UNIVERSITI PUTRA MALAYSIA

FABRICATION AND CHARACTERIZATION OF GLASS IONOMER CEMENT USING ALUMINO-SILICATE-FLUORIDE BASED-GLASS CERAMICS FROM WASTE RESOURCES

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By

ROHANIAH BINTI ABDUL JALIL

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

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DEDICATIONS

To my beloved parents, Abdul Jalil bin Ismail and Mainah binti Osman for their unconditional love and support.

To my family and siblings, Abdul Hafiz, Hairatul Nisak, Mohamad Arif, Mohd Razif and Nurul Humairah 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 a lot throughout this journey. Without all of them, this success would not be mine.

May the world soon recover from Covid-19.

Thank you

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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Chairman: Khamirul Amin bin Matori, PhD
Faculty : Science

The success of Glass Ionomer Cement (GIC) innovations in dentistry has attracted interest among researchers over the last 40 years. However, there is limited research regarding the usage of Alumino-Silicate-Fluoride (ASF) based glass ceramics derived from waste resources in the fabrication of GIC. This research's main focus is to fabricate GIC using ASF-based glass ceramics derived from waste resources of clam shell (CS) and soda lime silica (SLS) glass. Two batches of ASF glass ceramics sample labeled as Batch 1 (B1) and Batch 2 (B2) were prepared following the empirical formula of [(x)CS·(45-x) SLS·15CaF_2·20P_2O_5·20Al_2O_3] where x = 5 and 20 (wt.%). Both batches have a different composition in SLS and CS which will be studied in this work. The ASF based glass ceramics were synthesized by using conventional melt quenching techniques. Then, GIC was fabricated using three main components of ASF based glass ceramics, polyacrylic acid (PAA) and water. The thermal, physical, structural, chemical and mechanical properties of ASF glass ceramics and GIC had been determined by using X-ray fluorescence (XRF), differential scanning calorimetry (DSC), density measurement, X-ray diffraction (XRD), fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX) and compressive strength test (CST). The XRF showed the largest percentage of SLS glass elements is silicon dioxide (SiO_2) around 79 % while CS has 99.52 % of calcium oxide (CaO), which makes them suitable as a source for ASF glass composition. The DSC showed glass transition temperature, T_g around 764-785 °C and crystallization temperature, T_c around 918 - 986 °C. The density measurement of GIC showed an increasing pattern from 600 - 800 °C. Then, the density decreased at a high sintering temperature of 1000 - 1200 °C. This is due to the formation of anorthite ($Ca(Al_2Si_2O_8)$) and mullite ($Al_5SiO_{9.5}$) at high sintering temperatures. The XRD analysis revealed fluorapatite ($Ca_5(PO_4)_3F$) as a major phase in GIC samples which decomposed into anorthite and mullite at 1000 - 1200 °C. Next, FTIR revealed the presence of CO_3 group, Si–O–Si, P–O, crystalline phosphate, CH_3 and O–H band which indicates the structure of the glass matrix and the crystallization of the GIC sample. The uniform spherical microstructure was observed, which converted to a coarse structure at high sintering temperatures in FESEM due to devitrification. The EDX analysis revealed a Ca/P ratio obtained around 1.17 - 3.49 is suitable for body implantation. CST has the same pattern as the density measurement of GIC. The decline of the pattern was attributed to the decomposition of fluorapatite into mullite and anorthite. A selected sample of B1 GIC at 800 °C and 28 days was discovered as an optimum result due to the highest CST value achieved, 82.03 MPa. In conclusion, GIC shows a potential candidate for clinical applications in dentistry as their mechanical properties completed the requirement for dental application (70 MPa) following the International standard organization (ISO) 9917.

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

FABRIKASI DAN PENCIRIAN SIMEN IONOMER KACA MENGGUNAKAN KACA SERAMIK-BERASASKAN ALUMINO-SILIKAT-FLORIDA DARIPADA SISA BUANGAN

