UNIVERSITI PUTRA MALAYSIA

CHARACTERIZATION OF WOLLASTONITE-BASED GLASS CERAMIC FROM CLAMSHELL AND SODIUM LIME SILICA BOTTLES

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By

FATEMA RADEEF MAHMOUD

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

April 2019

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DEDICATION

To my sister and my brother for their unconditional love and support

To all my very wonderful friend for make my life full of joy and happiness

To all my lecturer 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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April 2019

Chairman : Professor Sidek Hj. Ab Aziz, PhD
Faculty : Science

Wollastonite glass-ceramics have been fabricated in this work using clamshell (CS) and soda lime silicate (SLS) glass waste. The samples were prepared using a melt-quenching technique based on the chemical formula $[(CS)_x(SLS)_{100-x}]$ with x = 10, 20, 30, 40, and 50. The samples were sintered at 600, 700 and 1000°C.The literature has not reported the utilization of CS and the SLS glass waste in the fabrication of wollastonite based glass ceramics. The CS is chosen as a source of CaO and the SLS glass waste provides the required SiO_2, and these wastes are expected to provide another alternative means of managing the disposed of SLS glass and CS wastes in the metropolitan cities around the world. Hence, the objectives of this research were to fabricate a series of wollastonite glass-ceramics from clamshell and SLS glass waste and to study both the clamshell concentration and sintering temperature effects on the physical and structural properties of the materials. To achieve the research objectives, measurements and characterizations such as density, linear shrinkage, X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), and energy dispersion X-ray fluorescence (EDX) analysis of the prepared samples were carried out. The density values and linear shrinkage mostly increased with the concentration of CS in the materials and sintering temperature where the density is 28.4 g/cm^3 and the linear is 7.11 mm. This increment is probably associated with structural changes leading to more interstitial space as shown by SEM micrograph structures. The XRD results showed the presence of peaks of wollastonite, cristobalite, augite, diopside and quartz phases. The XR results show that after the sintering at 600°C, the sample was still in the amorphous phase, the increased in the sintering temperature to 700°C the peaks were found through increasing the sintering temperature to 1000°C the phase appeared. The wollastonite intensity increases when sintering temperature increases. The elemental EDX analysis of the green and sintered samples at 600°C, 700°C and 1000°C revealed the presence of carbon (C), oxygen (O), sodium (Na), silicon (Si) and calcium (Ca). The FTIR analysis of the samples showed mainly the absorptions due to the vibrations of Si–O–Si linkages, symmetric

Si–O and bond bending of Si–O–Si, symmetric stretching vibrations of O–Si–O bending, and Ca–O stretching modes. The sintering temperature and clamshell concentration both have structural and physical effects on the material as revealed in FTIR spectra, that was clear when they obtained the bond of calcium groups and the bond of silicon groups.

