PHYSICAL, PHASE TRANSFORMATION AND ELASTIC PROPERTIES OF WOLLASTONITE GLASS-CERAMICS FABRICATED USING EGGSHELL AND WASTE GLASS

By

NURUL AFIQAH BINTI MOHAMAD YAMIN

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

September 2022

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DEDICATIONS

To my beloved parents, Mohamad Yamin Bin Naib and Fatimah Binti Muhamad
For their unconditional love and support

To my siblings and family
For making my life complete

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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Chair : Mohd Hafiz Bin Mohd Zaid, Phd
Faculty : Science

Wollastonite, also widely recognized as calcium silicate ($CaSiO_3$), has received extensive research due to its numerous application such a tiles and cement. A lot of attention has been paid recently to the physical characterization, transformation of phases, and elastic wollastonite glass-ceramics properties. The main aims of this research are to fabricate wollastonite glass-ceramics from waste products and to study the physical, structural, and elastic properties of wollastonite glass-ceramics. A series of glass with combine composition derived from ES–ZnO–B_2O_3–SLS and classified as EZBSLS glasses were prepared via melt-quenching method with empirical formula, *x*(ES)–5ZnO–10B_2O_3–100-*x*(SLS) where x = 15, 20, 25, and 30 wt.%. The wollastonite glass-ceramics were originated from the parent glasses by a controlled heat-treatment process at various temperatures of 700, 800, 900 and 1000 °C at 2 hours holding time. The detail of chemical composition of ES and SLS glass was discovered by using energy dispersive X-ray fluorescence (EDXRF). The results indicated that the major elements in SLS glass was SiO_2 with 70.5 wt.%. Meanwhile, for ES, the main element composed of CaO with 96.8 wt.% which confirms that the SLS glass waste and ES can be used as SiO_2 and CaO source. Archimedes method was used to measure bulk density of EZBSLS glasses and wollastonite glass-ceramics. Meanwhile, the molar volume of the samples were calculated by using formula from the molecular weight of the atom divided by the density of the sample. Based on the result, the bulk density of the EZBSLS glasses was increased from 2.684 to 2.779 g/cm^3 with the increasing of ES content. Furthermore, the density of the wollastonite glass-ceramics was also increased along with the advancement of heat-treatment temperature and the highest density referred to ELZBSLS4 at 1000 °C which is 2.843 g/cm^3. The structural properties of EZBSLS glass and wollastonite glass-ceramics samples were determined by X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), and Fourier Transform Infrared (FTIR) Spectroscopy. The XRD result revealed no peak appeared proving that

the EZBSLS glasses are fully amorphous in structure. For wollastonite glass-ceramics, the analysis showed the wollastonite crystal phase started to grow at the heat-treatment temperature of 800 °C and the peak intensity linearly increased with the increment of ES content and heat-treatment temperatures. From the result, the intense peak of wollastonite crystal phase (JCPDS 84-654) was detected at 900 °C with the optimum 25 wt.% ES content. FTIR reflection spectroscopy was the used to assess structural of glass and wollastonite glass-ceramics in the range 400 – 4000 cm^{-1}. The presence of several types of vibration such as Ca–O, Si–O–Si, and the detection of Ca-O-Si bands in FTIR measurement methods confirms the formation of wollastonite crystal phase in the EZBSLS glass matrix. Furthermore, the microstructure of wollastonite glass-ceramics was analyzed at 900 °C and the 25 wt.% ES sample showed an early stage of homogenous distribution in uniform shape of wollastonite crystal. Next, the EZBSLS glasses and wollastonite glass-ceramics were analyzed by their elastic properties by non-destructive ultrasonic velocity testing. As can be concluded that EZBSLS3 heat-treated at 900 °C is the most stable and optimal with value of bulk and Young’s modulus are 167.538 and 143.572 GPa.