Oleh

ROHANIAH BINTI ABDUL JALIL

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Kejayaan inovasi simen ionomer kaca (GIC) dalam bidang pergigian telah menarik minat para penyelidik sejak 40 tahun yang lalu. Walau bagaimanapun, terdapat kajian yang terhad mengenai penggunaan kaca seramik berasakan Alumino-Silikat-Florida (ASF) yang berasal dari sumber buangan dalam pembuatan GIC. Fokus utama penyelidikan ini adalah untuk membuat GIC menggunakan kaca seramik berasaskan ASF yang berasal daripada sumber buangan seperti kulit kerang (CS) dan sisa kaca silika (SLS). Dua kumpulan sampel kaca seramik ASF yang dilabelkan sebagai Kumpulan 1 (B1) dan Kumpulan 2 (B2) telah disediakan mengikut formula empirik [(x) CS (45-x) SLS $15CaF_2$ $20P_2O_5$ $20Al_2O_3$] di mana x = 5 dan 20 (% berat). Kedua-dua kumpulan mempunyai komposisi yang berbeza dari segi CS dan SLS yang akan dikaji dalam kajian ini. Kaca seramik berasaskan ASF disintesis menggunakan teknik lebur konvensional. Kemudian, GIC dibentuk menggunakan tiga komponen utama iaitu kaca seramik berasaskan ASF, asid poliakrilik (PAA) dan air. Sifat terma, fizikal, struktur, kimia dan mekanikal kaca seramik ASF dan GIC ditentukan menggunakan pendarfluor sinar-X (XRF), kalorimetri pembezaan imbasan (DSC), pengukuran ketumpatan, pembelauan sinar-X (XRD), spektroskopi transformasi fourier inframerah (FTIR), mikroskop pengimbas pelepasan medan elektron (FESEM), tenaga penyebaran sinar-X (EDX) dan ujian kekuatan mampatan (CST). XRF menunjukkan peratusan unsur terbesar dalam kaca SLS adalah silikon dioksida (SiO_2) iaitu sekitar 79 % sementara CS mempunyai 99.52 % kalsium oksida (CaO) yang menjadikannya sesuai sebagai sumber komposisi kaca ASF. DSC menunjukkan suhu peralihan kaca, T_g sekitar 764 - 785 °C dan suhu penghabluran, T_c sekitar 918 - 986 °C. Pengukuran ketumpatan GIC menunjukkan peningkatan corak dari 600 – 800 °C. Kemudian, ketumpatannya menurun pada suhu pensinteran tinggi 1000 - 1200 °C. Ini disebabkan oleh pembentukan *anorthite* ($Ca(Al_2Si_2O_8)$) dan *mullite* ($Al_5SiO_{9.5}$) pada suhu pensinteran yang tinggi. Analisis XRD

mendedahkan *fluorapatite* ($Ca_5(PO_4)_3F$) menjadi fasa utama dalam sampel GIC telah terurai menbentuk *anorthite* dan *mullite* pada suhu 1000 - 1200 °C. Seterusnya, FTIR mendedahkan kehadiran kumpulan CO_3, Si–O–Si, P–O, fosfat kristal, CH_3 dan O–H yang merujuk kepada struktur matriks kaca dan pengkristalan sampel GIC. Struktur mikrosfera diperhatikan berubah menjadi struktur kasar pada suhu pensinteran tinggi dalam FESEM disebabkan proses divitrifiksi. Analisis EDX mendedahkan nisbah Ca/P yang diperoleh adalah sekitar 1.17 - 3.49 sesuai untuk implantasi badan. CST mempunyai corak yang sama dengan pengukuran ketumpatan GIC. Pola menurun diperolehi kerana penguraian *fluorapatite* menjadi *mullite* dan *anorthite*. Sampel terpilih B1 GIC pada suhu 800 °C dan 28 hari menjadi hasil optimum kerana nilai tertinggi CST telah diperolehi, 82.03 MPa. Kesimpulannya, GIC merupakan calon yang berpotensi untuk aplikasi klinikal dalam pergigian kerana sifat mekanikalnya memenuhi syarat untuk aplikasi pergigian (70 MPa) mengikut organisasi standard antarabangsa (ISO) 9917.

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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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- the research conducted and the writing of this thesis was under our supervision;
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LIST OF ABBREVIATIONS AND SYMBOLS

