UNIVERSITI PUTRA MALAYSIA

SHYNTHESIS AND CHARACTERIZATION OF WOLLASTONITE GLASSCERAMICS PREPARED FROM CULLET DOPED WITH SAMARIUM OXIDE

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By

KARIMA A MOHAMED ALMASRI

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

May 2018

DEDICATION

To my beloved parents Amer Mohamed Almasri and Rabia Ali Essa
For their unconditional love and support

To my sisters
For making my life complete

To my husband and my kids
For their love and care

To all my very wonderful friends
For making my life full of joy and happiness

To all my lecturers
For helping me at a lot throughout this journey

Thank you all

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 : Professor Sidek Hj. Abd Aziz, PhD
Faculty : Science

Wollastonite ($CaSiO_3$) based glass ceramics doped with various amounts of samarium oxide (Sm_2O_3) were prepared using a melt-quenching technique based on the empirical formula $[(CaO)_{0.21}(SLS)_{0.79}]$1-y($Sm_2O_3$)y, y=(0,1,2,3,4,5) wt.%. The effect of different sintering temperatures on physical, optical and structural properties of wollastonite ($CaSiO_3$) based glass-ceramics were investigated for its potential application as a building material. In this work, soda lime silica glass waste was utilized as a source of silicon. The chemical composition and physical properties of glass were characterized by using Energy Dispersive X-ray Fluorescence (XRF) and Archimedes principle. The Archimedes measurement results show that the density increased with the increase of sintering temperature and doping Sm_2O_3 concentration. The generation of $CaSiO_3$, morphology, size and crystal phase with increasing the heat-treatment temperature was examined by field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD) and Fourier transform infrared reflection spectroscopy (FTIR). The average calculated crystal size gained from XRD was found to be in the range 60 nm. XRD results show that after the sintering at 700 °C, the sample still in the amorphous phase. The increasing of sintering temperature to 800 °C cause the nucleation of triclinic wollastonite ($CaSiO_3$), through increasing the sintering temperature to 900 ^oC, para wollastonite (β-$CaSiO_3$) phase appeared, and the progression of sintering temperature above 1000 ^oC improved almost of the positions and relative intensities of the crystalline peaks. The FESEM results show a uniform distribution of particles and the morphology of the wollastonite crystal is granular. FTIR results exhibited the structural evolution of wollastonite based glass ceramics. The appearance of CaO, SiO_2, and Ca-O-Si bands disclosed from FTIR which showed the formation of $CaSiO_3$ crystal phase. In addition to the calculation of the energy band gap which found to be increased from 3.96 eV to 4.1 eV with increasing sintering temperature, also increased from 4.1 eV to 4.9 eV with increasing of samarium oxide concentration.

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

SINTESIS DAN PENCIRIAN WOLLASTONITE KACA-SERAMIK DIHASILKAN DARIPADA KULET DIDOPKAN DENGAN SAMARIUM OKSIDA

Oleh

KARIMA A MOHAMED ALMASRI

Mei 2018

Pengerusi : Profesor Sidek Hj. Abd Aziz, PhD
Fakulti : Sains

Kaca seramik berasaskan wollastonite ($CaSiO_3$) yang didopkan dengan pelbagai jumlah samarium oksida (Sm_2O_3) telah disediakan menggunakan teknik pelindapkejutan yang berdasarkan formula empirikal $[(CaO)_{0.21}(SLS)_{0.79}]_{1-y}$ $(Sm_2O_3)y$, di mana y = 0, 1, 2, 3, 4, 5 wt.%. Kesan suhu rawatan haba pada sifat-sifat fizikal, optik dan struktur wollastonite kaca-seramik disiasat kerana kaca-seramik ini berpotensi untuk digunakan sebagai bahan binaan. Dalam penyelidikan ini, kaca terbuang (SLS) telah digunakan sebagai sumber silikon. Komposisi kimia dan sifat fizikal kaca dicirikan dengan menggunakan (EDXRF) dan prinsip Archimedes. Hasil pengukuran Archimedes menunjukkan bahawa ketumpatan sampel meningkat dengan kenaikan suhu rawatan haba dan kepekatan didopkan Sm_2O_3. Penjanaan morphologi, saiz dan fasa kristal $CaSiO_3$, dengan meningkatkan suhu rawatan haba telah diperiksa oleh (FESEM), (XRD) and (FTIR). Saiz purata kristal yang dikira daripada alat XRD didapati berada dalam lingkungan 60 nm. Keputusan XRD juga menunjukkan bahawa selepas proses rawatan haba pada 700 ° C, sampel masih berada dalam fasa amorphus. Peningkatan suhu rawatan haba kepada 800 ° C menyebabkan nukleasi wollastonite triklinik ($CaSiO_3$), dan seterusnya melalui peningkatan suhu rawatan haba kepada 900 ° C, fasa wollastonite (β-$CaSiO_3$) muncul, dan kemajuan suhu rawatan haba melebihi 1000 ° C telah merubah kedudukan dan meningkatkan intensiti relatif puncak kristal. Hasil analisis FESEM memperlihatkan pembahagian seragam zarah dan morfologi kristal wollastonit adalah dalam bentuk berbutir. Analisis FTIR pula berhasil mempamerkan perubahan struktur kaca-seramik wollastonite. Kehadiran dan pembetukan unit CaO, SiO_2, dan Ca-O-Si daripada analisis FTIR telah membuktikan pembentukan fasa kristal $CaSiO_3$. Di samping itu, pengiraan jurang jalur tenaga juga didapati meningkat dari 3.96 eV kepada 4.1 eV dengan peningkatan suhu rawatan haba, dan juga perubahan dari 4.1 eV ke 4.9 eV dengan peningkatan kepekatan samarium oksida.