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

CIRI-CIRI FIZIKAL, TRANSFORMASI FASA DAN SIFAT ELASTIK SERAMIK KACA WOLLASTONITE DENGAN MENGGUNAKAN KULIT TELUR DAN GELAS TERBUANG

Oleh

NURUL AFIQAH BINTI MOHAMAD YAMIN

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Wollastonite, juga dikenali sebagai kalsium silikat ($CaSiO_3$), telah dikaji secara meluas kerana aplikasinya yang luar biasa sebagai contoh jubin dan simen. Baru-baru ini, banyak pemerhatian yang telah diberikan kepada pencirian secara fizikal, transformasi fasa, dan sifat keanjalan wollastonite kaca seramik. Objektif utama penyelidikan ini adalah untuk menghasilkan wollastonite kaca seramik daripada bahan buangan dan menganalisis sifat fizikal, struktur dan keanjalan wollastonite kaca seramik. Satu siri kaca dengan komposisi gabungan daripada ES–ZnO–B_2O_3–SLS dan dikelaskan sebagai gelas EZBSLS telah disediakan melalui kaedah lindapan leburan dengan mengunakan formula empirik, x(ES)–5ZnO–10B_2O_3–100-x(SLS) dengan nilai x= 15, 20, 25, dan 30 wt.%. Seramik kaca wollastonite diperoleh daripada kaca induk melalui proses rawatan haba terkawal pada suhu berbeza 700, 800, 900 dan 1000 °C selama 2 jam. Perincian komposisi kimia kaca ES dan soda-lime-silika (SLS) ditentukan dengan menggunakan pendarfluor sinar-X penyebaran tenaga (EDXRF). Keputusan menunjukkan bahawa unsur utama dalam kaca SLS ialah SiO_2 dengan 70.5 wt.%. Manakala, bagi ES, unsur utama terdiri daripada CaO dengan 96.8 wt.%. Ketumpatan pukal gelas EZBSLS dan seramik kaca wollastonite diukur dengan kaedah Archimedes. Manakala isipadu molar sampel dikira menggunakan formula daripada berat molekul atom dibahagi dengan ketumpatan sampel. Berdasarkan keputusan, ketumpatan pukal gelas EZBSLS telah meningkat daripada 2.684 kepada 2.779 g/cm^3 dengan peningkatan kandungan ES. Tambahan pula, ketumpatan seramik kaca wollastonite juga meningkat dengan perkembangan suhu rawatan haba dan ketumpatan tertinggi dirujuk kepada ELZBSLS4 pada 1000 °C iaitu 2.843 g/cm^3. Ciri-ciri struktur sampel kaca EZBSLS dan wollastonite kaca seramik ditentukan oleh Pembelauan Sinar-X (XRD), Mikroskopi Elektron Pengimbasan

Pancaran Medan (FESEM), dan Spektroskopi Inframerah Transformasi Fourier (FTIR). Keputusan XRD mendedahkan tiada puncak yang muncul membuktikan bahawa kaca EZBSLS adalah struktur amorfus sepenuhnya. Bagi seramik kaca wollastonite, analisis menunjukkan fasa kristal wollastonite mula berkembang pada suhu rawatan haba 800°C dan keamatan puncak meningkat secara linear dengan kenaikan kandungan ES dan rawatan haba. Daripada hasilnya, puncak sengit fasa kristal wollastonite telah dikesan pada 900 °C dengan optimum 25 wt.% kandungan ES. Spektroskopi pantulan FTIR digunakan untuk menilai struktur kaca dan seramik kaca wollastonite. Kehadiran beberapa jenis getaran seperti ikatan Ca–O, Si–O–Si dan Ca–O–Si yang dikesan daripada pengukuran FTIR menunjukkan pembentukan fasa kristal wollastonite dalam matriks kaca EZBSLS. Struktur mikro seramik kaca wollastonite dianalisis pada suhu 900 °C dan EZBSLS3 menunjukkan peringkat awal pengedaran sekata dalam bentuk seragam kristal wollastonite. Seterusnya, kaca EZBSLS dan seramik kaca wollastonite dianalisis melalui ujian halaju ultrasonik yang tidak merosakkan. Seperti yang boleh disimpulkan bahawa EZBSLS3 yang dirawat haba pada 900 °C adalah yang paling stabil dan optimum dengan nilai moduli adalah 16.957 GPa167.538 and 143.572 GPa.

