UNIVERSITI PUTRA MALAYSIA

PHYSICAL, STRUCTURAL AND MECHANICAL PROPERTIES OF WOLLASTONITE-BASED FOAM GLASS CERAMICS USING EGGSHELL AND SODA-LIME-SILICA GLASS WASTE

DUR IFFA BINTI SAPARUDDIN

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By

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

December 2020

DEDICATIONS

To my beloved parents,
Who have made me what I am today

To siblings and family
For their love and care

To all of my lovely friends
For cheering up my day

And most importantly to all of my lecturers
For guiding me with a massive knowledge 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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December 2020

Chairman : Mohd Hafiz Mohd Zaid, PhD
Faculty : Science

Foam glass-ceramics represents a highly valuable solution for lightweight fill material, which has a low density, incombustible, and good in mechanical strength. In this work, foam glass-ceramics were prepared by the solid-state conventional method, using soda-lime-silica (SLS) glass waste as a matrix glass and eggshell (ES) as a foaming agent. The purpose of this study is to synthesize and study the changes that occur in physical, structural, and mechanical properties of foam glass-ceramic derived from waste materials by varying the ES content, sintering temperature, and sintering duration. Both of the raw materials were milled and sieved to the particle size of 45 μm. The powders then were homogenized with the empirical formula, $[ES]_x[SLS]_{100-x}$ where x = 1, 3, 6, and 9 wt.%. and uniaxially pressed with 5 MPa. The obtained compact powders were heat-treated at 700, 800, and 900 °C for 30, 60, and 120 min with the constant heating rate at 10 °C/min. The foam-glass ceramic samples were then analyzed based on physical properties; measured by density, linear expansion, and porosity. The structural properties of the foam glass-ceramic samples were determined by X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), and Fourier Transform Infrared Spectroscopy (FTIR). Then, the mechanical properties of the sample were measured using Universal Testing Machine (UTM). The optimum parameter of the foam glass-ceramics was obtained at 800 °C for 60 min with the substitution of 3 to 6 wt.% ES content. In fact, the optimum temperature was supported by the thermogravimetric analyzer (TGA) which indicates that the mass loss of $CaCO_3$ in ES occurs at the temperature of 800 °C. The samples prepared this way to provide a minimum bulk density of 0.326 – 0.421 g/cm^3 with the maximum porosity of 87.2 – 83.16 %. Furthermore, XRD analysis revealed that the formation and growth of cristobalite (SiO_2) and wollastonite ($CaSiO_3$) crystal phases after the heat-treatment process at 800 °C. Meanwhile, the FTIR analysis indicates the strong intensity of Ca–O bond at 620 to 650 cm^{-1} of wavenumber associate with decreasing the intensity peak of

C–O after the heat-treatment process at 800 °C. FESEM analysis showed that the pores are larger and abundant distributed with the size of the diameter's pore is 861 – 1200 μm in the samples with 3 to 6 wt.% of ES content. The results of compressive strength for the sample with 3 and 6 wt.% ES content increases from 0.04 and 0.42 MPa, respectively, depending on the crystalline phase content. Therefore, 6 wt.% ES is an appropriate ratio for fabricating foam glass-ceramics with a low density and high compressive strength. The preparation of foam glass-ceramics using solid wastes provide a promising way to prepare ceramic aggregate for the construction application as well as giving benefit to the economy and environment issue.

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

SIFAT FIZIKAL, STRUKTURAL DAN MEKANIKAL WOLLASTONITE-BERASASKAN KACA SERAMIK BERLIANG MENGGUNAKAN SISA KULIT TELUR DAN KACA-SODA-KAPUR-SILIKA

