UNIVERSITI PUTRA MALAYSIA

# SYNTHESIS AND CHARACTERIZATION OF ERBIUM-DOPED WILLEMITE- BASED GLASS AND GLASS CERAMIC FROM GREEN CULLET

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**By**

**SITI SYUHAIDA BINTI ABDUL RASHID**

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

**May 2018**

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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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**May 2018**

**Chairman : Sidek bin Hj. Ab Aziz, PhD**
**Faculty : Science**

Over the last few decades, the fabrication of glass ceramics using recycled waste glasses is of growing interest among some researchers to reduce energy consumption and cost of raw materials use. However, most of the previous studies were using only the clear soda lime silica (SLS) glass in the production of the glass ceramics. In this study, green SLS waste glass is used as a source of silica to produce the erbium doped willemite-based glass ceramic ($Zn_2SiO_4$:$Er^{3+}$) using the conventional melt-quenching technique followed by controlled sintering process. The effect of different sintering temperatures (700 to 1100 °C) and dopant compositions ($Er_2O_3$= 0, 1, 2, 3, 4, and 5 wt.%) on the physical, structural and optical properties of the glass and glass ceramic samples were investigated using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), fourier transform infrared (FTIR) spectroscopy, ultraviolet-visible (UV-Vis) spectroscopy, and photoluminescence (PL) spectroscopy. Experimental results show that the density and linear shrinkage of the samples increased from 2.35 to 2.86 g/cm$^3$ and 6.17 to 24.56%, respectively, with increasing $Er^{3+}$ dopant percentage and sintering temperature. The XRD analysis revealed the presence of thermodynamically stable zinc orthosilicate (α-$Zn_2SiO_4$) phase at sintering temperatures 800 ºC and above. The morphologies from FESEM image shows an increased in grain size from 65 to 283 nm and the formation of densely packed grains as the sintering temperature increases. The presence of the IR bands corresponding to both $SiO_4$ and $ZnO_4$ tetrahedral from FTIR analysis confirmed the formation of $Zn_2SiO_4$ crystal in the sintered samples. From UV-Vis analysis, the optical band gap shows an increment from 3.06 to 3.73 eV as the sintering temperature increased from 800 to 1100 °C which is expected due to the improved crystallinities of the samples. The optical band gap also shows a decrement as the $Er^{3+}$ composition increased from 3 to 5 wt.%, which might be due to the decrease in compactness of the network and the formation of more non-bridging oxygen (NBO). From PL analysis, the enhancement of the emission intensities of the glass and glass ceramic samples can be observed with the progression of sintering temperature under excitation



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of 385 nm. The PL analysis also shows that the luminescence intensities are strongly dependent on the improvement in crystallinity and the dopant composition.

