STRUCTURAL, MECHANICAL AND BIOACTIVITY PROPERTIES OF SiO_2-CaO-B_2O_3-Na_2O-P_2O_5-CaF_2 GLASS AND GLASS-CERAMICS UTILIZED EGGSHELL AS CALCIUM SOURCE

By

LOH ZHI WEI

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

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Chairman : Mohd Hafiz Mohd Zaid, PhD
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As humans age, the deterioration of teeth in adults and even children are unprotected and easy to fracture due to the loss of mechanical strength over time. Hence, biomaterials such as bioactive glasses have gained significant scientific interest from researchers for application in dentistry. In the present work, the bioactive glass was synthesized through a melt-quenching approach with the configuration of $45SiO_2$–$25CaO$–$10B_2O_3$–$10Na_2O$–$(10–x)P_2O_5$–$xCaF_2$, where x = 0, 2, 4, 6, 8 and 10 wt.%. The eggshell waste was chosen as the calcium source. Considering the intrinsic brittleness of the glass, heat treatment is performed to improve the mechanical properties of the bioactive glass. Meanwhile, both samples will be immersed in phosphate buffer saline for 7 and 14 days to evaluate their bioactivity and biodegradability properties. Among the samples, BG3F with a CaF_2/P_2O_5 ratio of 6/4 demonstrated the optimal performance based on the structural, mechanical, bioactivity and biodegradability properties. The density of BG3F showed increment with heat treatment temperature, reaching a maximum value of 2.626 g/cm^3 at a temperature of 700 °C. X-ray diffraction analysis revealed the phase formation of fluorapatite, wollastonite, and cuspidine in the BG3F samples after heat treatment. In terms of mechanical results, BG3F heat-treated at 700 °C revealed the highest microhardness range of 6.72 GPa to 6.95 GPa and fracture toughness of 3.55 MPa·m$^{1/2}$ to 3.62 MPa·m$^{1/2}$ after 14 days of immersion. Furthermore, the pH of the glass and glass-ceramics samples showed an increasing trend ranging from 7.437 to 9.636 and 7.437 to 8.217, respectively, after being immersed for 14 days, indicating their bioactivity in response to the immersion medium. In conclusion, the BG3F sample with a heat treatment of 700 °C revealed the optimum performance, demonstrating the highest mechanical properties while maintaining bioactivity and biodegradability. The microhardness of the studied samples is comparable to the human enamel (2.00 – 5.00 GPa) and the commercial dental glass-ceramics (4.00 – 6.50 GPa) by more than 39% and 6.92%, respectively. Therefore, this research contributes to a novel approach by using wastes in fabricating bioactive glass while improving the mechanical properties of glass-ceramics for dental applications.

Keywords: Waste materials, Bioactive glass, Glass-ceramics, Mechanical, Dental applications

SDG: GOAL 4: Quality Education and GOAL 9: Industry, Innovation and Infrastructure

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SIFAT STRUKTUR, MEKANIKAL DAN BIOAKTIVITI SiO_2-CaO-B_2O_3-Na_2O-P_2O_5-CaF_2 KACA DAN KACA-SERAMIK MENGGUNAKAN SISA KULIT TELUR SEBAGAI SUMBER KALSIUM