Oleh

DUR IFFA BINTI SAPARUDDIN

Disember 2020

Pengerusi : Mohd Hafiz Mohd Zaid, PhD
Fakulti : Sains

Seramik kaca berongga merupakan penyelesaian penting untuk bahan pengisi ringan, yang mempunyai ketumpatan rendah, tidak mudah terbakar dan kekuatan mekanik. Dalam tesis ini, seramik kaca berongga disediakan dengan kaedah konvensional keadaan pepejal, menggunakan kaca soda-kapur-silika (SLS) sebagai kaca matriks dan kulit telur (ES) sebagai agen pembuih. Tujuan kajian ini adalah untuk mensintesis dan mengkaji sifat fizikal, struktur, dan mekanikal kaca-seramik dari bahan buangan dengan mengubah kandungan ES, suhu sintering, dan tempoh pensinteran. Kedua-dua serbuk bahan mentah itu digiling dan diayak dengan saiz zarah 45 µm. Serbuk kemudian dihomogenkan dengan formula empirik, $[ES]_x[SLS]_{100-x}$ di mana x = 1, 3, 6 dan 9 wt.% komposisi berat dan ditekan dengan 5 MPa. Serbuk padat yang diperolehi dirawat panas pada suhu 700, 800, dan 900 °C selama 30, 60, dan 120 minit dengan kadar pemanasan pada 10 °C/min. Sampel seramik kaca berongga dianalisis berdasarkan sifat fizikal; diukur dengan ketumpatan, pengembangan linier, dan keliangan. Sifat struktur sampel busa-keramik kaca ditentukan oleh XRD, FESEM dan FTIR. Kemudian, sifat mekanik sampel diukur menggunakan UTM. Parameter optimum untuk menyediakan seramik kaca berongga diperoleh pada suhu 800 °C selama 60 minit dengan 3 hingga 6% berat ES. Pemerhatian suhu optimum disokong oleh TGA yang menunjukkan bahawa kehilangan jisim $CaCO_3$ di ES berlaku pada suhu 800 °C. Sampel disediakan dengan cara ini untuk memberikan ketumpatan pukal minimum 0.326 – 0.421 g/cm^3 dengan keliangan maksimum 87.2 – 83.16%. Selanjutnya, analisis XRD mendedahkan bahawa pembentukan dan pertumbuhan fasa kristal cristobalite dan wollastonite setelah proses perlakuan panas pada suhu 800 °C. Sementara itu, analisis FTIR menunjukkan intensiti kuat ikatan Ca–O pada 620 hingga 650 cm^{-1} dari bilangan gelombang dikaitkan dengan penurunan puncak intensiti C–O setelah proses rawatan panas pada 800 °C. Analisis FESEM menunjukkan bahawa pori-pori lebih besar dan banyak didistribusikan dengan ukuran pori diameter adalah 861 –

1200 μm pada sampel 3 hingga 6 wt.% peratusan ES. Hasil kekuatan mampatan untuk sampel dengan kandungan ES 3 dan 6 wt.% meningkat masing-masing dari 0.04 dan 0.42 MPa, bergantung pada kandungan fasa kristal. Oleh itu, kandungan ES pada 6 wt.% adalah nisbah yang sesuai untuk pembuatan seramik kaca berongga dengan ketumpatan rendah dan kekuatan mampatan yang tinggi. Penyediaan seramik kaca busa menggunakan sisa pepejal memberikan cara yang menjanjikan untuk menyiapkan agregat seramik untuk aplikasi pembinaan serta memberi manfaat kepada masalah ekonomi dan persekitaran.

ACKNOWLEDGEMENTS

Alhamdulillah, praise to Allah, Lord of the world because give me an opportunity in terms of strength, patience, courage, and provide me with good health and intellectual ability to capable finish this research within the given time. The thesis title physical, structural, and mechanical properties of wollastonite-based foam glass ceramics using eggshell and soda-lime-silica glass waste able to complete successfully. I take this opportunity to express my gratitude to the people who have been instrumental in the successful completion of this thesis.

Firstly, I would like to express a lot of thank goodness to my supervisor, Dr. Mohd Hafiz Mohd Zaid, and my co-supervisor, Prof. Dr. Sidek Hj. Ab Aziz with Assoc. Prof. Dr. Khamirul Amin Matori, the important person during my study who gave a lot of encouragement, guidance, and support during the completion of this research. Special appreciation also goes to my beloved parents and family members for their unflagging love and support throughout my life. Higher appreciation also to my members from Ceramic Ultrasonic Laboratory (CURL) for their opinions and suggestions incomplete this dissertation. Apart from that, I also acknowledge the financial support from Jabatan Perkhidmatan Awam (JPA). This dissertation would have been impossible without them.