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

**SINTESIS DAN PENCIRIAN ERBIUM DOP WILLEMITE BERASASKAN KACA DAN SERAMIK KACA DARIPADA SERPIHAN KACA HIJAU**

Oleh

**SITI SYUHAIDA BINTI ABDUL RASHID**

**Mei 2018**

**Pengerusi : Sidek bin Hj. Ab Aziz, PhD**
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Sejak beberapa dekad lalu, pemfabrikatan seramik kaca menggunakan kaca buangan yang dikitar semula semakin mendapat perhatian di kalangan sesetengah penyelidik bagi mengurangkan penggunaan tenaga dan kos penggunaan bahan mentah. Walau bagaimanapun, kebanyakan kajian yang lepas hanya menggunakan kaca kapur soda silika (SLS) jernih sahaja dalam pembuatan seramik kaca. Dalam kajian ini, kaca buangan SLS hijau telah digunakan sebagai sumber silika untuk menghasilkan erbium dop willemite berasaskan seramik kaca ($Zn_2SiO_4$:$Er^{3+}$) menggunakan teknik lindapan leburan yang diikuti oleh proses pensinteran terkawal. Kesan suhu pensinteran (700 hingga 1100 °C) dan komposisi dopan ($Er_2O_3$ = 0, 1, 2, 3, 4, dan 5 wt.%) yang berbeza terhadap sifat fizikal, struktur dan optik sampel kaca dan seramik kaca telah dikaji menggunakan pembelauan sinar-X (XRD), mikroskop pancaran medan pengimbasan elektron (FESEM), spekroskopi inframerah (FTIR), spektroskopi ultraungu-nyata (UV-Vis), dan spektroskopi kefotopendarcahayaan (PL). Hasil eksperimen menunjukkan ketumpatan dan pengecutan lelurus sampel masing-masing meningkat dari 2.35 hingga 2.86 g/cm$^3$ dan 6.17 hingga 24.56% dengan peningkatan peratusan dopan $Er^{3+}$ dan suhu pensinteran. Analisis XRD menunjukkan kehadiran fasa ortosilikat zink termodinamik yang stabil ($\alpha$-$Zn_2SiO_4$) pada suhu pensinteran 800 ºC dan ke atas. Morfologi dari imej FESEM menunjukkan peningkatan dalam saiz butiran dari 65 hingga 283 nm serta pembentukan butiran yang padat apabila suhu pensinteran meningkat. Kehadiran jalur IR bagi tetrahedron $SiO_4$ dan $ZnO_4$ dari analisis FTIR mengesahkan pembentukan kristal $Zn_2SiO_4$ dalam sampel yang telah disinter. Dari analisis UV-Vis, jurang jalur optik menunjukkan peningkatan dari 3.06 hingga 3.73 eV apabila suhu pensinteran meningkat dari 800 ke 1100 °C yang dijangka disebabkan oleh pengkristalan sampel yang bertambah baik. Jurang jalur optikal juga menunjukkan penurunan dengan peningkatan komposisi $Er^{3+}$ dari 3 hingga 5 wt.% yang mungkin disebabkan oleh penurunan kepadatan rangkaian serta pembentukan lebih banyak oksigen tidak bersambung (NBO). Dari analisis PL, peningkatan keamatan pancaran sampel kaca dan seramik



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kaca dapat dilihat dengan peningkatan suhu pensinteran di bawah pengujaan 385 nm. Analisis PL juga menunjukkan bahawa keamatan kependarkilauan sangat bergantung kepada peningkatan dalam kekristalan dan kandungan dopan.

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I certify that a Thesis Examination Committee has met on 28th May 2018 to conduct the final examination of Siti Syuhaida binti Abdul Rashid on her thesis entitled "Synthesis and Characterization of Erbium-Doped Willemite-Based Glass and Glass Ceramic from Green Cullet" 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 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 graduate student**

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This is to confirm that:

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# LIST OF ABBREVIATIONS

| | |
|---|---|
| $Al_2O_3$ | Aluminium oxide |
| BaO | Barium oxide |
| CaO | Calcium oxide |
| $Ce^{3+}$ | Trivalent cerium ion |
| $Co^{2+}$ | Divalent cobalt ion |
| $Cr_2O_3$ | Chromium oxide |
| DSC | Differential scanning calorimetry |
| EDXRF | Energy Dispersive X-ray Fluorescence |
| $E_g$ | Optical energy band gap |
| $Er_2O_3$ | Erbium oxide |
| $Er^{3+}$ | Trivalent erbium ion |
| $Eu^{3+}$ | Trivalent europium ion |
| $Fe_2O_3$ | Ferric oxide |
| FESEM | Field Emission Scanning Electron Microscopy |
| FTIR | Fourier Transform Infrared |
| GSLS | Green soda lime silica |
| JCPDS | Joint Committee on Powder Diffraction Standards |
| $K_2O$ | Potassium oxide |
| MgO | Magnesium oxide |
| $Mn^{2+}$ | Manganese |
| $Na_2O$ | Sodium oxide |
| $Ni^{2+}$ | Divalent nickel ion |
| PL | Photoluminescence |
| PVA | Polyvinyl alcohol |
| $SiO_2$ | Silicon dioxide |
| SLS | Soda lime silica |
| $Sm^{3+}$ | Trivalent samarium ion |
| $Tb^{3+}$ | Trivalent terbium ion |
| UV-Vis | Ultraviolet-Visible |
| XRD | X-ray Diffraction |
| $Zn_2SiO_4$ | Zinc silicate/ Willemite |
| ZnO | Zinc oxide |
| $\alpha$ | Alpha |
| $\beta$ | Beta |
| $\gamma$ | Gamma |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Research background

