UNIVERSITI PUTRA MALAYSIA

PHYSICAL, STRUCTURAL AND OPTICAL PROPERTIES OF WILLEMITE DERIVED FROM WHITE RICE HUSK ASH DOPED WITH MANGANESE DIOXIDE

ALI JABBAR ABED ALNIDAWI

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By

ALI JABBAR ABED ALNIDAWI

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

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DEDICATION

- To my wife for all his contribution, patience, and understanding throughout my master's studies. She supported me a lot, made it all possible for me and cheering up, and stood with me at the good times and bad.

- To my kids, who accompanied me through the different parts of my study. Their love has always been my greatest inspiration.

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

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Chairman : Associate Professor Khamirul Amin bin Matori, PhD
Faculty : Science

Over the last few years, the manganese doped willemite (Zn_2SiO_4) draw much attention in the solid state lasers, phosphors, lighting and optical devices. Intensive research has been carried out to use pure SiO_2 and not much work had been done on used waste solid materials for synthesis $Zn_2SiO_4{:}Mn^{2+}$ and little of them used SiO_2 derived from white rice husk ash. The purpose of the current study is to produce the willemite derived from white rice husk ash and enhancement of their properties by MnO_2 doping, which expected to reduce the cost of electroluminescence devices. Thus, in this study, willemite glass-ceramic and MnO_2 doped willemite were synthesised from white rice husk ash as a source of silicon, ZnO and MnO_2 using the conventional solid-state method. The current study involves an investigation of the effect of sintering temperature and Mn-dopant concentration on the physical, structure and optical properties of willemite.

This study indicates that the average density and linear shrinkage of MnO_2-willemite increased with increasing of concentration of MnO_2 dopant and sintering temperature. The result of x-ray diffraction revealed that the amorphous phase at un-sintered, 500, 600, and 700 °C respectively, metastable phase β-Zn_2SiO_4 at 800 °C changed to stable phase α-Zn_2SiO_4 at 900 and 1000 °C. The FESEM micrographs of $Zn_2SiO_4{:}Mn^{2+}$ showed at lower sintering temperature 500, 600 and 700 °C the morphology showed that the grains are still in irregular shape and aggregated. After 800 °C the microstructure of sample started to change which is the sharper edge of the glass microstructure start to decrease cause of the softening effect. Therefore, at 900 and 1000 °C the morphology of the willemite glass-ceramic became granular due to product the crystal in the sample. The FTIR result suggested that the formation of willemite crystal phase based on the

presence SiO_2 and ZnO_4 bands. The result from UV-VIS spectroscopy indicate that the optical band gap for glass and glass-ceramic sample decreased as the concentration of dopant and sintering temperature increased. The emission intensity of all Zn_2SiO_4 sample exhibited a strong green luminescence at 530 nm which due to transition of unshielded $3d^5$ electrons of Mn ion from the first excited state of $^4T_1(^4G)$ to the ground state of $^6A_1(^6S)$. The emission intensity of glass and glass-ceramic sample increased with the increased of the MnO_2 concentration and the highest intensity of it at 5 wt.% doping.

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

CIRI-CIRI FIZIKAL, STRUKTUR DAN OPTIK WILLEMITE DIHASILKAN DARI ABU PUTIH SEKAM PADI DIDOPKAN DENGAN DIOKSIDA MANGAN

Oleh

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Sejak beberapa tahun yang lalu, willemite (Zn_2SiO_4) yang didopkan dengan mangan telah menarik banyak perhatian dalam bidang keadaan pepejal laser, fosfor, lampu dan alat-alat optik. Penyelidikan yang intensif telah dijalankan dengan menggunakan SiO_2 yang tulen dan tidak banyak penyelididkan yang telah dilakukan untuk sintesis $Zn_2SiO_4:Mn^{2+}$ dengan menggunakan bahan sisa pepejal dan sedikit daripada mereka yang menggunakan SiO_2 yang berasal dari abu putih sekam padi. Tujuan kajian ini adalah untuk menghasilkan willemite daripada abu putih sekam padi dan meningkatkan sifat-sifat mereka dengan pendopan Mn, yang dijangkakan akan mengurangkan kos penghasilan alat-alat elektroluminesen. Oleh itu, dalam kajian ini, seramik kaca willemite dan seramik kaca willemite yang didopkan dengan MnO_2 telah disintesis dari abu putih sekam padi sebagai sumber silikon, ZnO dan MnO_2 menggunakan kaedah keadaan pepejal yang konvensional. Kajian ini melibatkan penyelidikan kesan suhu pensinteran dan kepekatan Mn-dopan pada fizikal, struktur dan sifat optik willemite.