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

PENCIRIAN SERAMIK KACA WOLLASTONIT DARI KERANG DAN BOTOL NATRIUM KAPUR

Oleh

FATEMA RADEEF MAHMOUD

April 2019

Pengerusi : Profesor Sidek Hj. Ab Aziz, PhD
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Seramik kaca wolastonit telah dihasilkan di dalam kajian ini dengan menggunakan kerang (CS) dan sisa kaca kapur soda silika (SLS). Sampel-sampel tersebut telah disediakan dengan menggunakan teknik sepuhlindap berdasarkan formula kimia $[(CS)_x(SLS)_{100-x}]$ dengan x = 10, 20, 30, 40 dan 50. Sampel-sampel itu telah dibakar pada suhu 600, 700 dan 1000°C. Kajian literatur masih belum lagi melaporkan tentang penggunaan CS and sisa kaca SLS dalam pembuatan wolastonit berasaskan kaca seramik. CS dipilih sebagai sumber CaO and sisa kaca SLS membekalkan SiO_2 yang diperlukan dan sisa-sisa ini dijangkakan alternatif lain sebagai untuk menguruskan menguruskan kaca SLS yang terbuang dan sisa CS di dalam bandar metropolitan di seluruh dunia. Oleh itu, objektif penyelidikan ini adalah untuk menghasilkan satu siri seramik kaca wolastonit daripada kerang dan sisa kaca SLS dan juga untuk mempelajari kesan kepekatan kerang dan kesan suhu pembakaran pada ciri-ciri fizikal dan struktur bahan itu. Untuk mencapai objektif tersebut, pengukuran dan pengujian seperti ketumpatan, pengecutan linear, pembelauan sinar-X (XRD), spektroskopi Inframerah Jelmaan Fourier (FTIR), Mikroskopi Elektron Pengimbasan (SEM) dan analisis penyebaran tenaga pendarfluor Sinar-X (EDX) telah dijalankan. Nilai ketumpatan dan pengecutan linear kebanyakannya menaik dengan kenaikan kepekatan kerang di dalam bahan itu dan suhu sinter di mana ketumpatannya adalah 28.4 g/cm^3 dan pengecutan linear adalah 7.11 mm. Kenaikan ini mungkin ada kaitan dengan perubahan struktur yang menyebabkan lebih banyak ruang interstis seperti yang ditunjukkan di dalam struktur mikrograf SEM. Keputusan XRD menunjukkan kehadiran puncak-puncak wolastonit, kristobalit, augit, diopsid dan kuartz. Keamatan wolastonit meningkat dengan kenaikan suhu pembakaran. Analisis elemen EDX bagi sampel yang tidak disinter dan disinter pada 600°C, 700°C dan 1000°C menunjukkan kehadiran karbon (C), oksigen (O), natrium (Na), silikon (Si) and kalsium (Ca). Analisis FTIR didapati sampel-sampel itu menunjukkan penyerapan itu terutamanya disebabkan oleh getaran hubungan Si–O–Si, Si–O simetri dan pembengkokan ikatan Si–O–Si, getaran regangan simetri pembengkokan Si–O–Si, dan mod regangan Ca–

O. Suhu sinter dan kepekatan kerang mempunyai kedua-dua kesan struktur dan fizikal pada bahan itu seperti yang ditunjukkan pada spektrum FTIR, di mana ia jelas menunjukkan ikatan kumpulan kalsium dan ikatan kumpulan silikon.

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I certify that a Thesis Examination Committee has met on 2 April 2019 to conduct the final examination of Fatema Radeef Mahmoud on her thesis entitled "Characterization of Wollastonite-Based Glass Ceramic from Clamshell and Sodium Lime Silica Bottles" 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 is to confirm that:

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LIST OF ABBREVIATIONS

$CaSiO_3$	Wollastonite
SLS	Soda lime silica
SiO_2	Silica oxide
CaO	Calcium oxide
CS	Clam shell
Na_2O	Sodium oxide
K_2O	Potassium oxide
MgO	Magnesium oxide
ZnO	Zinc oxide
Fe_2O_3	Ferric oxide
TeO_2	Tellurium oxide
Al_2O	Aluminum oxide
$Ca(OH)_2$	Calcium Hydroxide
P_2O_5	Phosphorus Pentoxide
α	Alpha
β	Beta
ρ	Density
LS	Linear shrinkage
PVA	Polyvinyl alcohol
EDX	Energy dispersive X-ray
XRD	X-Ray diffraction
FTIR	Fourier transform infrared
FESEM	Field emission scanning electron microscopy

CHAPTER 1

INTRODUCTION

1.1 Research background

As the world energy demand and products consumption grow, raw material availability and their management processes become very important. In the last few decades, many different methods have been developed by scientists for recycling the unwanted wastes to wealth materials for various applications. The waste recycling process may ensure both the industrial raw material demand and the environmental hazards are reduced to minimal levels (Saddeek et al., 2005; Benzerga et al., 2014).

The most crucial wollastonite based on glass-ceramics for application in 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 identified as calcium silicate $CaSiO_3$ and has been widely studied according to its beneficial application in ceramic, teeth implant, architecture design, and structure building where these materials are utilized as ground materials as exchange the stone and natural limestone (Boccaccini et al., 2000). The fundamental advantage of this material is curved panels and big flat can be manufactured commercially (Teixeira et al., 2014a).

Usually, wollastonite based glass-ceramics was made from SiO_2–CaO–Al_2O_3 glass scheme through influenced surface crystallization. Such glass-ceramic materials may appearance specific visual effects and other main characteristics, for example better hardness than natural stones, perfect strength, low shrink, absence of unstable ingredients, fluxing characteristics, form permeability, lightness, zero water absorption and low density (Zhang et al., 2013; Liu et al., 2014). It is invented on a massive scale and is used as floor coating outside as well as the interior of a building. One main benefit of calcium silicate based on glass-ceramics material over natural stones is that it can be fabricated to produce big flat and curved panels.