Glass ceramics are considered as crystalline materials that can be produced through the controlled crystallization of a precursor glass during specific heat treatment (Alizadeh and Marghussian, 2000). The fact that crystallization can take place in the conventional glassy structure when they are heated has long been appreciated. When the glass structure is heated to a certain temperature, it will induce crystallization, crystal growth, crystal transitions, and a gradual disappearance of the glassy matrix (Carter and Norton, 2013). The properties of the glass ceramics are depended on the temperature of heat treatment, the composition of the precursor glass, and the addition of nucleating agents (Hu *et al*., 2005).

Glass ceramic can provide significant advantages over the conventional glass or ceramic materials by combining the flexibility of forming and inspection of glass. The variation of compositions and the possibility of developing special microstructures with specific technological properties have allowed glass ceramic materials to be used in a wide range applications (Khater and Mahmoud, 2017).

It has now been established that wide range of silicate-rich wastes, such as coal combustion ash, slag from steel production, fly ash and filter dusts from waste incinerators, mud from metal hydrometallurgy, different types of sludge as well as glass cullet or mixtures of them can be reused in the production of glass ceramic materials (Rawlings *et al*., 2006; Isa, 2011). It is well established that the usage of recycled waste glass in glass ceramics provide an environmental safeguard by reducing the process energy consumption, saving natural resources, and lowering the pollutant emission levels (Shi and Zheng, 2007; Marinoni *et al*., 2013; Andreola *et al.*, 2016). Many studies have investigated the use of waste glass in the production of glass ceramic materials such as willemite (Samsudin *et al*., 2015; Effendy *et al*., 2016; Nurzilla *et al*., 2016; Omar *et al*., 2016a; Zaid *et al*., 2016a), wollastonite (Bernardo *et al*., 2006), ceramic tiles (Lin, 2007; Kim *et al*., 2016), cement, and concrete (Poutos *et al*., 2008; Jani and Hogland, 2014).

Glass ceramics containing willemite have been known, for many years, as materials for electronic applications and also for the fabrication of glazes (Omar and Abdel-hameed, 2009). Willemite or zinc silicate ($Zn_2SiO_4$) is identified as an ideal host material to incorporate with rare earth ions and transition ions such as $Er^{3+}$, $Tb^{3+}$, $Ce^{3+}$, $Eu^{3+}$, $Sm^{3+}$, $Ni^{2+}$, $Co^{2+}$, and $Mn^{2+}$ with various visible range emission colors like blue, green, yellow and red. This is due to the properties of



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willemite that have a large band gap (~5.5 eV), chemical stability, and transparency in the UV-visible range, which make it suitable for use as phosphor material in various application such as in electroluminescent devices, cathode ray tubes (CRT), and plasma display panels (PDPs) (Feldmann *et al*., 2003; Takesue *et al*., 2009a; Krsmanovi *et al*., 2011).

Over the last decade, numerous investigations on the synthesis routes of willemite have been carried out, including the conventional solid state method, hydrothermal, solvothermal, supercritical water, co-precipitation, spray pyrolysis method, sol-gel technique, and vapor method (Takesue *et al*., 2009b). These methods have been proposed regardless of either the purpose of the study was to reduce the energy consumption or to improve the material performance.

## 1.2 Problem statement

Willemite is known as an ideal host matrix for transition metal and rare earth dopant ions for its efficient luminescence which make it suitable for applications in electronic and optoelectronic systems. Among the rare earth ions, erbium oxide has been attractive to be used as a dopant for its special luminescent properties due to 4f intra-levels with emissions in the ultraviolet, visible and infrared regions (Maia *et al*., 2016).