Al_2O_3	Aluminium oxide
ASF	Alumino-silicate-fluoride
$CaCO_3$	Calcium carbonate
CaO	Calcium oxide
CaF_2	Calcium fluoride
CS	Clam shell
CST	Compressive strength test
DSC	Differential scanning calorimetry
EDX	Energy dispersive x-ray
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy
GIC	Glass ionomer cement
Na_2CO	Sodium carbonate
Na_2O	Sodium oxide
PAA	Polyacrylic acid
P_2O_5	Phosphorus pentoxide
SiO_2	Silicon dioxide
SLS	Soda lime silica glass
SrO	Strontium oxide
XRD	X-ray diffraction
XRF	X-ray fluorescent
ZnO	Zinc oxide
°C	Degree celcius
h	Hour
min	Minute
mm	Milimetre
MPa	Megapascal
M_w	Molecular weight
r.p.m	Revolutions per minute
λ	Wavelength
µm	Micrometre
θ	Angle between the incident and lattice plane
ρ	Density
σ_c	Compressive strength
r	Radius of the cements cylinder
T_g	Glass transition temperature
T_c	Crystallization temperature
T_m	Melting point temperature
wt.%	Weight percentage

CHAPTER 1

INTRODUCTION

1.1 Research Background

Biomaterials can be classified into two groups which are natural and artificial products. The significant role of biomaterials is to restore the biological function of the body acceptably and reliably (Williams, 1987). The vital objective of biomaterials is to enhance individual health by keeping the function of living tissues and organs in the body (Ramakrishna et al., 2001). Furthermore, the important terminologies relating to the study of biological functions are biomaterials and biocompatibility. The biomaterials must be biocompatible to work well in the biological system. Biomaterials also have excellent features such as bioactivity, biodegradability, bioinert and biostability behavior to become an ideal candidate in the medical field (Hench et al., 1991). Some biomaterial categories consist of polymers, metals, composites and ceramics (bioglass) are shown in Figure 1.1.

The polymers are known for their unique property, which has good composition flexibility. However, low mechanical strength causes the polymers to not be able to withstand the stress needed in various applications. Next, metals have good ductility, high strength and wear resistance. Still, they also have low biocompatibility, faster corrosion rate and high release of metal ions, leading to allergic issues (Eliaz, 2019). Meanwhile, composites become potential candidates in biomaterials as they consist of high elastic strength but short-term durability. Generally, ceramics is well known as biocompatible material with good corrosion and compression resistance, but it has low fracture, brittle, and high density (Kang and Fang, 2018). Above all, polymer scaffold uses in bone regeneration is challenging and very limited as it consists of low mechanical strength (Fu et al., 2011; Basha et al., 2015). However, scaffolds made from bioceramics or calcium phosphate such as bioactive glass have been shown to provide good mechanical strength (Kaur et al., 2014).

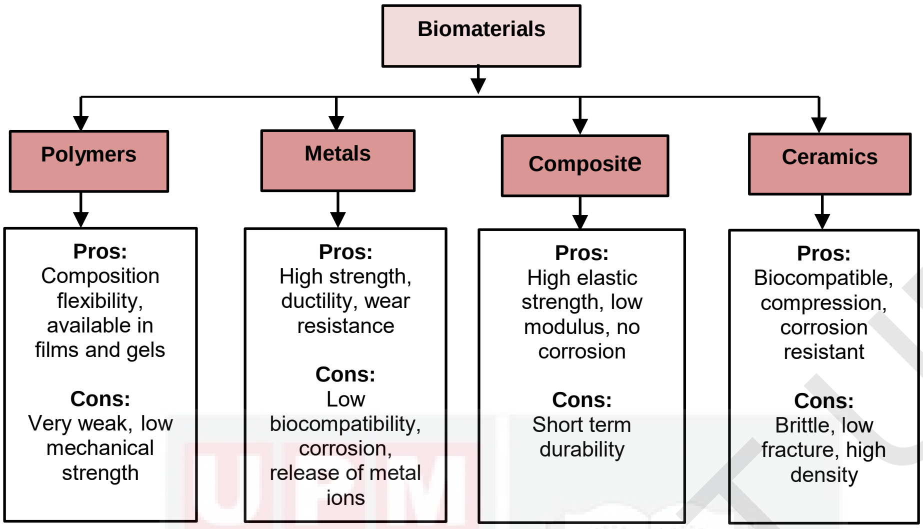

Figure 1.1: The schematic diagram of biomaterials classification (Kaur et al., 2014).

After the invention of biomaterials, many clinical applications were developed with similar functions to biological systems. Glass ceramics and bioactive glass have been widely used in medicine and researches in tissue engineering on both materials are still in progress. This is due to their unique nature, in which their composition can be modified by following the specifications required (Ducheyne et al., 1999). The primary bioactive glass system, which consists of SiO_2-Na_2O-CaO-P_2O_5, was created by Hench and a co-worker in 1969.