Oleh

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Seiring bertambahnya usia manusia, penurunan keadaan gigi pada orang dewasa dan bahkan kanak-kanak tidak dilindungi dan mudah patah disebabkan kehilangan kekuatan mekanikal sepanjang masa. Oleh itu, bahan bioaktif seperti kaca bioaktif telah menarik minat saintifik yang signifikan daripada penyelidik untuk aplikasi dalam bidang pergigian. Dalam kajian ini, kaca bioaktif disintesis melalui teknik lebur dan pelindap secara konvensional dengan konfigurasi $45SiO_2–25CaO–10B_2O_3–10Na_2O–(10–x)P_2O_5–xCaF_2$, di mana x = 0, 2, 4, 6, 8, dan 10% berat. Sisa kulit telur telah dipilih sebagai sumber kalsium. Mengambil kira kerapuhan intrinsik kaca, rawatan haba dijalankan untuk meningkatkan sifat mekanikal kaca bioaktif. Sementara itu, kedua-dua kaca bioaktif dan kaca-seramik sampel akan direndam dalam larutan garam penampan fosfat selama 7 dan 14 hari untuk menilai sifat bioaktiviti dan biodegradasi mereka. Antara sampel-sampel tersebut, BG3F dengan nisbah CaF_2/P_2O_5 6/4 menunjukkan prestasi optimum berdasarkan sifat-sifat struktur, mekanikal, bioaktiviti, dan biodegradasi. Ketumpatan BG3F meningkat dengan suhu rawatan haba, mencapai nilai maksimum 2.626 g/cm^3 pada suhu 700 °C. Analisis pembelauan sinar-X menunjukkan pembentukan fasa fluorapatit, wollastonit, dan cuspidin dalam sampel BG3F selepas rawatan haba. Dari segi keputusan mekanikal, BG3F yang dirawat haba pada 700 °C menunjukkan julat kekerasan mikro Vickers tertinggi dari 6.72 GPa hingga 6.95 GPa dan kekuatan fraktur dari 3.55 MPa·m$^{1/2}$ hingga 3.62 MPa·m$^{1/2}$ selepas 14 hari rendaman. Selain itu, pH sampel-sampel kaca dan kaca-seramik menunjukkan trend peningkatan dari 7.437 hingga 9.636, dan 7.437 hingga 8.217, masing-masing, selepas direndam selama 14 hari, menunjukkan bioaktiviti mereka sebagai respons kepada medium rendaman. Kesimpulannya, sampel BG3F dengan rawatan haba pada suhu 700 °C menunjukkan prestasi optimum dengan sifat mekanikal tertinggi pada masa yang sama mengekalkan bioaktiviti dan biodegradasi. Kekerasan mikro sampel yang dikaji adalah setanding dengan enamel manusia (2.00 – 5.00 GPa) dan kaca-seramik pergigian komersial (4.00 – 6.50 GPa) masing-masing lebih daripada 39% dan 6.92%. Oleh itu, penyelidikan ini m menyumbang kepada pendekatan baru dengan menggunakan bahan buangan dalam fabrikasi kaca bioaktif sambil menambah baik sifat mekanikal kaca-seramik untuk aplikasi pergigian.

Kata Kunci: Bahan buangan, Kaca bioaktif, Kaca-seramik, Mekanikal, Aplikasi pergigian

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A	Area
a	Half-length of the indented area
Å	Angstrom
atm	Atmospheric pressure
c	Half-length of the cracked area
cm	Centimetre
d	Spacing distance between the adjacent lattice planes
°C	Degree Celsius
F	Maximum load
g	Gram
GPa	Gigapascals
kV	Kilovolts
m	Meter
mA	Milliampere
min(s)	Minute(s)
mL	Millilitre
mm	Millimeter
mol.%	Molar percent
N	Newton
^{o}C	Degree Celsius
s	Second
wt.%	Weight percent
μ	Micro
Al_2O_3	Aluminium oxide
B_2O_3	Boron Trioxide
$Ca(OH)_2$	Calcium Hydroxide
$Ca_{10}(PO_4)_6F_2$	Fluorapatite
$Ca_3Si_3O_9$	Pseudo-wollastonite
$Ca_4Si_2O_7F_{2,}$	Cuspidine
$CaCl_2$	Calcium chloride
$CaCO_3$	Calcium carbonate / Calcite

CaF_2	Calcium fluoride
CaO	Calcium oxide
$CaSiO_3$	Wollastonite
CO_2	Carbon dioxide
CuO	Copper (II) oxide
Fe_2O_3	Iron (III) oxide
K_2O	Potassium oxide
MgF_2	Magnesium fluoride
MgO	Magnesium oxide
Na_2CO_3	Sodium carbonate
Na_2O	Sodium oxide
P_2O_5	Phosphorus pentoxide
SiO_2	Silicon dioxide
SO_3	Sulphur Trioxide
SrO	Strontium oxide
AS	Artificial Saliva
BO	Bridging oxygen
CAp	Carbonate Apatite
CS	Compressive strength
EDX	Energy dispersive X-ray
FAp	Fluorapatite
FTIR	Fourier transform infrared spectroscopy
HA	Hydroxyapatite
HCA	Hydroxy carbonate apatite
HV	Vickers microhardness
ICSD	Inorganic Crystal Structure Database
IR	Infrared ray
ISO	International Organization for Standardization
K_{IC}	Fracture toughness
M	Molar mass
MPa	Megapascals
NBO	Non-bridging oxygen
PBS	Phosphate buffer saline

pH	Potential of hydrogen
PMMA	polymethyl methacrylate
S	Standard deviation
SBF	Simulated body fluid
SEM	Scanning electron microscopy
TEM	Transmission electron microscopy
T_f	Fictive temperature
T_G	Maximum crystallization temperature
T_g,	Glass transition temperature
T_m	Glass melting temperature
T_N	Nucleation temperature
T_{NG}	Crystallization temperature
Tris	Tris-hydroxymethyl-aminomethane buffer
Vm	Molar volume
W	Weight
$\bar{x}$	Average value
XRD	X-ray diffraction
XRF	X-ray Fluorescence
α	Alpha
β	Beta
θ	Diffraction angle
λ	Wavelength
ρ	Density

CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter aims to provide an overview of the research background related to biomaterial, particularly bioactive glass and glass-ceramics. The utilization of waste resources, such as eggshell waste, is used in this research to synthesize the bioactive glass and glass-ceramics as potential candidates for dental materials. The chapter extensively addresses the problem statement, outlining the objectives to overcome the challenges. Furthermore, the novelty of research work is discussed, highlighting its unique contributions to the field. The scope of the study is also described, indicating the specific areas and aspects that will be covered. Additionally, the importance of the study is emphasized, highlighting its potential impact and relevance in the relevant field. Lastly, an outline of the thesis is provided, giving a brief overview of the structure and organization of the research work.

1.2 Research background

As the world population ages, the deterioration of bones and teeth in adults and even children are vulnerable to fractures due to the gradual decrease in mechanical strength over a period of time. The challenge is to replace the fractured area with a material that can retain the same properties as the host materials before they become defective. Therefore, the invention of biomaterials has played a crucial role in addressing this challenge. Biomaterials are substances that can interact with the human body and act as an excellent alternative to conventional materials in many biomedical and dental applications. They are extensively used in bone and tooth replacement, repair and regeneration, and implantation (Filip et al., 2022).

Larry Hench pioneered the creation of the initial bioactive glass, 45S5 Bioglass®, during the 1960s. He developed a material capable of bonding with hard and soft tissues without causing rejection by the body. Consequently, this breakthrough garnered attention from various experts, including orthopedic surgeons, dental researchers, biomechanics specialists, and biologists, who were eager to explore the potential of biomaterials in different applications (Hench, 2006). However, despite its groundbreaking properties, bioglass does have certain limitations. One drawback is its relatively lower mechanical strength compared to other materials. Additionally, the composition of bioglass includes higher amounts of alkali elements, such as sodium, which lead to toxicity and reduced bioactivity, which may pose challenges in specific applications (Damen & Ten Cate, 1992; Ali et al., 2014). These shortcomings highlight the need for further research and development in bioactive materials to address these concerns and enhance their overall performance by modifying their compositions.

In 1977, Professor Ulrich Gross and his colleagues made a significant advancement by discovering bioactive glass-ceramics. They found that these glass-ceramics materials had a stronger bonding strength to the bone than bioactive glass. Furthermore, the progression of glass-ceramics was propelled forward by the innovation of A/W bioactive glass-ceramics (Kokubo et al., 1982). These materials demonstrated enhanced mechanical strength, toughness and stability in the physiological environment. As a result, they have been successfully utilized in vertebral replacement and spinal repair procedures, showcasing their practical and clinical potential. In addition, the glass-ceramics with better mechanical strength, specifically for dental restoration, were patented by previous researchers in the 1980s (Barrett et al., 1980). With the opened up new possibilities after the discovery of bioactive glass-ceramics, this research highlights the importance of tuning the composition and controlling the heat treatment temperature as crucial steps in the synthesis process. These optimizations ensure that the resulting materials meet the specific requirements of dental applications, further enhancing their suitability.