Kajian ini menunjukkan bahawa purata ketumpatan dan linear pengecutan MnO_2-willemite meningkat dengan peningkatan kepekatan pendopan MnO_2 dan suhu pensinteran. Hasil pembelauan x-ray menunjukkan bahawa fasa amorfus masing-masing bagi sebelum sinter, dan disinter pada 500, 600, dan 700 °C, fasa metastabil β-Zn_2SiO_4 pada 800 °C bertukar kepada fasa stabil α-Zn_2SiO_4 pada 900 dan 1000 °C. Mikrograf FESEM daripada willemite menunjukkan pada suhu pensinteran lebih rendah iaitu 500, 600 dan 700 °C morfologi menunjukkan bahawa butiran masih dalam keadaan yang tidak teratur dan agregat. Selepas 800 °C mikrostruktur sampel mula berubah iaitu ketajaman hujung butiran semakin mengurang disebabkan oleh kesan pelembutan. Oleh itu, pada 900 dan 1000 °C morfologi seramik kaca willemite menjadi berbutir disebabkan oleh peroses pengkristalan pada sampel. Hasil FTIR mencadangkan pembentukan fasa kristal willemite dari kewujudan jalur-jalur SiO_2,

ZnO_4 dan Zn-O-Si. Hasil dari UV-VIS spektroskopi pula menunjukkan bahawa sela jalur optik untuk sampel-sampel kaca dan seramik kaca menurun dengan peningkatan kepekatan pendopan dan peningkatan suhu pensinteran. Keamatan pelepasan untuk semua sampel Zn_2SiO_4 mempamerkan luminescence hijau yang kuat pada 530 nm yang disebabkan oleh peralihan elektron tanpa lindung $3d^5$ ion Mn daripada keadaan teruja pertama $^4T_1(^4G)$ untuk keadaan asas $^6A_1(^6S)$. Keamatan pelepasan daripada sampel-sampel kaca dan seramik kaca meningkat dengan peningkatan kepekatan MnO_2 dan keamatan tertinggi itu pada 5 wt.%.

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I certify that a Thesis Examination Committee has met on 25 May 2017 to conduct the final examination of Ali Jabbar Abed Al-Nidawi on his thesis entitled "Physical, Structural and Optical Properties of Willemite Derived from White Rice Husk Ash Doped with Manganese Dioxide" 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.

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

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LIST OF ABBREVIATIONS

Zn_2SiO_4	Willemite
$Zn_2SiO_4:Mn^{2+}$	Willemite doped manganese
RH	Rice husk
RHA	Rice husk ash
WRHA	White rice husk ash
UV	Ultraviolet
α	Alpha
β	Beta
γ	Gamma
PVA	Polyvinyl alcohol
XRF	X-Ray Fluorescence
XRD	X-Ray Diffraction
FTIR	Fourier Transform Infrared
FESEM	Field Emission Scanning Electron Microscopy
SEM	Scanning Electron Microscopy
UV-Vis	Ultraviolet-Visible
PL	Photoluminescence
E_{opt}	Optical band gap
JCPDS	Joint Committee on Powder Diffraction Standards

CHAPTER 1

INTRODUCTION

1.1 Background of study

The conversion of waste materials into wealth materials has gradually become an important in improving the recycling of solid wastes into value-added materials. In addition, the changes of lifestyle in the last decades and the increase in consumption of materials have led to an increase in the amount of wastes in the environment. The rapid industrial development has produced different types of waste materials such as glass, plastic, paper, steel as well as agricultural products. The wastes from our homes that we leave in the bins in our own backyards are just the tip of the iceberg of the total waste materials dumped each week, responsible for many serious environmental problems. Therefore, it is necessary to find new ways to solve the waste problem. Most countries in the world have started to take actions to deal with the problem by actively finding ways to add value to these wastes by recycling them into secondary source of materials.

In the last century, intensive work has been done to utilize waste materials in industrial and manufacturing processes, as recycling is the best way to dispose solid wastes. Recycling offers several advantages as it helps to save resources and energy, and by reducing incineration, it helps to protect the environment (Marinoni et al., 2013; Luo et al., 2017). Many leftovers and unwanted materials are dumped daily as a result of manufacturing processes (commercial, mining, industrial or agricultural operations) or community and household activities.

A considerable amount of literature has been published on the use of glass method in a wide range of applications such as electric, electronic and telecommunication industries. These studies focused on the use of the melt quenching technique to synthesize various shapes. With an aim to optimize the properties of glass and to gain advantages from glass with special glass-ceramic properties, many researchers have studied glass ceramic phase by re-sintering the glass at specific temperature to control crystallization in order to produce the best glass ceramic materials. Glass ceramics has many advantageous properties such as high strength, high chemical durability, low thermal expansion, high temperature stability, fluorescent and high resistivity (Höland et al., 2003). Therefore, there is large volume of published studies describing used the glass-ceramics in many industrial.