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I certify that a Thesis Examination Committee has met on 22 May 2018 to conduct the final examination of Karima A Mohamed Almasri on her thesis entitled "Synthesis and Characterization of Wollastonite Glassceramics Prepared from Cullet Doped with Samarium Oxide" 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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LIST OF ABBREVIATIONS

$CaSiO_3$	= Wollastonite
$CaSiO_3:Sm^{3+}$	= Wollastonite doped Samarium Oxide
Sm^{3+}	= Samarium
UV	= Ultraviolet
SLS	= Soda lime silica
SiO_2	= Silica oxide
CaO	= Calcium oxide
Na_2O	= Sodium oxide
Al_2O_3	= Aluminium oxide
K_2O	= Potassium oxide
MgO	= Magnesium oxide
B_2O_3	= Boron trioxide
ZnO	= Zinc Oxide
Fe_2O_3	= Ferric oxide
$CdSiO_3$	= Cadmium silicate
TeO_2	= Tellurium dioxide
NaAlSiO4	= Nepheline
$Ca(OH)_2$	= Calcium Hydroxide
P_2O_5	= Phosphorus Pentoxide
Dy^{3+}	= Dysprosium oxide
TiO_2	= Titanium dioxide
Cr_2O_3	= Chromium oxide
Ga_2O_3	= Gallium oxide
RE	= Rare Earth
α	= Alpha
β	= Beta
PVA	= Polyvinyl alcohol
EDXRF	= Energy dispersive X-ray fluorescence
XRD	= X-Ray diffraction
FTIR	= Fourier transform infrared
FESEM	= Field emission scanning electron microscopy
IR	= Infrared spectroscopy
UV-Vis	= Ultraviolet-Visible
E_g	= Optical band gap
FWHM	= Full width at half maxima
JCPDS	= Joint committee on powder diffraction standards

CHAPTER 1

INTRODUCTION

1.1 General Introduction

The most crucial calcium silicate based glass-ceramic for application in the building industry manufactured by the Japanese firm "Nippon Electric Glass" was recently named Neoparis ® (Montazerian *et al*., 2015). Wollastonite, which is found in nature, is also known as calcium silicate ($CaSiO_3$) and has been widely studied due to its beneficial application in ceramic, dental implant, architecture, and construction where these materials are used as floor materials as a substitute the granite and natural marble (Boccaccini *et al.*, 2000; Lu *et al.*, 2014). The fundamental feature of this material is curved panels and big flat can be produced commercially (Perez *et al.*, 2012; Teixeira *et al*., 2014a).

Conventionally, wollastonite based glass-ceramic was produced from SiO_2-CaO-Al_2O_3 glass system through controlled surface crystallization. Such glass-ceramic materials may show specific visual impacts and other major characteristics, such as better hardness than natural stones, good strength, low shrinkage, absence of volatile constituents, fluxing characteristics, body permeability, whiteness, zero water absorption and low density (Zhang *et al*., 2013; Liu *et al*., 2014a). It is fabricated on a massive scale and is utilized as floor coating outside as well as interior of a building. One major advantage of wollastonite based glass-ceramics material over natural stones is that it can be fabricated to produce big flat and curved panels. Wollastonite begins to crystallize at temperatures above 950 °C as the wollastonite phase (triclinic) begins to emerge. As the temperature increases, the formation of needle-like β-wollastonite (monoclinic) through the glass surface in the direction of the inner grain of the glass increases, so that the compound produced is similar to marble or granite due to variation in light diffraction indicators between the glass and the crystals in the matrix (Holland *et al*., 2002). At higher temperature, α-wollastonite (monoclinic, pseudo-wollastonite) with grainy crystalloid morphology with obscure crystals, are formed (Holland *et al*., 2002).