The bioactive glasses, which are also known as bioglass consisting of selected glass compositions, provide good biocompatibility and adhesion to the human bone (Lin et al., 1991). The first bioglass discovery known as 45S5 bioglass consists of 45 % SiO_2, 24.5 % Na_2O, 24.4 % CaO and 6 % P_2O_5 commercialized for an industrial purpose (Fernandes et al., 2018). The selection of constituent elements in the bioglass composition is similar to natural minerals in the body, such as Si, Na, O, H, Al, Ca and P (Wallace et al., 1999). Additionally, the molecule proportion between Ca and P mimics the mineral found in the bone, which is $Ca_5(PO_4)_3OH$ or hydroxyapatite (Denry and Holloway, 2014; Erasmus et al., 2017).

The compositional ranges of bioglass consisting of SiO_2, CaO, Na_2O and P_2O_5 present in the specific percentage provide bonding properties to the tissue system. The implantation of bioglass in the tissue's surface will produce silica-Ca/P gel layer, which mineralizes the biological system (Loy et al., 2017). There are three essential components of bioglass composition, which are a low percentage of SiO_2 around 60 %, high content of CaO/Na_2O and a high ratio of CaO/P_2O_5. The presence of SiO_2/Al_2O_3, CaO/P_2O_5 and Na_2O as a glass former, glass modifier and fluxing agent are included in the glass system, respectively (Bansode and Sakharkar, 2015). These components produce high surface reactivity when the bioglass has a contact in an aqueous solution.

Moreover, various bioglass compositions are developed in addition to new elements such as F, Mg, Sr, Fe and Ag (Hoppe et al., 2011). These elements are added to enhance the properties of the bioglass which is available in the market. The bioglass has been used clinically in dental applications such as endodontic and dentistry treatments (El-Meliegy and Noort, 2011). In the last decade, many improvements have been made to meet market trends and customer needs.

The chemical structure of bioglass shows an amorphous arrangement, while glass ceramics exhibit crystallized order of arrangement. The formation of glass ceramics results from the sintering of glass at specific time duration, heating temperature and under a controlled atmosphere (Kaur et al., 2014). The fabrication of glass ceramics is believed to produce high flexural strength and excellent mechanical properties relating to their hardness, toughness and viscous behaviour. This is due to the existence of a crystalline phase inside the amorphous phase of the glass structure. The crystallization process also affects the bioactivity of the glass, which turns the glass to be more inert (Filho et al., 1996). The previous studies of Clupper and co-workers showed that the crystalline growth decreases the kinetic reaction of the glass but does not disturb the formation of hydroxyapatite, which acts as an indicator of glass bioactivity (Clupper et al., 2003).

On the other hand, ASF based glass ceramics become an example of the next generation of 45S5 bioglass. In this research, the ASF based glass ceramics become the basic glass for the preparation of the dental application, which is GIC (Rahman et al., 2019). The selection of ASF glass in the biomedical application is due to its good bioactivity, high biocompatibility and excellent mechanical properties to the biological system (Wolfe and Boyde, 1992).

Besides, ASF glass will form an apatite layer and excellent chemical bonding to the tissues' surface. The apatite layer, which is known as ($Ca_{10}(PO_4)_6F_2$) or fluorapatite has similarities in the crystallographic and chemical structure of hydroxyapatite in the human body (Denry and

Holloway, 2014). The sintering process of the ASF glass also produces other apatite phases such as anorthite ($Ca(Al_2Si_2O_8)$) and mullite ($Al_5SiO_{9.5}$) (Jusoh et al., 2019). Both phases can enhance the properties of the glass ceramics (O'Donnell et al., 2010).

Due to their unique properties in the biological system, ASF based glass ceramics have been used as the glass components in the GIC. The GIC was produced between ASF glass powder and polyalkenoic acid under an acid-base reaction through the polymerization process (Thoo et al., 2013). Generally, the GIC is well-known in restorative dentistry as a tooth-colored material, which had been discovered by Wilson and Kent in 1969 (Sidhu, 2011). The usability of GIC in various clinical applications is due to their excellent function in modification of the physical properties through the alteration of glass powder/liquid ratio and chemical formula (Nicholson, 1998). Additionally, GIC is more attractive in esthetic terms than other restorative materials due to their color, similar to the tooth structure (Davidson, 2006). Moreover, fluoride content in their composition has been known to exhibit anticarcinogenic properties, high biocompatibility and adhesion to the mineral tissues (Pelka et al., 1996; Xie et al., 2000).