Last but not least, I offer my regards and blessings to all of those who supported me in any aspect during the completion of the project, and may Allah S.W.T. repay all your kindness.

This thesis was submitted to the Senate of the 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:

Mohd Hafiz bin Mohd Zaid, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Sidek bin Hj. Ab Aziz, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

Khamirul Amin bin Matori, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 11 March 2021

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Signature: ________________________
Name of Chairman of Supervisory Committee: Dr. Mohd Hafiz bin Mohd Zaid

Signature: ________________________
Name of Member of Supervisory Committee: Professor Dr. Sidek bin Hj. Ab Aziz

Signature: ________________________
Name of Member of Supervisory Committee: Associate Professor Dr. Khamirul Amin bin Matori

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LIST OF ABBREVIATIONS AND SYMBOLS

ES	Eggshell
SLS	Soda-lime silicate
$CaCO_3$	Calcium carbonate
SiO_2	Silicon dioxide
wt.%	Weight percentage
XRF	X-ray fluorescence
TGA	Thermogravimetric analysis
XRD	X-ray diffraction
FTIR	Fourier transform infrared
FESEM	Field emission scanning electron microscopy
UTM	Universal testing machine
Na_2O	Sodium oxide
K_2O	Potassium oxide
CaO	Calcium oxide
MgO	Magnesium oxide
Na_2CO_3	Sodium carbonate
$MgCO_3$	Magnesium carbonate
Al_2O_3	Aluminium oxide
$CaSiO_3$	Calcium silicate (Wollastonite)
PVA	Polyvinyl alcohol
µm	micrometer
ρ_b	Bulk density
ρ_t	True density
ε	Porosity
D_i	Initial diameter
D_f	Final diameter
n	integer
λ	wavelength
d	Distance between atomic layers in the crystal

δ	Stress/pressure applied on a material
ε	Strain
JCPDS	Joint Committee on Powder Diffraction Standards

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Nowadays, municipal solid wastes are abundant in the landfill sites. In 2012, the World Bank estimates waste generation in world cities is around 1.3 billion tonnes/year and expected to reach approximately around 2.2 billion tonnes by 2025 (Hoornweg & Bhada-Tata, 2012). In Malaysia, 80% of solid waste categorized as recyclable waste is disposed of at landfills. However, these wastes are unable to recycle completely due to the limited source of separation (Moh & Manaf, 2014). Therefore, these kinds of recyclable waste materials such as paper, plastics, and glass having the greatest potential to be converted from waste to new valuable products. Recently, wastes have provided value-added opportunities and cost-effective for use as raw materials and give products of wealth technology. Several previous studies use waste as a matrix that has a similar chemical composition such as glass bottles, rice husk (Fernandes et al., 2019), fly ash (Guo et al., 2014), red mud (Liu et al., 2019) and mineral wool (Ji et al., 2019). The type of waste glass such as cathode ray tube panel, SLS glass, lab-ware glass, and printed circuit board.

There is a different type of glass waste, having its chemical composition which contributes to potential applications. As an example, soda-lime-silica (SLS) glass is a conventional glass type used daily, hence it has been a significant amount of urban wastes. The reuse of SLS glass is still limited due to the societal and technological factors, though its production is non-hazardous (Pontikes et al., 2007). SLS glass is the type of waste glass produced from crushed glass, bottles, containers, and lighting products manufactured by melting calcium carbonate ($CaCO_3$), silicon dioxide (SiO_2), sodium carbonate (Na_2CO_3), and other minor constituents (Silva et al., 2017). The 'lime' given because of the presence of additives of calcium oxide (CaO), and sodium oxide (Na_2O) to reduce the softening point from 1600 to 700 ºC for the preparation of this type of glass. The softening point temperature of SLS glass is slightly low compared to other silicate glass which promotes less energy usage during the sintering process.