In many countries, industrial by-product, fly ash, or slag ash are employed as base materials in the ceramic industry to produce glass-ceramic (Rawlings *et al*., 2006; Lunip *et al*., 2016). This process relies on waste composition and additives, which generally consists of impurities and secondary components. Interest in soda lime silica (SLS) glass waste is by virtue of its constitution and the large amount of SLS manufactured in Malaysia. This glass makes up a large portion of domestic waste. Amongst the conventional glasses, SLS glass is known as the most typical commercial glass merchandise that comprise up to 90 - 95% of the glass produced throughout the world (Sinton *et al*., 2001). These types of glasses are commonly used since they have a virtuous glass-forming characteristics in comparison to other typical glass systems. SLS glasses are usually used for making windowpane, glass containers, flat glasses, packaging and insulating materials, bioactive materials, and building material. Consumption of raw materials decreases when SLS glass is recycled, thus yielding economic and environmental benefits (Juoi *et al*., 2013; Zaid *et al*., 2015). Preparation of wollastonite using high purity silicon dioxide (SiO_2) powder is costly and its synthesis requires a high temperature. Therefore, SLS glasses are chosen as a substitute for SiO_2 source as it can reduce production cost and has the benefit of being an attractive host matrix due to its good mechanical and optical properties, such as high transparency, perfect chemical stability, high thermal stability, and low melting point (Zaid *et al.,* 2011).

One of the primary concern in rare earth doped glasses is in defining the dopant environment. Hypersensitive transitions has been discovered in the spectra of all rare earth ions (Eraiah, 2006). The rare earth ion Sm_2O_3 may be employed as a dopant in various crystal hosts as well as glass hosts. By doping rare earth ions, the structural and optical properties of glasses can be improved and obtaining optimized concentrations is challenging in laser glass research, Raman studies suggested that Sm_2O_3 could modify the properties of glass, these glasses are expected to give interesting application in the field of optics. Notable improvements of optical and structural properties due to the doping samarium ions are evidenced (Reddy *et al*., 2017).

To the extent of the author's knowledge studies on the optical properties of wollastonite based glass-ceramics are very limited. In this work, the fabrication of wollastonite based glass-ceramics through controlled sintering of prepared CaO-SLS glasses is discussed. The synthesis of Sm_2O_3 doped wollastonite-based glass ceramic using waste materials in different weight percentages (1-5 wt. %) of Sm^{3+} through the conventional melt-quenching method was studied. The crystallization process was studied using the Fourier transform infrared reflection (FTIR) spectroscopy, X-ray diffraction (XRD), and field emission scanning electron microscopy (FESEM). The glass and the derived glass-ceramics were characterized by studying their physical, optical, and structural properties, including their optical band gap. The primary usefulness of the CaO-SLS glass system in comparison to the normal CaO-SiO_2 glass system is that the processing and melting as well as temperature will be reduced substantially. This reduces the production cost for this glass-ceramic. In fact, the core idea of this article is the blending and description of wollastonite based glass-ceramic derived from CaO-SLS glass system doped with samarium oxide.

1.2 Problem Statement

Over the last few years, many research of rare earth doped glasses has been given a lot of focused by reason of their extensive utilization in the optical areas, such as optical switches for laser, sensor, and optical communication (Maheshvaran *et al*., 2011). The most important concern with rare earth doped glasses is in defining the dopant effect on the host materials (Eraiah, 2006). Manjunatha *et al*. (2013) has analyzed the structure, morphology, and optical characteristics of Sm^{3+} doped $CdSiO_3$. However, no much work has been done with regard to wollastonite glass and its optical properties, and only a few studies regarding it has been published, while no studies on samarium oxide has been published, and due to the uses of Sm_2O_3 in optical and infrared absorbing glass to absorb infrared radiation, Sm_2O_3 was doped to wollastonite in this study.