In this research, the uses of waste were introduced to minimize the usage of pure materials such as SiO_2 and CaO. Hence, this is to reduce the cost of the sample preparation. Additionally, the waste becomes a potential raw material for industrial purposes due to beneficial elements such as carbon, calcium and oxygen in their composition. Examples of waste materials that have been used in this research are SLS glass and CS. The SLS glass contains a high percentage of silica, making it suitable as a starting raw material in the production of glass ceramics (Zaid et al., 2011). From the previous studies, a high percentage of calcium carbonate ($CaCO_3$) had been reported in the composition of CS, which is around 99 % of their weight percentage (Ruiz et al., 2009). Thus, CS becomes a potential CaO source from waste to prepare the glass (Rahman et al., 2019).

1.2 Problem Statements

The implementation of pure calcium and silica in ASF based glass ceramics lead to high fabrication cost due to their expensive price. The CS and SLS glass from the waste material are utilized in this study to reduce the fabrication cost of GIC. Thus, preserve nature from pollution through the recycling of waste. Besides, due to the high percentage of calcium and silica content for each CS and SLS, it might contribute to some particular phases in the ASF composition (Rahman et al., 2019). Thus, the suitability of CS and SLS in the GIC application needs to be studied in this research.

Also, the utilization of waste in the GIC fabrication is limited in research. Hence, this study is conducted to analyze the effectiveness of waste to the GIC.

The utilization of ASF based glass ceramics into the GIC is genuinely introduced, and there is limited research regarding the ceramics usage in GIC. Sintering of ASF glass had contributed to the growth of various microstructure and crystalline phases, which contribute to dental application such as mechanical improvement (Jusoh et al., 2019). The growth of microstructure in various sintering temperatures is believes have an impact on the GIC. Thus, the phase transformation changes during the sintering process of ASF based glass ceramics were investigated in this study.

GIC has excellent chemical and physical properties with unique adhesion characteristics into the biological environment (Singh et al., 2011). The only major limitations of GIC are low fracture and poor mechanical properties, which limits their use in the dentistry application (Yip et al., 2001; Nagaraja and Kishore, 2005; Khiri et al., 2020). Many researches had been made to fabricate an advanced GIC with enhancement in their physical, durability, toughness and mechanical properties (Culbertson, 2001). Several methods have been introduced to improve the strength of mechanical properties in GIC, such as the incorporation of ceramics, metal particles and glass fibers (Wilson and McLean, 1988). Thus, the incorporation of ASF based glass ceramics into the GIC was investigated in this study.

The study of ASF based glass ceramics sintered under various temperatures fabricated from waste is expected to give a better outcome in the GIC properties. The presence of ageing time in the GIC initiates the chemical reaction in the sample. Besides, different ageing time used in this research is believed to enhance the properties of GIC (Khiri et al., 2020). This study is conducted to improve the GIC properties in term of physical, structural and mechanical to go along with market trends and meet many aesthetic and functional requirements.

1.3 Research Objectives

Based on the problem statements above, the objectives for this study were:

1. To prepare GIC using ASF based glass ceramics derived from CS and SLS waste materials for calcium and silica sources.
2. To determine the effect of various sintering temperatures on ASF based glass ceramics.
3. To investigate the influence of ASF based glass ceramics on the physical, structural and mechanical properties of GIC.
4. To analyze the effect of different ageing times on the physical, structural and mechanical properties of GIC.

