



UNIVERSITI PUTRA MALAYSIA

***PHYSICAL, STRUCTURAL AND MECHANICAL PROPERTIES OF
FOAM GLASS-CERAMICS DERIVED FROM WASTE SODA-LIME-
SILICA GLASS AND CLAMSHELL***

NOOR AIZAT BIN NOOR HISHAM

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**PHYSICAL, STRUCTURAL AND MECHANICAL PROPERTIES OF FOAM
GLASS-CERAMICS DERIVED FROM WASTE SODA-LIME-SILICA GLASS
AND CLAMSHELL**

By

NOOR AIZAT BIN NOOR HISHAM

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

April 2021

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DEDICATIONS

In the name Allah (S.W.T), the Most Gracious and Merciful
The thesis is dedicated

To my beloved mother and father, Sabariah Yatini and Noor Hisham Noordin

Who gave me unconditional love, support, and care

To siblings and families
Who are always there when in need

To my fiancé, Nurul Nadhirah Ahmad Shukri, and her family
For sharing their love and support throughout my study

To all my wonderful peers
For making my social life full of joy and happiness

To my supervisor, Dr. Mohd Hafiz Mohd Zaid, and member of supervisory
committees
For helping me a lot throughout this journey and made my thesis materialize

This research is dedicated to you

Thank you

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PHYSICAL, STRUCTURAL AND MECHANICAL PROPERTIES OF FOAM GLASS-CERAMICS DERIVED FROM WASTE SODA-LIME-SILICA GLASS AND CLAMSHELL

By

NOOR AIZAT BIN NOOR HISHAM

April 2021

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Nowadays, municipal solid waste has risen to a very alarming level throughout the nation. Our country is heavily dependent on open dumping and landfills, although these methods are expensive and time-consuming. The enormous number of solid waste management problems has encouraged scientists and researchers to think about converting waste materials into other beneficial materials. Therefore, an initiative has been taken through this research to utilize the soda-lime-silica (SLS) waste as the glass matrix and the source of silicon (SiO_2) and clamshell (CS) as the foaming agent (pore former) to fabricate the light-weight foam glass-ceramics (FGC). A series of FGC samples with 1, 3, 6 and 9 wt.% CS were successfully prepared by the conventional solid-state sintering method at 700, 800 and 900 °C sintered for 30, 60 and 120 min. The physical, structural, and mechanical properties of the raw materials and FGC were characterized by wavelength dispersive X-ray fluorescence (WDXRF), thermogravimetric analysis (TGA) density, linear expansion, total porosity, X-ray diffraction (XRD), fourier transform infrared (FTIR), field-emission scanning electron spectroscopy (FESEM) and compressive strength measurement. From the result of WDXRF to study the chemical composition of raw materials, SLS glass mainly composed of 71.90 wt.% of SiO_2 meanwhile CS has high weight percentage of CaO which is 53.41 wt.%. Based on these results, CS is found to contain 98.60 wt.% CaCO_3 . For the structural properties of raw materials, the XRD pattern of SLS glass reveals no continuous or discrete sharp peak with a broad feature of the amorphous halo at an angle around $2\theta = 20$ to 40° and meanwhile, the shape of the CS powder peak signifies the reflection of the aragonite crystalline phase with an orthorhombic crystal system. Based on TGA profile of CS, the thermal properties of CS indicate the gradual decline of CaCO_3 had occurred after undergoing the thermal treatment around 860 °C with a weight loss of approximately 43.56 %. The bulk density of the FGC samples is found to achieve minimum density (0.525 g/cm^3) with maximum expansion (83.98 %) at

3 wt.% CS sintered at an optimum sintering temperature and holding time which is 800 °C for 60 min. The bulk density increases as the linear expansion and total porosity decreases with the progression of CS contents, sintering temperature, and holding time, where the results agree with the FESEM micrograph. The result of XRD and FTIR transmittance spectra have shown the formation of wollastonite crystal starting at 3 wt.% CS sintered at 800 °C for 30 min. The crystallization of the wollastonite crystal phase increases with the progression of CS content, sintering temperature, and holding time. The trend of the compressive strength corresponds well with the trend of the bulk density. The highest mechanical performance (0.78 MPa) with an acceptable low bulk density (0.612 g/cm³) and average total porosity (74.81 %) is obtained for the sample containing 3 wt.% CS sintered at an optimum temperature and holding time. It can be concluded that the compressive strength increases with the decrease in linear expansion and the total porosity value of the FGC. The overall result promotes that the FGC derived from SLS glass and CS waste materials have a high potential to be used as concrete aggregate materials in the building and construction industry.