Recently, a great deal has focused on the synthesizing method and optical properties of a variety of oxide-based phosphors. Zinc silicate (Zn_2SiO_4), also known as willemite, is an ideal host material for rare earth and transition metal ions. Willemite is a very important material for efficient luminescence because it has high luminescent efficiency and excellent chemical stability (Selomulya et al., 2005). Thus, willemite has a promising future, as it is a favorably flexible luminescent material due to the broad range of multi-colors that can be emitted from it such as red, green and blue. These colors can be emitted by willemite at different dopant with europium (Eu^{3+}), manganese (Mn^{2+}) or cobalt (Co^{2+}) (Takesue et al., 2009). Therefore, willemite appears to be promising in producing traditional phosphors to be used in plasma display panels, cathode ray tubes and lamps because it has many good properties such as having very strong luminescence, high-saturated color, long lifespan and chemical stability.

Previous studies have primarily concentrated on the use of pure SiO_2 as the starting material to produce willemite that has a very high melting point. Most of these studies aimed to develop new methods to produce zinc silicate phosphor in order to reduce temperature and lower energy process. In the past few years, a large and growing body of literature has described several methods to prepare willemite such as solid-state, sol-gel, hydrothermal and supercritical water (Wang and Li, 2002; Mohd Zaid et al., 2016; Babu et al., 2017). The conventional solid-state method offers several advantages over other methods as it is able to produce willemite in a large scale, and this method saves time and energy, is less complicated and lowers product cost.

Recently, researchers have shown an increase use of solid wastes as the starting material for producing willemite phosphor (Insiripong et al., 2013; Marinoni et al., 2013; Samsudin et al., 2016). In general, using waste materials in the scientific field is affordable because majority of the waste materials are cheap and easily accessible. These help reduce the time and cost for their preparation.

Rice husk (RH) is one of the most material received high attentions in the last few years. A number of studies have shown that RH can be converted into an economically advantageous, viable product that can be used in future nanotechnology to reinforce materials (Soltani et al., 2015; Fernandes et al., 2016). RH is valuable as it can be used for many industrial applications due to its high silica content. RHA has many good properties such as highly porous and lightweight with a very high external surface area and is high in silica content in the forms of crystalline or amorphous materials (87-97 wt.% silica in an amorphous form and some amount of metallic impurities) (Kumar et al., 2012).

In this study, focus is on the use of white rice husk ash (WRHA) as the source of SiO_2 to prepared willemite derived from glass system (ZnO-WRHA) doped with MnO_2 using conventional solid-state method by controlling the sintering proses. The physical, structural and optical properties of the (ZnO-WRHA) glass and willemite glass-ceramic were characterized in order to determine the effects of the sintering process and MnO_2 doping. The density and linear shrinkage were then analyzed to study the physical properties. X-ray diffraction (XRD), Fourier transform infrared (FTIR) and field emission scanning electron microscopy (FESEM) were used to measure and identify the structure of the properties. The UV-Visible (UV-Vis) spectroscopy and photoluminescence (PL) spectroscopy were used to investigate the optical properties. The aim of this study was to evaluate and validate the synthesis of

MnO_2 doped willemite derived from white rice husk ash to reduce the cost of electroluminescent devices.

1.2 Problem statement

Over the last few years, the manganese doped willemite $Zn_2SiO_4:Mn^{2+}$ draw much attention in solid state laser, phosphor, optical and lighting devises due to their high-saturated color, long life span and strong luminescence (Tsai et al., 2010). In general, the conventional solid-state reaction method used to synthesis the commercial Zn_2SiO_4: Mn^{2+} phosphors. However, in this conventional solid-state method, the starting raw material such as ZnO and SiO_2 mixed well together and sintered for several hours at above 1000 ºC due to the high melting point of the starting materials which lead to loss in the time and energy.

Intensive research has been carried out to use pure SiO_2 as a starting material in the synthesis of $Zn_2SiO_4:Mn^{2+}$ to improve their physical, structural and optical properties through new synthetic processes and optimization of the host and activator species. In addition, a new method is used to improve the properties of the materials, reduce the time and processing temperature such as sol-gel (Omri et al., 2013), spray pyrolysis (Roh et al., 2003), hydrothermal (Pozas et al., 2005) and solvothermal method (Xiong et al., 2013).