Wollastonite begins to crystallize at a high temperature above 950°C as the calcium silicate phase (triclinic) begins to emerge. As the temperature increases, the formation of needle-like β-wollastonite (monoclinic) from side to side the glass surface in direction of the inner grain of glass increases, so that the compound produced is similar to marble or stone due to variation in light diffraction signal between the glass then the crystals in the matrix. At higher heat, α- wollastonite (pseudo- wollastonite) with granulated crystalloid morphology with obscure crystals, are formed (Holland et al., 2002).

In many countries, an industrial by-product, fly dust, or slag dust are employed as base materials in ceramic industry to produce glass-ceramic (Rawlings et al., 2006; Lunip et al., 2016). This method depends on waste composition and additives, which mostly consist of impurity and secondary components. Attention in soda lime silica (SLS) glass unused is by virtue of its constitution and the large amount of SLS have been manufactured. 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 comprises able to 90 - 94% of the glass produced throughout the world (Sinton et al., 2001). These kinds of glasses are normally used since they have a worthy glass-forming characteristic in comparison to other typical glass systems. Soda lime silica glasses are commonly utilized for making windowpane, glass containers, flat glasses, packaging materials and isolate materials, bioactive tools, and structure 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). Production of wollastonite using great purity silicon oxide (SiO_2) powder is costly and its synthesis requires a high temperature. As a result, SLS glasses are chosen as exchange for SiO_2 source as it be able to reduce production cost and have the benefit of being an appealing host matrix according to its good mechanical and optical properties, for example, high limpidity, good chemical stability, great thermal stability and small melting point (Zaid et al., 2011).

Glass-ceramics are fine microstructures of polycrystalline materials produced through the process of controlled crystallization (devitrification) of a glass (Chinnam et al., 2013). Waste such as steel production slag, fly ash and filter dust, mud from metal hydrometallurgy, combustion ash from coal, glass cullet or their mixture and many different types of silicate wastes have been used for the production of glass-ceramics (Yoon et al., 2013). SLS glass wastes and rice husk ash have been used in several works as a source of SiO_2 for the production of glasses and glass-ceramics (Lee et al., 2017; Umar et al., 2017a). Due to their high quality in terms of chemical durability and thermo-mechanical properties, glass-ceramics are used as architectural, decorative and construction components (Yoon et al., 2013). They are generally applied as biomaterials in orthopedics for bone replacement and dental restoration, material coating for dies in industries, equipment for chemical processing and metallurgical industries and also for optical applications (Yoon et al., 2013).

Glass-ceramics are produced in a process involving two steps: the first step is the glass production using the conventional method and the second step involves the heat treatment to precipitate the crystal phase. The heat treatment is for crystalline separation from the glassy matrix in a form of small crystals (Krsmanović et al., 2007). The temperature applied and heating duration controls the number of crystals formed, growth rate and final size of the formed crystals (Krsmanović et al., 2007). Glass-ceramics made from a naturally occurring calcium metasilicate material called wollastonite has been under study lately (Laura et al., 2016). With a low dielectric constant, low dielectric lose, low thermal expansion, low thermal conductivity and high thermal stability, wollastonite has been considered a promising material for ceramic fabrication (Prakasam et al., 2015). Wollastonite ceramics are used as biomaterials in medicine, as filler material in resins and plastics, construction, metallurgical industry, paints, asbestos replacement, abrasives and glasses (Yazdani et al., 2010). Wollastonite is found in natural form, as acicular, inert, calcium metasilicate with elastic modulus values high and less expensive fibres than any of steel or carbon micro-fibres. They used in many different products, both synthetic and ceramic micro-fibres reinforcement. In cement industry, wollastonite is utilized to enhance the flexural strength, pre-peak modification and post-peak load behaviour of a system of hydrated cement and cement-silica (Mathur et al., 2007).

This work utilizes the recycle SLS glasses as the source of silica (SiO_2) and CS as the source of calcium oxide (CaO) as the raw materials for the fabrication of wollastonite based glass-ceramic. The density of the prepared samples will be determined. The EDX, XRD, FTIR and SEM analysis of the samples will be carried out to determine the chemical composition, and structural properties of the glass and glass-ceramic samples.