Initially, the base composition of the bioactive glass includes silicon dioxide (SiO_2), sodium oxide (Na_2O), calcium oxide (CaO), and phosphorus pentoxide (P_2O_5). In order to have better performance of the designed glass and glass-ceramics systems, each composition plays a vital role in modifying the glass structure. The bioactive glass consists of three crucial components. For instance, the SiO_2 content should remain below 60%, with a substantial presence of Na_2O and CaO, along with a higher CaO/P_2O_5 ratio (Hench, 1991). In addition to the primary components, additional elements can be integrated into the glass composition to alter its properties. One such element is boron, which, when added in small amounts, can enhance the bioactivity and hardness of the glass. The inclusion of boron has been shown to improve biocompatibility, biodegradability, and anti-bacterial properties. However, it is essential to note that adding excessive amounts of boron beyond a certain threshold can adversely affect the glass (Deilmann et al., 2020; Yusof et al., 2022). Apart from that, incorporating fluoride into the glass system forms fluorapatite, the main component of human enamel and dentin (Chen et al., 2022b). The inclusion of fluoride helps improve the mechanical properties of the glass and plays a role in preventing dental cavities by inhibiting the demineralization of enamel and dentin (Shah, 2016a). The rapid economic growth in developing countries such as Malaysia has indirectly boosted solid waste generation. Effective ways to manage solid waste should be considered in reducing the percentage of solid waste in Malaysia (Moh & Manaf, 2017). Hence, to address the solid waste issue, applying the concept of recycling, reducing and reusing methods is preferable, cost-effective and environmentally friendly for waste management. These approaches promote the transformation of waste into valuable resources, minimize waste generation at the source, and encourage the reuse of items. Moreover, environmental technology has been promoted in every country, where it is a new technology introduced to protect the Earth against environmental pollution (Doble & Kruthiventi, 2007). Disposing of waste materials in landfills is not a productive approach; however, these materials can be repurposed into valuable products with considerable potential for industrial applications (Waheed et al., 2020).

At the beginning of fabricating the bioactive glasses from previous work, the starting materials used are pure chemical elements, which will lead to the high costs of the final materials (De Caluwe et al., 2016; Pei et al., 2020). The challenge faced by researchers in the twenty-first century is to fabricate a cost-effective bioactive glass system with good performance for the final application (Hench & Thompson, 2010). Therefore, eggshells are selected as the calcium source material for synthesizing the bioactive glass. Eggshells contain 94% of calcium carbonate ($CaCO_3$). The purity of the CaO obtained from the eggshells after calcination is approximately 96% to 98% (Mohadi et al., 2016; Palakurthy et al., 2020; Jakfar et al., 2023). Utilizing eggshells in the fabrication of bioactive glass offers an environmentally friendly solution for disposing of waste eggshells, minimizing landfill waste. Furthermore, eggshell-based glass possesses distinct properties such as an anti-bacterial effect and improved hardness (Kamalanathan et al., 2014; Tshizanga et al., 2017; Onwubu et al., 2019) that make it well-suited for dental applications, providing a sustainable substitute for conventional glass materials.

In the present study, a series of bioactive glasses are fabricated using eggshell waste as a source of calcium through a conventional melt-quenching technique. The glass compositions are varied with different fluoride and phosphate (CaF_2/P_2O_5) ratios, and a controlled heat treatment is employed to transform the glass into glass-ceramics. The physical, structural and mechanical properties of the bioactive glass and glass-ceramics are characterized, including density, molar volume, phase formation, molecular structure, surface morphology, elemental analysis, compressive strength, microhardness and fracture toughness. Immersion tests are conducted to evaluate the in-vitro bioactivity and biodegradability properties of the synthesized materials. In-vitro bioactivity analysis assesses the phase formation, molecular structure, surface morphology and elemental analysis before and after immersion. In contrast, biodegradability analysis involves the pH changes, weight gain or loss, and mechanical properties before and after immersion. This research aims to develop a novel approach for fabricating bioactive glass from waste materials and improving the mechanical properties of bioactive glass-ceramics for dental applications.

1.3 Problem statement

There is a growing demand for dental materials due to various factors like an increasingly ageing population, advancement in dental procedures and rising awareness about oral health. This demand has prompted researchers to focus on developing innovative dental materials, such as bioactive glass, to meet the evolving needs. Initial bioactive glass production often starts with pure chemical elements as starting materials, which can be expensive and lead to higher production costs. Besides, the disposal of waste materials such as eggshells in Malaysia has become a significant concern. Around 13 million tonnes of eggs were produced in 2021, as reported by the Food and Agriculture Organization of the United Nations (2022). Some researchers use waste materials such as clam shells, soda-lime-silica glass waste, and rice husk to synthesize the bioactive glass system for dental fillings and biomedical applications but not for dental prosthetics (Khiri et al., 2020; Nayak et al., 2010b). Hence, in this study, the fabrication of bioactive glass involves the utilization of waste material such as eggshell as a calcium source is developed. Using waste materials as an alternative to the synthetic form reduces the overall production cost and shows several biologically beneficial effects on the bioactive glass system.