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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:

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LIST OF ABBREVIATIONS

$CaSiO_3$	Wollastonite
EZBSLS	ES–B_2O_3–ZnO–SLS
SLS	Soda lime silica
SiO2	Silica oxide
ZnO	Zinc oxide
CaO	Calcium oxide
Na_2O	Sodium oxide
Al_2O_3	Aluminium oxide
K_2O	Potassium oxide
MgO	Magnesium oxide
Fe_2O_3	Ferric oxide
B_2O_3	Boron trioxide
α	Alpha
β	Beta
EDXRF	Energy dispersive X-ray fluorescence
XRD	X-Ray diffraction
FTIR	Fourier transform infrared
FESEM	Field emission scanning electron microscopy

CHAPTER 1

INTRODUCTION

1.1 Research Background

The building sector is among the most important roles in this world for the past few decades. The development and economic growth in this world mainly comes from this industry so it is important to sustain the construction industry and continue its calibre and product. Regrettably, as the modern era progresses, the industrialization produces million tons of waste such as plastic, concrete, glass bottles, and paper (Blair & Mataraarachchi, 2021). This concern has prompted a call for a solution to the environmental problem via true treatment and disposal management.

The annually report by Malaysia Municipal Solid Waste (MSW) needs the disposal of approximately 98% of the total waste in landfills come from industrial and food waste (Sipra et al., 2018; Michel et al., 2021). The current landfill disposal method requires advancements in order to extend landfill life and reduce the severity of land scarcity. Rapid industrialization and Malaysian growth necessitate a better and more efficient waste management strategy. Increased urbanization and rural-urban migration have expanded per capita earnings leading to variations in consumption patterns, which have resulted in massive waste generation. The research involves the collection of waste generation information from Malaysian municipalities. The study also supports waste composition analysis based on income level to ascertain patterns in the composition produced (Hoang et al., 2020). However, this approach appears to cause numerous environmental issues, including improper disposal, the waste of large amounts of impurities, and the need for new land for the founding of disposal sites (Oyedotun et al., 2021).

Wollastonite ($CaSiO_3$), also known as calcium silicate, has properties that make it suitable for use in biomedical applications. Wollastonite have excellent bioactivity and degradability/resolvability and thereby could be applied in hard tissue repair or as 3D scaffolds for tissue engineering. Furthermore, wollastonite has also been recognized as a bioactive material with potential applications in bone tissue engineering (Hoang et al., 2020). The previous research has been reported that the commercial bone tissue can be developed on wollastonite with addition of hydroxyl carbonated apatite layer deposition. Moreover, these types of ceramics can promote the connection, proliferation, and differentiation of human bone-derived cells (Palakurthy et al., 2020).

Recently, wollastonite glass-ceramics derived from binary $CaO–SiO_2$ glass system has been attract many researchers for it interesting properties as material used in building applications. The utilization of industrial waste in the manufacture of construction material, concrete, and cement considered to be an effective method of preventing environmental issues, lowering production costs, and conserving energy (Tabit et al., 2020). Wollastonite glass-ceramics is useful in a construction industry and other applications due to a variety of advantageous properties including low shrinkage, good strength, lack of volatile constituents, body permeability, fluxing characteristics, whiteness, and acicular shape and the stable rise worldwide in recent years alludes to the market growth for wollastonite glass-ceramics (Ponsot et al., 2015).

In the recent study, a series of precursor $ES–ZnO–B_2O_3–SLS$ glass system and symbolized as EZBSLS glasses are prepared using the conventional melt-quenching approach. The wollastonite glass-ceramics are derived from EZBSLS glasses by the controlled heat-treatment process. The EZBSLS glasses and wollastonite glass- ceramics were fabricated by different weight percentage of ES content which are 15, 20, 25, and 30 wt.% and were symbolized as EZBSLS1, EZBSLS2, EZBSLS3, and EZBSLS4 glass samples. The properties of the EZBSLS glass and wollastonite glass-ceramics have been characterized for physical, structural, and elastic behaviour intending to investigate the effect of ES addition and heat-treatment temperature to the formation and properties of wollastonite glass-ceramics. The structural and phase transformation of EZBSLS glass and wollastonite glass-ceramics have been studied by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), and fourier transform infrared (FTIR). Besides, the elastic properties of EZBSLS glass and wollastonite glass-ceramics have been researched by non-destructive ultrasonic velocity testing. Given the foregoing, the goal of this work is to fabricate and characterize wollastonite glass-ceramics as potential materials for use in building materials applications.