There is, however, a scarcity of information on the effects of wollastonite doped transition metals on soda lime silica (SLS) glass as a source of silica, SiO_2. The majority of researchers employed pure SiO_2 as a base material in the blending process. Substituting SLS for SiO_2 as a source of silica to produce wollastonite could also help reduce the problem of solid waste disposal in Malaysia. On average, every Malaysian produce approximately 1.2 kg of waste each day. Wastes are generated in various forms, such as paper, glass, construction material, wood, plastic, food scrap, and aluminum. In Malaysia, these wastes are sent to landfill or incineration center. The volume of solid waste produced in the country has increased to 33,000 tons in 2013 from the 19,000 tons generated in 2005; 3% of the total waste is composed of glass materials.

Many of the previous studies used preparation methods such as hydrothermal method and sol-gel methods rather than solid-state method. This is because solid-state method requires the use of high amount of thermal or electrical energy and the resulting products have morphologies that are limited to agglomerated shapes. However, due to the complexity and high cost of these new fabrication methods, it would be difficult to commercialize samarium oxide (Cho and Chang, 2003). On the other hand, a solid-state method offers several advantages over other methods. For examples, this method of producing wollastonite is less complex and is able to produce wollastonite on a large scale, hence saves time and energy and reduce production cost. The crystallinity of wollastonite produced using the solid-state method is considerably higher than those prepared using hydrothermal and solvothermal methods (Takesue *et al*., 2009). Due to the important usages of wollastonite as building materials, thus, in this study, wollastonite doped samarium oxide was prepared using SLS glass as a source of SiO_2 by using conventional solid-state method.

1.3 Hypotheses

Wollastonite has beneficial application in ceramic, architecture, and construction where these materials are used as floor materials.

The density of the samples will be increased with increasing the sintering temperatures due to decreasing the pore size of the glass-ceramic samples and increased the densification, also it will be increased with increasing the concentration of Sm_2O_3 due to the addition of modifier oxide which breaks up the Si–O–Si linkage, and increase the free space in the glass network. Para wollastonite β-$CaSiO_3$ will be formed at temperatures 900 ˚C and above and the crystallinity of the glass-ceramic samples will be improved at high sintering temperatures. The grain sizes of the samples will be increased with increasing the sintering temperatures and with increasing the concentration of Sm_2O_3. The energy band gap of the samples will be increased with increasing the sintering temperatures due to the increase of sizes and number of the crystals formed also it will be increased with increase the concentration of Sm_2O_3.

1.4 Objectives of the Study

The objectives of this study can be summarized as follows:

1) To synthesis wollastonite doped samarium oxide based glass-ceramics by using conventional melt quenching method followed by sintering method.
2) To study the effect of different sintering temperatures on the physical, structural, and optical characteristics of wollastonite based glass-ceramics
3) To examine the effect of Sm^{3+} doping on the physical, structural, and optical characteristics of wollastonite based glass-ceramics.

1.5 Scope of the Research

The sphere of the research are:

1. Wollastonite doped samarium oxide, $CaSiO_3:Sm^{3+}$, is prepared using SLS glass, CaO, and Sm_2O_3 powder based on the stoichiometric equation $[(CaO)_{0.21}(SLS)_{0.79}]_{1-y}[Sm_2O_3]_y$ where y = (0,1,2,3,4,5) wt.% using conventional solid state method. All starting materials will be used as reference materials.
2. The sintering temperatures for producing $CaSiO_3:Sm^{3+}$ varies between 700 °C and 1100 °C.
3. The structure, bonding and morphology of the $CaSiO_3:Sm^{3+}$ will be characterized using XRD, FTIR and FESEM respectively.
4. The optical properties, namely band gap of the samples, will be measured using UV-Vis spectroscopy.

1.6 Importance of the Study

This study focuses on wollastonite, or $CaSiO_3$, since it is used in the production of ceramics for use as sanitary ware and tableware. More recently, wollastonite is being used in electrical application as high voltage insulator (Puntharod *et al*., 2013). Researchers are currently working on improving the properties of wollastonite as a material for bio-ceramics and biomaterials, for example artificial bond, antibacterial growth, and as a platform for regeneration of hard tissue (Ortega *et al*., 2010; Magallanes *et al*., 2011).

1.7 Outline of the Thesis

This thesis is organized as follows. Chapter 1 presents an introduction to wollastonite doped with samarium oxide, problem statements, objectives, scope, and significance of the study. Previous and current works done by other researchers throughout the world are discussed in Chapter 2. Chapter 3 explains the methods used to prepare wollastonite doped with samarium oxide and the characterization method. Chapter 4 discusses and analyzes the effect of the content of samarium and sintering temperatures on the physical, structural and optical properties of wollastonite doped with samarium oxide. Lastly, Chapter 5 presents the conclusion of the study and suggestions for upcoming works.

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