In the past few decades, significant research efforts have been focused on the development of biomaterials, particularly calcium phosphate-based ceramics and bioactive glass in bone substitution, while glass ionomer cement for dental restoration materials (Alatawi et al., 2019; Borkowski et al., 2020). However, there has been minimal research on bioactive glass designed explicitly for prosthetics or implants. This is because of the high alkali content and low toughness of the bioactive glass, which restrict their application in the dental field. To address these limitations, optimizing the composition of the starting materials to achieve properties comparable to those of human enamel and dentin is crucial. Research conducted earlier by Chen et al. (2022), Prasad et al. (2020) and Esmati et al. (2018) have shown that incorporating fluoride into the glass system enhances density and microhardness, promotes apatite development and provides anti-bacterial properties. However, excessive fluoride content can adversely affect the bioactivity and mechanical properties. In addition, the presence of phosphate is essential for the bioactivity of the glass, with optimal apatite growth observed within 2% to 10% of the phosphate content range (Adam et al., 2021). Nevertheless, an excess of more than 7% phosphate inhibits the bioactivity reaction and affects the strength of glass. (Marghussian & Mesgar, 2000; Al-eesa et al., 2017). Therefore, in this study, the fluoride and phosphate (CaF_2/P_2O_5) ratios are essential in fabricating the bioactive glass system to achieve optimal properties that improve mechanical properties while maintaining bioactivity.

Considering the intrinsic brittleness of bioactive glass that could not withstand the high mechanical load (Bellucci et al., 2011), a heat treatment process will be performed with the aim of enhancing the mechanical properties of the bioactive glass system. The commercial 45S5 Bioglass® has lower mechanical properties, limiting its properties to be used in various applications (Skallevold et al., 2019). Alternatively, the apatite-wollastonite glass-ceramics developed by Kokubo & Takadaman (1982) exhibit higher mechanical properties but are primarily utilized in bone substitution. Similarly, previous studies reported that apatite-mullite glass-ceramics demonstrate improved mechanical strength for bone substitution and dental restorative materials (Freeman et al., 2003; Fathi et al., 2005). However, the presence of aluminium oxide (Al_2O_3) content in these materials may inhibit their bioactivity. Hence, developing bioactive glass-ceramics with properties that closely resemble human enamel, including high microhardness and fracture toughness, is desirable (Dimitriadis et al., 2022). Therefore, selecting the optimal heat treatment temperature for the bioactive glass system is expected to improve the mechanical strength of the final product.

Moreover, in vitro studies are conducted to evaluate the potential for apatite formation and to assess the suitability of the bioactive glass and glass-ceramics for application in living organisms. Previous research was focused on studying the bioactivity and biodegradability properties of bioactive glass (Al-Noaman et al., 2012b; Al-eesa et al., 2017; Sriranganathan et al., 2017). However, there is limited research on the properties of glass-ceramics, as the conversion of bioactive glass to glass-ceramics through heat treatment may potentially affect these properties. The ion exchange phenomena occurring at the interface between the surface of the material and medium are primarily associated with the residual glassy phase. Glass-ceramics, having a denser structure, exhibit a reduced contact area between the materials and the immersed medium (Panah et al., 2022). Conversely, some studies have shown that although apatite formation may slow down when the crystallinity reaches 100%, it does not completely inhibit the bioactivity and biodegradability properties (Kaur et al., 2019). Therefore, a comprehensive analysis of the bioactivity and biodegradability properties of the novel bioactive glass and glass-ceramics, both before and after PBS immersion, will be thoroughly evaluated.

1.4 Research objective

The main objective of this study is to develop and optimize the mechanical properties while maintaining the bioactivity properties of the glass and glass-ceramics derived from waste materials. This research involved designing glass compositions, implementing the melt-quenching method, precise control of the heat treatment process, and conducting immersion tests to evaluate the performance of the materials.

The specific objectives of this study are as follows:

1. To synthesize the bioactive glass derived from waste eggshell as a source of calcium.
2. To investigate the effect of fluoride and phosphate ratios on the physical, structural and mechanical properties of bioactive glass.
3. To determine the effect of heat treatment temperatures on the physical, structural and mechanical properties of bioactive glass-ceramics.
4. To evaluate the effect of immersion periods on the in-vitro bioactivity and biodegradability properties of bioactive glass and glass-ceramics.

1.5 Novelty of research work

This study aims to contribute new knowledge regarding the properties and cost-effectiveness of this innovative material. Hence, the research work in this study brings several novel aspects to the field:

1. This innovative approach presents a novel way of utilizing eggshells as a calcium source to produce bioactive glass materials for dental prostheses.