Foam glass-ceramic is a porous material, lightweight and characterized as a heterophase system (Sasmal et al., 2015). It is useful and can be a candidate to be used in thermal insulation materials such as ceramic materials in the furnace, insulating for roofs, ceilings, and walls (Hurley, 2003; Souza et al., 2017). Producers of foam glass-ceramics from Europe and North America currently use a high percentage of processed post-consumer glass in their products. There are three types of foam glass-ceramics as shown in Figure 1.1. From Figure 1.1, loose foam glass-ceramic aggregates are the type that continuous the sheet of foam glass-ceramics then broken into loose foam glass-ceramics aggregate. Next, blocks and shapes of foam glass-ceramics are produced by a batch process. It also continuous production of blocks and shapes in molds that are then cut and shaped to a specific size. The last type of foam glass-ceramic is in the form of pelletization which is a continuous production of spherical pellets of foam glass-ceramics that can apply in the manufacture of blocks, panels and slabs. The significance of this foam type material for the industrial where it can minimize heat loss or heat gain (Aditya et al., 2017).

Figure 1.1 : Three main types of foam glass-ceramics form
(Source: Hurley, 2003)

In the glass-ceramic foam research, the porous structure formed inside softened glass makes it advantageous to construction applications. There is a high production cost in fabricating commercial foam glass-ceramics nowadays because of the superior physical and mechanical properties. Hence, there is limited usage of this material in building and industry because of the high-priced. Besides, current insulator (polymeric materials) has high flammability and poor sound absorption property compared to foam glass (Ji et al., 2019). Moreover, the production of commercial foam glass-ceramics from Pittsburgh Corning Company required a high sintering temperature at 1000 ºC. The properties of final product foam glass-ceramics are highly depending on the size and distribution of the foaming agent, and sintering temperature, duration as well as heating rate. The type of porous could be closed porosity and open porosity depending on the several parameters that affect the viscosity of the system. The closed porosity is mainly applicable to heat-insulating materials. Meanwhile, open porosity is used for soundproof material. Open porosity usually formed in porous glass-ceramic using a decomposing type of foaming agent and in glass that able to crystallizes (König et al., 2020).

In this research work, low-cost foam glass-ceramics using waste materials (SLS glass as a glass matrix and eggshells (ES) as a foaming agent) as a precursor has been studied which having a low bulk density, homogenous close porosity, and desired mechanical strength. A series of $[ES]_x[SLS]_{100-x}$ where x = 1, 3, 6, and 9 wt.% are prepared from solid-state conventional method to fabricate foam glass-ceramics. The properties of starting materials such as SLS glass and ES have been characterized by X-ray fluorescence (XRF) and thermogravimetric analyzer (TGA), respectively. Meanwhile, the characteristic of foam glass-ceramics has been characterized to study the effect of ES content, sintering temperature, and sintering duration. The structural and mechanical properties of foam glass-ceramics have been studied by X-ray diffraction (XRD), Fourier transforms infrared spectroscopy (FTIR), Field emission scanning electron microscopy (FESEM), and compressive strength by Universal testing machine (UTM). In view of the above fact, the aim of this study is the fabrication and characterization of foam glass-ceramics derived from waste materials, SLS glass, and ES waste as potential materials used in the field of construction for concrete aggregate.

1.2 Problem Statement

Nowadays, there is an abundant of solid wastes accumulate in landfill sites. The food waste encounter 49.3% among other type of solid waste as the worldwide consume it in daily life (Moh & Manaf, 2014). Generally, the food waste would give a negative impact to the environment, consequently led to the solid waste pollution. In this research, the foam glass-ceramics is fabricated derived from waste materials which are SLS glass and ES wastes in order to solve environment issue. Furthermore, there are limited reports and systematic study regarding to the physical, structural and mechanical properties of foam glass-ceramics derived from waste materials. ES is used as a foaming agent because it contains 95% of $CaCO_3$ and these carbonate is decomposed to CO_2 gas at 800 °C of temperature (Fernandes et al., 2013).