1.4 Scope of Study

This research has been focused on the incorporation of ASF based glass ceramics into the GIC. This research is conducted to enhance GIC samples' properties such as physical, structural, and mechanical properties. The ASF based glass ceramics sample is prepared using melt quenching technique and controlled sintering process following an empirical formula of [(x)CS·(45-x) SLS·15CaF_2·20P_2O_5·20Al_2O_3] where x = 5 and 20 (wt.%). The empirical formula was chosen by considering the fluorapatite phase's stability and excellent structural analysis from previous references (Rahman et al., 2019). The sintering temperatures for ASF based glass ceramics production are 600, 800, 1000 and 1200 °C for 4 h. The range of sintering temperature from 600 until 1200 °C was selected due to glass stability in this range following the previous reference (Khiri et al., 2020). The GIC is fabricated from three major components of ASF based glass ceramics powder, PAA and H_2O, using a 3:1:1 ratio through an acid-base reaction. Then, the GIC is immersed in deionized water for the ageing time from 1, 7, 14, 21 and 28 days. The selection for the duration of the ageing times following the previous reference where the physical and mechanical properties of GIC show a good result in these range of ageing times (Thoo et al., 2013; Khiri et al., 2020). The chemical and structural properties of waste and raw glass material will be characterized through XRF and XRD. The DSC is a thermal analysis technique used in the characterization of raw ASF glass samples. The density measurement of GIC samples is calculated according to Archimedes principle of distilled water as the immersion liquid. The structural properties of GIC samples are analyzed using XRD, FTIR, FESEM and EDX. The mechanical properties of the sample are determined by using CST for each composition, sintering temperatures and ageing time. The main focus on the CST for analysis of the mechanical properties is due to the mastication forces in the biological system. The mastication process is known as compressive in its nature. Hence, the analysis like compressive strength is suitable for this phenomenon. Moreover, this analysis can determine the qualities of dental application and compare the resin composites, dental amalgam, and dental cement in terms of their mechanical strength.

1.5 Significance of Study

This study focused on the fabrication of GIC from ASF based glass ceramics derived from the waste of CS and SLS glass. The ASF based glass ceramics are sintered through a controlled sintering process to obtain different phase transformations. The phase transformation of ASF based glass ceramics is important in the dentistry field (Jusoh et al., 2019). Basically, glass and glass ceramics have different phases: amorphous and crystalline structures (Wesolowski et al., 2020). Then, each phase is believed to provide a high advantage in the dental application (Bellucci et al., 2010). The sintering process conducted in this study can transform the ASF glass into glass ceramics through the changes in the glass crystallinity. The implementation of ASF based glass ceramics is expected to generate better outcomes in the performances and properties of GIC.

GIC is made up of the acid-base reaction using glass, deionized water and polymeric acid in the polymerization process (Thoo et al., 2013). The GIC is a well-known application in dental applications such as for luting cement, restorative and sealant (Moheet et al., 2018). This is due to their excellent adhesion, high biocompatibility and better aesthetic appearance (Lohbauer, 2010). This advantage has made GIC an excellent potential candidate in the dental and biomedical fields. However, researchers are still working on improving GIC's properties as their major limitation is low mechanical strength (Nicholson, 2010). The difference in sintering temperature and ageing times of GIC was expected to contribute to the enhancement of GIC in terms of mechanical properties. Thus, the main focus of this research, which is to obtain better mechanical properties for the GIC, is expected to be achieved in this study.

From the approximation, about 278 to 416 million tonnes of food waste will be produced in the next 25 years due to the rapid economy and population growth (Kiran et al., 2014). Some innovative methods have been introduced to consume and improve waste management, such as the utilization of the waste into beneficial resources for the industrial market (Hoornweg et al., 2013). Due to its high-value minerals, food waste can be implemented as a raw material for the production of glass, glass ceramic and ceramic (Cornejo et al., 2014). The examples of food waste that can be utilized in the industrial market are CS and SLS waste glass. From the previous studies, CS contains more than 90 % of calcium oxide composition while SLS waste glass consists of 70 % of silicon dioxide composition (Awang-Hazmi et al., 2007; Thoo et al., 2013). The ASF based glass ceramics for GIC fabrication make waste a source of chemical compounds that are silica and calcium sources in the ASF glass composition. The waste materials of CS and SLS glass are utilized in ASF fabrication to obtain the compositions of CaO and SiO_2. Both wastes had become an initiation to the fabrication of GIC for dental application. The utilization of waste in the GIC fabrication is limited in research. Hence this study is conducted to analyze the effectiveness of waste to the GIC.

1.6 Outline of the Thesis

The thesis is designed and arranged as follows Chapter 1 gives an introduction to biomaterials, bioceramics, GIC, problem statements, objectives of work, scope of study and significance of study. Reviewing previous works related to current research focusing on research conducted with different sintering temperatures and ageing time of GIC from waste materials is covered in Chapter 2. In Chapter 3, the methodology, including preparation, characterization and analysis of the samples, is described. The results due to the different sintering temperatures and ageing time on physical, structural, and mechanical properties of GIC are analyzed using the specific instruments and discussed in Chapter 4. Lastly, the conclusion and suggestions for future work are presented in Chapter 5.

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LIST OF PUBLICATIONS

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