1.2 Problem Statement

Many researchers have reported different works on the fabrication of glass ceramics from both domestic and industrial wastes. Taurino and other coworkers (2016) reported having prepared wollastonite based glass-ceramic from borosilicate glass waste and destroyed washing machine glasses. The required $CaCO_3$ is sourced from the washing machine glass, whereas SiO_2 was obtained from the borosilicate glass waste. Considering that limestone contains a high proportion of $CaCO_3$ (about 55.1%) and silica sand with a proportion of SiO_2 up to 99%, Rashid and other coworkers (2014) utilized these two raw materials to prepare wollastonite by conventional solid-state reaction. The study revealed that limestone and silica sand can produce wollastonite with good quality. Yoon and coworkers used glass waste and coal fly ash to prepare wollastonite glass ceramic with the aim of achieving high mechanical quality and better chemical durability. The report suggested that for the prepared material, the mechanical quality properties of the sample obtained at 1000 and 1050°C sintering temperature are the best. This is established from the chemical durability, density, bending strength and compressive strength (Yoon et al., 2013). Other waste materials reported to have utilized for the fabrication of wollastonite glass ceramic in the literature include the eggshells (ES) and the SLS glass waste. The SLS was used as the source SiO_2 while the ES provides the required CaO (Hazlin et al., 2015). However, the literature has not reported the utilization of CS and the SLS glass waste in the fabrication of wollastonite based glass ceramics. The CS is chosen as a source of CaO and the SLS glass waste provides the required SiO_2 and these wastes are expected to provide another alternative means of managing the disposed of SLS glass and CS wastes in the metropolitan cities around the world. This work studies the chemical, physical and structural properties of the fabricated wollastonite based glass-ceramic through EDX, XRD, FTIR, SEM characterizations. The work also studies the effects of temperature of sintering on the physical, chemical, morphological, and structural properties of the wollastonite based ceramics.

1.3 Research objectives

The aim of the study was to fabricate and characterize the wollastonite based glass-ceramics from clamshells and SLS glass waste. The objectives through which the aim was to be achieved are as follows;

1. To produce and synthesis CaO-SiO_2 glasses from clamshell (CS) and soda lime silica SLS glass waste
2. To investigate the effects of different CS and SLS concentrations on the properties of the wollastonite based glass-ceramics.
3. To study the effects of different sintering temperature on the physical and structural properties of the wollastonite based glass-ceramics.

1.4 Scope of Study

The scope of the study includes the utilization of clamshells and the soda lime silica (SLS) glass waste to fabricate a wollastonite glass ceramic with imperial formula $[(CS)_x (SLS)_{100-x}]$ for x= 10, 20, 30, 40, and 50. The study also includes the EDX characterization to determine the chemical composition of the prepared material. XRD, FTIR and SEM characterizations performed to study the structural properties of the materials.

1.5 Significance of Study

The ever increasing population density growth in the world and utilization of electronic and glassy materials by the populace brought about by the rise in their living standard has become a cause for alarm (Hazlin et al., 2015). These used and unwanted or broken materials and other industrial waste if not properly managed may be harmful to the population and the environment (Mugoni et al., 2015). The process of recycling helps in reducing the usual high demand for natural raw materials, to save energy in manufacturing, and to minimize waste environmental impacts (Chinnam et al., 2013). This study opens a door for more utilization of the waste glass through application in the glass-ceramic technology. The study also attempts to improve the quality of the wollastonite glass-ceramic material in terms of the physical and structural properties. And open another door for more studies on the utilization of the two raw materials for different scientific, medical, industrial and domestic applications.

1.6 Thesis Organization

The thesis consists of five chapters, with each chapter describing the sequence of study as described as follows;

Chapter one provides the introduction to the background of glass-ceramics and wollastonite based glass-ceramics with their applications. It contains the problem statement, the scope of the study, aim and objective, significance of study and thesis organization.

The second chapter describes an overview of related literature on glass-ceramics, wollastonite, SLS glasses waste, wollastonite glass-ceramic. The chapter also presents the principles and theories employed in the study. The theories applied in some of the characterizations as EDX, XRD, FTIR, the ultrasonic non-destructive characterizations and density. Chapter three describes the materials, methods and the experimental techniques employed in the study. Also mentioned are the equipment and chemicals used in the process of the study and all the characterizations involved.

The fourth chapter presents and discusses the results from the EDX analysis result, through density, XRD, FTIR and the non-destructive ultrasonic analysis results of the various characterizations, measurements and calculations carried out. Chapter five, which is the final chapter presents the overall conclusion based on the obtained results and gives recommendations on some future study in the field

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CHARACTERIZATION OF WOLLASTONITE-BASED GLASS CERAMIC FROM CLAMSHELL AND SODIUM LIME SILICA BOTTLES

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