Over the past few decades, there is a growing demand for waste glass to be re-used or recycled in the fabrication of new glass or glass ceramics to economize raw materials and energy resources substantially (Min'Ko *et al*., 2010). The uses of recycled SLS glasses in many researches are well-known because of various reasons such as its fine mechanical and optical properties besides its low production temperature.

In recent years, a great deal of interest has been focused on the utilization of waste SLS glass as a source of silica in the preparation of willemite-based glass ceramic by the melt-quenching technique followed by controlled sintering process (Samsudin *et al*., 2015; Sarrigani *et al*., 2015a; Omar *et al*., 2016b; Zaid *et al*., 2016b). However, most of the studies in the literatures were using only the clear SLS glass and no attempt was done to explore the potential of utilizing the green SLS (GSLS) glass in the production of willemite-based glass ceramic so far. It is known that the significant difference between the clear and the green SLS glass is only by the presence of chromium oxide ($Cr_2O_3$) which give the green color to the glasses.

Several researchers have described that the presence of $Cr_2O_3$ is highly effective as nucleating agents to instigate crystallization centers in inducing volume crystallization and the formation of fine-grained microstructures in order to obtain the nanocomposite glass ceramics (Krishna *et al*., 2007; Omar and Abdel-hameed, 2009; Khater and Mahmoud, 2017).



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Therefore, this research will focus on the preparation and characterization of erbium doped willemite-based glass ceramic using the waste GSLS glass as a replacement of the raw $SiO_2$. The effects of different sintering temperatures and dopant compositions on the physical, structural and optical properties of the glass and glass-ceramics samples will also be investigated.

## 1.3 Objectives

This research was carried out based on several objectives that are mentioned below:

i. To synthesize the glass and $Zn_2SiO_4$: $Er^{3+}$ glass ceramic samples from waste GSLS glass, ZnO and $Er_2O_3$ by using the conventional melt-quenching technique followed by controlled sintering process.
ii. To study the effect of different sintering temperatures on the physical, structural and optical properties of $Zn_2SiO_4$: $Er^{3+}$ based glass ceramic.
iii. To investigate the effect of various $Er_2O_3$ doping concentration towards physical, structural and optical properties of $Zn_2SiO_4$: $Er^{3+}$ based glass ceramic.

## 1.4 Scope of the study

In order to reach the objectives of this research, the scope of the study are as follows:

1) Preparation of a series of precursor glass using GSLS glass , ZnO and $Er_2O_3$ powder based on the stoichiometric equation of $(ZnO_{0.5}GSLS_{0.5})_{1-x}(Er_2O_3)_x$ where x = 0, 1, 2, 3, 4, and 5 wt.%, by conventional melt-quenching technique.
2) Confirming the chemical composition of the precursor glass using EDXRF spectroscopy.
3) Sintering the pellet form of the precursor glass to produce the Er-doped willemite-based glass ceramics, $Zn_2SiO_4$: $Er^{3+}$ with the temperature varied from 700 °C to 1100 °C.
4) Analyzing the physical, structural and optical properties of the precursor glass and Er-doped willemite-based glass ceramic samples using densitometer, linear shrinkage, XRD, FESEM, FTIR, UV-Vis and PL spectroscopy.

## 1.5 Chapter organization

This thesis consists of 5 chapters including introduction, literature review, methodology, results and discussion, and conclusion. Chapter 1 is the introduction about this research including the problem statements, the objectives and the scope of this study. In Chapter 2, some literatures that related to the study are reviewed. Chapter 3 focuses on the experimental details in this study, where the



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procedures and methods to prepare the erbium doped willemite-based glass ceramic samples, and the characterization techniques used, are stated. The experimental results and discussions concerning the effect of sintering temperatures and erbium content towards the physical, structural and optical properties of the precursor glass and Er-doped willemite-based glass ceramic samples are presented in Chapter 4. The last chapter, which is Chapter 5 serves the conclusion and suggestion for future study.



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