1.2 Problem statement

These days, there are a massive amount of solid wastes piled up at dumping ground. The waste of food accounts for 49.3% of all solid waste as people around the world that have been consumed on a daily basis (Moh & Abd Manaf, 2014). In general, food waste has a harmful effect on the environment, which has resulted in solid pollutant. In this study, the fabrication of wollastonite glass-ceramics are made from waste materials which are soda-lime-silica (SLS) glass and eggshell (ES) wastes to be able to solve the environmental concerns. ES is used as a CaO source because its contain about 97% of $CaCO_3$ and this carbonate is degraded to CaO and release CO_2 gas after heating at 800 °C (Commey & Mensah, 2019).

Further to that, it is stated that zinc oxide (ZnO) can reconstruct the atom arrangement in glass system to enhance the rigidity as well as contribute to the glass network rigidity (Rammah et al., 2020). Furthermore, the inclusion of B_2O_3 into the glass system can help to lower the melting point of the materials (Turkeman et al., 2016). However, there are few reports and comprehensive studies on the phase transformation and elastic properties of wollastonite glass-ceramics derived from ES–ZnO–B_2O_3–SLS glass system. This is due to the fact that from the previous study, there is no addition of ZnO and B_2O_3 in the glass system.

Moreover, scientist are focusing to expand novel technique and utilizing the starting materials from the waste product for producing wollastonite as a result of which low energy processes may be formed. Commonly, in industry conventional solid-state methods for commercial building materials are fully developed. Referring to this method; natural raw materials for example CaO, ZnO, B_2O_3, and SiO_2 are thoroughly mixed and fired at extremely high temperatures for a few hours due to the high melting point between 1450-1600 °C of starting materials (Zaid et al., 2016). Hence, the inclusion of B_2O_3 in the glass system would be an assistance to lower the melting temperature of the starting materials (Turkeman et al., 2016).

Besides, the poor mechanical and elastic properties of wollastonite glass-ceramics (25 MPa for tile installation) also still being discussed nowadays (Joy et al., 2019). To resolve this issue, fabrication of wollastonite glass-ceramics utilizing waste material including SLS glass as a SiO_2 source and ES as CaO source are developed via the melt-quenching approach. Besides, the addition of ZnO and B_2O_3 in the parent glass are expected to enhance the elastic and mechanical properties of the glass-ceramics.

Based on Hossain et al., 2019, they fabricated the wollastonite glass-ceramics from eggshell and rice husk ash and the obtained results show that it reduced the physical and mechanical properties of tile samples at low sintering temperature but at high temperature, mechanical properties do not have significant influence. This due to the absence of other oxides that can helps to enhance the mechanical properties of the glass-ceramics such as ZnO.

This glass evolved into glass-ceramics with $CaSiO_3$ as the primary crystal phase. Wollastonite has a promising future in advanced materials as a building material applications and biomedical applications due to its strength and structure of the glass ceramics. As a result, a detailed analysis of the crystallization, properties, and impact of heat-treatment on wollastonite glass-ceramics obtained from the EZBSLS glass system is being conducted, with the hopes of discovering promising materials for use in building applications.

1.3 Research objectives

This project's primary objectives are to synthesis and optimize wollastonite-based glass-ceramics derived from EZBSLS glasses. This work included the development of suitable glass compositions, melt-quenching, the advancement of the ES/SLS ratio process, and a sequence of fundamental research of the crystallization process. The objectives are as follows:

1. To fabricate wollastonite glass-ceramics derived from ES–B_2O_3–ZnO–SLS (EBZSLS) glasses using SLS glass waste and eggshells as a silica and calcium source.
2. To analyze the effect of ES/SLS ratio to the physical, phase transformation and elastic properties of EBZSLS glass and wollastonite glass-ceramics.
3. To investigate the effect of different heat-treatment temperature to the physical, phase transformation and elastic properties of wollastonite glass-ceramics derived from ES–B_2O_3–ZnO–SLS.