2. Development of sustainable bioactive glass and glass-ceramics with the optimal CaF_2/P_2O_5 ratio and heat treatment temperature to obtain the ideal mechanical properties of the materials for dental materials, an alternative to traditional dental materials.
3. This novel material contributes to new knowledge on physical, structural, mechanical, bioactivity and biodegradability properties.

1.6 Scope of study

To achieve the objective of this study, the scope of the study includes the following:

1. A series of bioactive glasses based on the empirical formula of $45SiO_2–25CaO–10B_2O_3–10Na_2O–(10–x)P_2O_5–xCaF_2$, where x = 0, 2, 4, 6, 8 and 10 wt.% will be prepared using the conventional melt-quenching technique.
2. The chemical composition of the eggshell waste will be measured using X-ray fluorescence (XRF) spectroscopy to confirm the percentage of the chemical oxide and purity of CaO before synthesizing the bioactive glass.
3. The bioactive glasses will be subjected to heat treatment temperatures varying from 600 °C to 900 °C to produce bioactive glass-ceramics.
4. The physical, structural and mechanical properties of bioactive glasses and glass-ceramics will be analyzed using the Archimedes method, molar volume formulation, X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy with energy dispersive X-ray (SEM-EDX), compressive strength, Vickers microhardness and fracture toughness.
5. The optimum bioactive glass and glass-ceramics samples will be selected and immersed in phosphate buffer saline medium for 7 and 14 days.

6. The bioactivity and biodegradability properties of the optimum samples before and after immersion will be evaluated using XRD, FTIR, SEM-EDX, change in pH, weight gain or loss, compressive strength, Vickers microhardness and fracture toughness.

1.7 Importance of study

The study focuses on developing and optimizing bioactive glass and glass-ceramics derived from waste materials, reducing environmental impact and promoting the circular economy. Repurposing resources like eggshells showcases the transformation of waste into valuable products, lessening reliance on new materials and waste production. This research broadens biomaterial options, offering an eco-friendly alternative to conventional sources.

Besides, the increasing demand for biomaterials, including bioactive glass and glass-ceramics, arises from their appealing aesthetics, functionality, strength and bioactivity. Researchers globally, including from Japan, Malaysia, India and Pakistan, are exploring bioactive glasses for orthopedic and dental applications. Although dental materials are gaining popularity, the susceptibility to fractures remains a significant concern, leading to the need to replace these dental restorations. Indeed, the composition of materials and the parameters employed during processing, such as the heat treatment process, play a crucial role in modifying their properties to ensure suitability for specific applications.

Dental materials demand unique properties, including bioactivity, biodegradability, and strength. Hence, heat treatment is anticipated to enhance the physical, structural and mechanical properties of the bioactive glass and glass-ceramics. In addition, the immersion tests conducted on the selected bioactive glass and glass-ceramics are expected to provide valuable insights into their bioactivity and biodegradability. Consequently, this research is expected to show the optimum properties of the final products can lead to a novel approach for the low-cost fabrication of material from waste materials with better mechanical properties to apply in dental applications, such as prosthetics and implants. Ultimately, these findings will shed fresh light on the properties and economic viability of this innovative material.

1.8 Outline of thesis

The thesis consists of five chapters that are structured as follows. Chapter 1 provides a general overview of the research field and the need for biomaterials such as bioactive glass and glass-ceramics, the glass composition and eggshell-derived glass. This chapter also presents the problem statements, research objectives, novelty of research work, scope of study and importance of study. Chapter 2 reviews the utilization of eggshell waste, fabrication and types of bioactive glass and glass-ceramics. This chapter also reviews the physical, structural, mechanical, bioactivity and biodegradability properties of the bioactive glass and glass-ceramics from previous works conducted by other researchers in the field, serving as a guideline and reference for the current study. The methods used to prepare the bioactive glass and glass-ceramics samples and the characterization techniques employed throughout this study are explained in Chapter 3. Chapter 4 analyzes and discusses the results obtained from the experiments, focusing on the effects of CaF_2/P_2O_5 ratios, varying heat treatment temperatures, and the outcomes after immersion tests. This chapter covers the physical, structural, mechanical, bioactivity and biodegradability properties of the bioactive glass and glass-ceramics samples. Finally, Chapter 5 concludes the research findings and their significance and provides suggestions for future studies and areas of further exploration. The references, publications and conferences attended by the author will be placed in the last part of the thesis.

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