In recent years, foam glass-ceramics have attracted great attention due to its properties and a wide range of applications. Foam glass-ceramics exhibit excellent properties such as long-lifetime, reusability, water- and chemical-resistant, and dimensionally stable. Foam glass-ceramics is a valuable product that provides the cellular structure inside softened glass particles. The porous structure contributes to the lightweight of the product and it is easily brittle. Therefore, the strength of the foam glass-ceramics needs to be considered to enhance the stability of the porous structure during the installation process. Nowadays, there is an insufficient of information related to the mechanical properties of foam glass-ceramics because the foam glass-ceramics have much attention on heat insulation, sound absorption, non-flammable, and moisture-resistant (Guo et al., 2020).

For that reason, a comprehensive study of the crystallization, properties, and effect of heat-treatment on foam glass-ceramics derived from SLS glass and ES as a foaming agent is carried out to improve the physical, structural and mechanical properties of foam glass-ceramics. The transformation of the glass to glass-ceramics has become an integral part to improve the quality and properties of the final products. The results of this research are expected to find potential applications for the aggregate in construction building blocks.

1.3 Objectives

1. To synthesis and optimize the fabrication of foam glass-ceramic derived from SLS glass and ES waste using solid-state sintering method.
2. To study the effect of difference ES content on the physical, structural, and mechanical properties of foam glass-ceramics.
3. To determine the effect of heat-treatment temperature and duration on the physical, structural, and mechanical properties of foam glass-ceramics.

1.4 Significant of the study

Nowadays, there are a huge number of solid wastes are placed in the landfill. The amount of this solid waste continues to increase which is more than 30,000 tons in Peninsular Malaysia (Jereme et al., 2015). In fact, there is a lack of landfill sites to dispose the solid wastes. Therefore, it is an essential part to encourage the reuse the wastes such as SLS glass from glass bottles and the ES food waste to the wealth product. Hence, it can minimize the waste dump issue. In this research, the foam glass-ceramic is fabricated by mixing the SLS glass and ES as a foaming agent. It's useful as thermal insulation materials and acoustic appearance, especially to the construction industry (De Moraes et al., 2019). Besides, it can be applied to the various area of industry depend on the pore structure either the isolated pores or communicating pores. Therefore, the effect of the porosity of foam glass-ceramics is determined in this study by varying the different content of ES, different sintering temperature, and duration.

1.5 Outline of the thesis

In this study, Chapter 1 will give introduction of foam glass-ceramics from SLS glass and ES, the problem statements, the objectives, and also the importance of this study. The theory of glass, glass-ceramic and previous works including past and current has been carried out by other researchers are covered in Chapter 2. The apparatus, methodology and characterization of the precursor glass and foam glass-ceramics are explained in Chapter 3. The results concerning the effect of ES addition to the SLS glass, progression of heat treatment temperatures and duration towards the physical, structural and mechanical properties of foam glass-ceramics are analyzed and discussed in Chapter 4. Finally, the conclusion and suggestion for future works are state in Chapter 5.

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BIODATA OF STUDENT

Dur Iffa binti Saparuddin was born on 6th August 1993 in Kuantan, Pahang, Malaysia. She received her primary education at Sekolah Kebangsaan Indera Mahkota Utama and continued her secondary education at Sekolah Menengah Kebangsaan Sultanah Hajjah Kalsom, Kuantan, Pahang. After receiving SPM results, she furthered her study at Universiti Teknologi Pahang (UITM) Pahang for the Diploma in Science before continuing her undergraduate studies in Bachelor of Science (Hons.) Physics for 3 years, at the same university and graduated on April 2018. In the present, she further her study in Master Science in the field of Materials Science at Universiti Putra Malaysia under supervision by Dr. Mohd Hafiz Mohd Zaid.

LIST OF PUBLICATIONS

Journal

Saparuddin, D. I., Hisham, N. A. N., Sidek, H. A. A., Matori, K. A., Honda, S., Iwamoto, Y., Zaid, M. H. M. (2020). Effect of sintering temperature on the crystal growth, microstructure and mechanical strength of foam glass-ceramic from waste materials. Journal of Materials Research and Technology, 9(3), 5640–5647.

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