1.4 Scope of Study

The scopes of the study are as follows in effort to accomplish the study's objectives:

1. Series of precursor glass based on x(ES)–5ZnO–10B_2O_3–100-x(SLS) where x = 15, 20, 25, and 30 wt.% have been prepared using SLS glass powder and eggshell powder with constant ZnO and B_2O_3 powder by conventional melt- quenching technique. Based on previous research by Elsayed et al., 2019, the standard ratio of wollastonite glass-ceramics is 52:48. Hence, the nominal and optimum composition that has been used is similar to what I used in this research.
2. The chemical composition of the ES and SLS glass system has been measured using EDXRF spectroscopy to prove the chemical oxide percentage in the starting materials.
3. The glass transition temperature (T_g) and glass crystallization temperature (T_c) has been measured using DSC.
4. Wollastonite glass-ceramics have been derived from the precursor ES–ZnO– B_2O_3–SLS glass system by a controlled heat-treatment process. The heat-treatment temperature chosen were 700, 800, 900 and 1000 °C. As reported by Almasri et al., 2017, the wollastonite nucleation phase begins at 800 until 1000 °C. However, the sample will melt when the temperature exceeds 1000 °C and started to change the shape of the sample. Thus, no result can be obtained from the melted sample.

5. The physical, phase transformation and elastic properties of EZBSLS glass and wollastonite glass-ceramic have been analyzed using the Archimedes method, DSC, EDXRF, XRD, FESEM, FTIR, and ultrasonic testing.

1.5 Important of the study

Wollastonite active ingredients reduce ceramic firing temperatures, improve strength, and reduce firing and drying shrinkages and temperature parameters. Wollastonite ceramics have been used in electrical and radio engineering (Sahar et al., 2020). In addition, wollastonite also has promising applications in the ceramics sector (Fuertes et al., 2022). Silicate glass-ceramic containing wollastonite ($CaSiO_3$) crystals have received a great deal of attention due to their good bioactivity, biocompatibility and mechanical properties (Kherifi et al., 2021). These glass-ceramics strengthen bone regeneration and can be applied in bone tissue engineering (Ribas et al., 2019). The wollastonite crystalline phase often presents acicular morphology, which potentially increases the toughness and strength of silicate glass-ceramics (Soares et al., 2018).

Furthermore, the comprehensive use of natural products in the ceramic tile sector has increased in a troubling shortage of these natural resources. Tile industries provide empowering climates for the appropriate waste power stations in this regard (Almeida et al., 2016). As a result, many researchers are seeking to create new compositions for the production of tiles from different type of waste. A wide range of waste materials, including rice husk ash, ceramic waste, and glass waste have been investigated for production of tile (Kim et al., 2016; Tarhan et al., 2016; Miou et al., 2020).

As a conclusion, the significant of the study is the elastic properties which is the hardness of the sample. It can be conclude that the optimum composition of the sample is at 30 wt.% heat-treated at 800 °C. In this work, the best hardness obtained is 16.957 GPa compared to previous study which is 25 MPa. Therefore, the wollastonite glass-ceramics derived from ES–B_2O_3–ZnO–SLS has improved the strength and hardness of the sample compared to previous work.

1.6 Outline of thesis

The thesis framework is structured as follows. Chapter 1 gives an introduction of precursor EZBSLS glass and wollastonite glass-ceramics, the problem statements, the objectives, the scopes and also the importance of this study. Chapter 2 discusses glass theory, glass-ceramics, and past research, both past and current, carried out by other research teams. Chapter 3 describes the apparatus, methodology, and characterization of the precursor glass and wollastonite glass-ceramics. In Chapter 4, the effects of the ES/SLS glass ratio, the advancement of heat treatment temperatures, and the physical, structural, and elastic properties of precursor EZBSLS glass and wollastonite glass- ceramics are investigated and discussed. Finally, in Chapter 5, the conclusion and future work suggestions are clarified.

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