RHR Hotel UNITEN, Kajang Selangor.

Oral and poster presenter at Materials Technology Challenges 2019 (MTC 2019) on 27th March 2019 at Dewan Sri Harmoni, 5th college, UPM Serdang Selangor.

Competitions

Silver medal award at Materials Technology Challenges 2019 (MTC 2019) on 27th March 2019 at Dewan Sri Harmoni, 5th college, UPM Serdang Selangor.

Best poster award at Materials Technology Challenges 2019 (MTC 2019) on 27th March 2019 at Dewan Sri Harmoni, 5th college, UPM Serdang Selangor.

Silver medal award and participants of Perlis International Engineering Invention & Innovation Exhibition (PI-ENVEX 2020). 20-22 March 2020. UniMAP.

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