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

**SIFAT FIZIKAL, STRUKTURAL DAN MEKANIKAL KACA-SERAMIK
BERLIANG BERDASARKAN DARIPADA BUANGAN KACA SODA-KAPUR-
SILIKA DAN KULIT KERANG**

Oleh

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Pada masa sekarang, bahan buangan pepejal perbandaran semakin meningkat hingga ke tahap yang membimbangkan di seluruh negara. Negara kita sangat bergantung kepada cara pembuangan secara terbuka dan tanah timbus, walaupun kaedah ini sebenarnya menelan belanja yang besar dan juga membazirkan masa. Percambahan masalah yang berkaitan dengan pengurusan bahan buangan ini telah mengubah cara berfikir para saintis dan pengkaji dalam mencari jalan untuk menukarkan bahan buangan kepada bahan yang mampu membawa manfaat. Oleh itu, satu inisiatif telah diambil melalui kajian ini untuk menggunakan soda-kapur-silika (SLS) terbuang sebagai matriks kaca, dan sumber silikat (SiO_2) and kulit kerang (CS) sebagai agen pembuihan (pembentuk liang) untuk membentuk kaca-seramik berliang (FGC) yang ringan. Satu siri sampel FGC dengan 1, 3, 6 dan 9 wt.% CS berjaya disiapkan dengan kaedah sintering konvensional keadaan pepejal pada suhu 700, 800 dan 900 °C yang disinter selama 30, 60 dan 120 min. Ciri-ciri fizikal, structural dan mekanikal bahan-bahan mentah dan FGC telah dicirikan melalui WDXRF, TGA, ketumpatan, pengembangan linear, jumlah keliangan, XRD, FTIR, FESEM dan pengukuran kekuatan mampatan. Dari hasil WDXRF untuk mengkaji komposisi kimia bahan mentah, kaca SLS terdiri terutamanya dari SiO_2 iaitu sebanyak 71.90 wt.% manakala CS mempunyai peratusan berat CaO yang tinggi iaitu sebanyak 53.41 wt.%. Berdasarkan keputusan ini, CS didapati mengandungi 98.60 wt.% CaCO_3 . Untuk sifat struktur bahan mentah, corak XRD kaca SLS tidak menunjukkan puncak tajam berterusan atau diskrit dengan ciri luas lingkaran amorf pada sudut sekitar $2\theta = 20$ hingga 40° dan sementara itu, bentuk puncak serbuk CS menandakan pantulan fasa kristal aragonite dengan sistem kristal orthorhombik. Berdasarkan profil TGA CS, sifat termal CS menunjukkan penurunan CaCO_3 secara beransur-ansur berlaku setelah menjalani rawatan terma sekitar 860 °C dengan penurunan berat sekitar 43.56 %. Ketumpatan pukal FGC telah dipastikan mencecah ketumpatan minimum (0.525 g/cm^3)

dengan pengembangan maksima (83.98 %) pada 3 wt.% kandungan kulit kerang yang disinter pada suhu sinter dan masa pembakaran yang optimum yaitu 800 °C selama 60 min. Ketumpatan pukal meningkat apabila pengembangan linear dan jumlah keliangan berkurang sejajar dengan perkembangan kandungan kulit kerang, suhu sinter dan masa pembakaran, dimana hasilnya sepadan dengan graf mikro FESEM. Keputusan XRD dan spektrum FTIR telah menunjukkan pembentukan kristal wollastonite terjadi bermula pada 3 wt.% kandungan kulit kerang yang disinter pada 800 °C selama 30 min. Fasa pengkristalan kristal wollastonite meningkat sejajar dengan perkembangan kandungan kulit kerang, suhu pensinteran dan masa pembakaran. Aliran kekuatan mampatan adalah sepadan dengan aliran ketumpatan pukal. Prestasi tertinggi (0.78 MPa) dengan ketumpatan pukal rendah yang dapat diterima (0.612 g/cm³) dengan purata keliangan (74.81 %) telah diperoleh untuk sampel yang mengandungi 3 wt.% kulit kerang yang disinter pada suhu dan masa pembakaran yang optimum. Kesimpulannya, kekuatan mampatan meningkat sejajar dengan penurunan pengembangan linear dan jumlah nilai keliangan FGC. Keputusan keseluruhan menyimpulkan bahawa pembuatan FGC yang dihasilkan daripada kaca buangan SLS dan CS sebagai agen pembuihan, mempunyai potensi yang tinggi untuk digunakan sebagai bahan agregat konkrit dalam industri pembinaan bangunan.