Nevertheless, not much work has been carried out on the use of waste solid materials in synthesizing $Zn_2SiO_4:Mn^{2+}$. Therefore, very little information is known on the use of WRHA as a source of SiO_2 in synthesis $Zn_2SiO_4:Mn^{2+}$. RH has a high carbon and silicon ratio, which makes it useful not only as a fuel candidate, but also as a low-cost Si source. After combustion of the RH leave some solid wastes that is RHA with silica as the predominant component. Thus, RHA can be used as a Si source or a SiO_2 starting material to fabricate a variety of high value-added products. Recently, many attempts have been made to utilize RHs or RHA in chemical synthesis efficiently. Therefore, the objective of the present study is to determine the effects of sintering temperature on MnO_2 doped willemite derived from ZnO-SiO_2 glass, with WRHA as a source for SiO_2 in the conventional solid-state method. This study can help in the problem of disposal of solid wastes and reduce the cost of electroluminescent devices.

1.3 Research objectives

The current study involves an investigation of the effects of WRHA as a source of SiO_2 on the properties of willemite at different temperatures and concentrations of MnO_2 doped willemite. Therefore, this research was done to achieve several objectives as mentioned in the following:

1. To study the effect of SiO_2 derived from WRHA as based materials for preparation of Zinc Silicate Glass.
2. To study the physical, structural and optical properties of Zinc Silicate Glass doped MnO_2.

3. To investigate the effect of different sintering temperature on produce willemite doped with Manganese dioxide.

1.4 Scope of the study

In order to achieve the aims of the study, the scopes of the study stated as follow:

- ✓ WRHA was prepared in the electric furnace for a double heating treatment with temperatures ranging from room temperature up to 500 °C for 1 hour before being sintered at a temperature of 900 °C for 3 hours.
- ✓ A series of glass was prepared $[(ZnO)_{55} + (WRHA)_{45}]$ using the quenching in water technique which must be fast enough to prevent crystal growth.
- ✓ Willemite doped manganese, $Zn_2SiO_4:Mn^{2+}$ was prepared using WRHA, ZnO and MnO_2 powder based on the stoichiometric equation, $[(ZnO)_{55} + (WRHA)_{45}]_{100-x} [MnO_2]_x$ where x = 0, 1, 3 and 5 wt.% using the conventional solid state method.
- ✓ Archimedes principle was used to measure the density and study the linear shrinkage to determine the physical properties of sample.
- ✓ XRD, FTIR and FESEM were used to investigate the structure and morphology of willemite derived from WRHA.
- ✓ UV-Vis and PL spectroscopy were used to measure the optical properties of willemite, which are absorption, energy band gap and emission intensity of the sample.

1.5 The importance of the Study

Recently, much attention has studied on the utilization of waste materials to produce glass and glass-ceramic materials. A large volume of published studies have described the use of rare earth ions and transition metals in doped glass to enhance the properties of glass especially in terms of its optical properties. This is to produce glass with better properties to fabricate new materials such as solid-state lasers, phosphors, and optics. A lot of researches have focused on developing glass and glass-ceramic based phosphor to produce luminescent materials (Patra et al., 2005; Li et al., 2016). Many researchers have used luminescent materials in a wide range of applications which include cathode ray tubes (CRT), electroluminescence (EL), plasma display panels (PDP), fluorescent lamps and solid state lasers (Ronda, 1997; Van Der Kolk et al., 2000; Chen et al., 2015).

Among the inorganic silicate phosphors, willemite doped with transition metal are well known for its high luminescence efficiency coupled with excellent color purity (Babu et al., 2014). Mn-doped Zn_2SiO_4 green phosphor has been known to be a material of particular interest in many industries because of its high luminescence efficiency, high photo-stability (under UV excitation) and stability to moisture. These properties make it a common material of choice in fluorescent lamps, plasma display panels and vacuum fluorescent displays.

The processing methods are being studied with the intention of achieving low environmental burden processes, saving cost and developing a new technique for various applications (Omri and El Mir, 2014; Samsudin et al., 2016). Combustion of RH produces a lot of ash containing a huge amount of silica. Many studies have used RHA as a source of SiO_2 starting material to synthesize a different type of high value-added products. In the last few decades, numerous studies have attempted to utilize RHA in chemical synthesis. In the same context, the specific aim of this research is to characterize and study the physical, structural, and optical properties of willemite prepared from WRHA as resource of SiO_2 doped with MnO_2 through the conventional solid-state technique in producing high-performance silicate phosphors at lower cost. The evaluation is based in relation to the physical theories.

1.6 Thesis organization

The thesis is arranged as follows: Chapter 1 presents a brief introduction of willemite doped with manganese, the problem statements, the objectives, the scope and also the importance of the study. Chapter 2 presents past and present work together with brief information on willemite preparation and willemite derived from WRHA doped with MnO_2. Chapter 3 explains the methodology and characterization to prepare WRHA and MnO_2 doped Zn_2SiO_4. Chapter 4 presents the results and discussion of density, linear shrinkage, XRF, XRD, FTIR spectra, FESEM and UV-VIS analyses; and finally, Chapter 5 presents the conclusion and recommendations for future work.

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