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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 AND SYMBOL

GC	Glass-ceramics
FGC	Foam glass-ceramics
SLS	Soda-lime-silica
CaCO ₃	Calcium carbonate
CS	Clamshell
MS	Mussel shell
ES	Eggshell
OS	Oyster shell
SiO ₂	Silicon dioxide
CO ₂	Carbon dioxide
CaO	Calcium oxide
SiO ₄	Silicate
CaSiO ₃	Wollastonite
PVA	Polyvinyl alcohol
TGA	Thermogravimetric analysis
XRD	X-ray diffraction
WDXRF	Wavelength-dispersive X-ray fluorescence
FTIR	Fourier transform infrared
FESEM	Field emission scanning electron microscope
UTM	Universal testing machine
JCPDS	Join Committee on Powder Diffraction Standard
µm	Micrometre
mm	Millimetre

cm	Centimetre
MPa	Megapascal
Wt.%	Weight percentage
Pa.s	Pascal second
°C	Degree Celsius
h	Hour
g	Gram
g/cm ³	gram per cubic centimetre
T _g	Glass transition temperature



CHAPTER 1

INTRODUCTION

1.1 Research background

Municipal solid waste is dominated by the overall composition of solid waste in Malaysia, with the remaining composition being industrial waste, agricultural waste, and construction waste (Latifah *et al.*, 2009). In Malaysia, 80% of solid waste categorized as recyclable waste is disposed of at landfills and glass has contributed to 3 % of the total recyclable solid waste composition (Moh and Latifah, 2017). Glass is known for its ability to provide fascinating properties in products based on ceramics (Zanotto *et al.*, 2010). Glass with an acceptable chemical composition can be converted into glass-ceramics (GC) via a controlled heat treatment process (Rawlings *et al.*, 2006).

Soda-lime-silica (SLS) glass is the most common glass type and is widely used for post-consumer containers and flat glass, such as window and door glass panels (Andela and Zavitz, 2004). SLS glass is manufactured by melting silicon dioxide (SiO_2), sodium carbonate (Na_2CO_3), calcium carbonate (CaCO_3) and other minor constituents (Silva *et al.*, 2017). SLS glass's softening point temperature is slightly low compared to other silicate glass, promoting less energy usage during the sintering process. The softening temperature of SLS glass is about $\sim 700^\circ\text{C}$ and this interesting feature may enable the production of GC at a lower temperature compared to the normal temperature used to crystallize the GC product (Ayadi *et al.*, 2011).

Foam glass-ceramics (FGC) is a porous material that requires a foaming agent as a surfactant or a blowing agent to enhance foam formation. To obtain the porous material, the foaming agent usually made up of carbonaceous materials is mixed with the parent glass before any heat-treatment occurs (Silva *et al.*, 2017). The foaming agent reaction occurs close to the parent glass sintering temperature to achieve optimum performance (Rawlings *et al.*, 2006). The uses FGC from waste materials as aggregates for light-weight concrete have attracted significant interest from the construction industry because of a characteristic such as lightweight, water permeable, good compressive strength, and chemically inert and non-toxic (Da Silva Fernandes *et al.*, 2019). For instance, these factors favour their uses in thermal and acoustic insulation applications such as catalytic support for liquid metal and gas filtration, electromagnetic radiation absorbers, mineral casting, combustion engineering membranes, and lightweight bearing structure (Petrella *et al.*, 2010; Chinnam *et al.*, 2013; Chakartnarodom and Ineure, 2014; Benhaoua *et al.*, 2015).

Introducing a foaming agent or pore-forming agent into the ceramic matrix is the most common technique for the manufacture of FGC (Souza *et al.*, 2017). The foaming ability usually depends on the decomposition process of the carbonates or sulphates. Moreover, the foaming agent's particle size also affects the size of the pores (Bernado *et al.*, 2007). Various types of foaming agents had been used in previous studies, including aluminum nitride (AlN), calcium carbonate (CaCO₃), manganese dioxide (MnO₂), and silicon carbide (SiC), which have shown varying degrees of success (Llaudis *et al.*, 2009; Francis *et al.*, 2013; Tang *et al.*, 2014; Ding *et al.*, 2015).

Various research has recently been conducted using waste products from animal shell waste containing carbonated as a foaming agent including clamshells (CS). CS consists of a relatively high content of CaCO₃ composition, which is approximately 97 wt.% and is a significant replacement for pure CaCO₃ (Scarinci *et al.*, 2005). The use of CS waste in the manufacture of FGC may also be of significant environmental benefit.

From the recent research, the hypothesis that can be concluded for this study are as follows:

1. As the foaming agent content increases, the expansion and porosity formation of FGC increased.
2. As the sintering temperature increases, the density and compressive strength of FGC increased.
3. As the holding time increases, the coalescences of pores increased hence results in inhomogeneous pore distribution.

1.2 Problem statement

Nowadays, abundance of animal shells from food waste such as clamshell (CS), had been disposed of in landfill (Rahman *et al.*, 2019). CS is disposed of without considering its benefit as a natural source of CaCO₃ (Ismail *et al.*, 2016). Besides, the food waste would give a negative impact to the environment and ecosystem, consequently led to solid waste pollution. In this study, the FGC is fabricated from the SLS glass bottles and CS waste with the intention to reduce the solid waste in landfill. Moreover, there are limited reports and systematic study of the physical, structural, and mechanical properties of FGC derived from SLS glass bottles as the precursor glass and CS waste as a natural foaming agent. CS is a good candidate for a natural foaming agent because it contains about 95 to 90 % of CaCO₃ (Mo *et al.*, 2018).

In recent times, FGC have captured an attention in building and construction industries due to its properties and wide range of application. The pore characteristics of the FGC such as size, distribution, and morphology may determine its basic properties such as low density, low thermal conductivity, high surface area, permeability, high chemical resistance, and incombustibility (Binner, 2005; Scarinci *et al.*, 2005; Scheffler and Colombo, 2005). FGC has a porous structure that contributes to its light-weight characteristic. Kurpińska and Ferenc (2017) has reported that the uses of FGC as concrete aggregates has decreased the total weight of the concrete body. Light-weight concrete (LWC) has porosity with both open-pore and close-pore structures due to the addition of FGC and the porosity increases as the porosity of FGC increased (Li *et al.*, 2015). However, some consideration regarding the strength of the FGC for building materials application needs to be given to ensure the stability of the porous structure. Furthermore, there is limited report regarding the mechanical properties of FGC as it is usually used in application such as heat insulation and sound absorption (Guo *et al.*, 2020).

To address the problem, a comprehensive study on the effect of different composition of CS, sintering temperature and holding time of FGC derived from SLS glass bottles and CS waste is carried out to improve the physical, structural, and mechanical properties of the final product. The results of this study will be used to determine the potential application of FGC to be used as aggregates for lightweight concrete in building materials.

1.3 Research objectives

The main objective of this study is to fabricate and optimize the synthesis of foam glass-ceramics (FGC) derived from SLS glass and CS waste. This study involves designing suitable glass and foaming agent composition, heat treatment development, holding time progression, and a series of fundamental studies on the physical, structural, and mechanical strength of the final product. This research is conducted based on several objectives as follows:

1. To synthesize the SLS glass and CS waste as the raw material for the fabrication of FGC.
2. To elucidate the effect of different composition of CS on the formation of FGC.
3. To investigate the effect of different sintering temperature and holding time on the physical, structural, and mechanical properties of FGC.

To achieve those objectives, a series of FGC with 1, 3, 6 and 9 wt.% CS is prepared by a controlled sintering process at 700, 800 and 900 °C sintered for 30, 60 and 120 min by using the single-stage conventional solid-state method. The physical, structural, and mechanical properties of FGC have been characterized to investigate the effect of CS composition, progression of the sintering temperature, and holding time for the FGC formation. The physical, structural, and mechanical properties of FGC have been studied through

measurements such as bulk density, linear expansion, total porosity, X-ray diffraction (XRD), fourier transform infrared (FTIR), field emission scanning electron microscope (FESEM), and compressive strength using universal test machine (UTM). The main purpose of this work is to fabricate FGC from waste materials as a potential concrete aggregate material for application in building and construction.

1.4 Outline of the thesis

The thesis arrangement for the study is organized as follows. Chapter 1 presents the introduction of SLS glass and CS as raw materials, FGC, problem statements, objectives, and research scopes. Chapter 2 addresses the theory of SLS glass, foaming agent, FGC, and previous work, including past and current work. Chapter 3 describes the process, methodology, and characterization of FGC. Results on the effect of different compositions of CS content, sintering temperature development, and progression of different holding time towards the physical, structural, and mechanical properties of FGC are analyzed and discussed in Chapter 4. Ultimately, Chapter 5 provides a conclusion and recommendation for future work.

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