



**Faculty of Manufacturing Engineering**

**THE EFFECT OF HEATING RATE AND SINTERING  
TEMPERATURE ON GLASS CERAMIC COMPOSITE AT  
DIFFERENT FILLER LOADING**

**Nur Syahidah binti Salleh**

**Master of Science in Manufacturing Engineering**

**2016**

**THE EFFECT OF HEATING RATE AND SINTERING TEMPERATURE ON  
GLASS CERAMIC COMPOSITE AT DIFFERENT FILLER LOADING**

**NUR SYAHIDAH BINTI SALLEH**

**A thesis submitted  
in fulfillment of the requirements for the degree of Master of Science  
in Manufacturing Engineering**

**FACULTY OF MANUFACTURING ENGINEERING**

**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**2016**

## DECLARATION

I declare that this thesis entitled “The Effect of Heating Rate and Sintering Temperature on Glass Ceramic Composite at Different Filler Loading” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : .....

Name : .....

Date : .....

## APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.

Signature :.....

Supervisor Name :.....

Date :.....

## **DEDICATION**

This thesis is special dedicated to:

My supportive husband, Mohd Rasyuyadi bin Salleh

Our cores of heart, Nur Raisya binti Mohd Rasyuyadi and Muhammad Rasydan bin Mohd Rasyuyadi.

My life inspiration parents, Hj. Salleh bin Maidin and Hjh. Fatimah binti Muda

My understanding mother in-law, Hjh. Aishah binti Daud

My intelligent supervisors, Dr.Zurina Shamsudin and Profesor Madya Dr. Jariah Mohamad Juoi

& to all of my siblings and close friends

## ABSTRACT

The present work aims to develop fundamental information for the fabrication of glass ceramic composite (GCC) from recycled soda lime silicate (SLS) glass integrated with high loading of filler which is spent bleach earth (SBE). The aim of this study is to investigate the effects of different heating rate and sintering temperature on physical properties, microstructure and mechanical properties of GCC at different SBE loading. The recycled SLS glass and SBE were prepared by sieving to particle size  $< 45 \mu\text{m}$  and removing the oil in SBE by sonification process, respectively. The particle size distribution for both powders was determined using Laser Particle Size Analyzer (LPA). The main elements of recycled SLS glass were silicon dioxide ( $\text{SiO}_2$ ), and calcium oxide ( $\text{CaO}$ ), while the main elements of SBE were silicon dioxide ( $\text{SiO}_2$ ) and aluminium oxide ( $\text{Al}_2\text{O}_3$ ) determined via X-ray Fluorescence (XRF) analysis. Glass transition temperature ( $T_g$ ) of recycled SLS glass was observed around  $514^\circ\text{C}$ . The compositions were selected based on high SBE loading which were SLS to SBE; 60:40, 55:45 and 45:55 wt. %. The type of sintering involved for 60:40 and 55: 45 wt. % of SLS to SBE was viscous flow sintering (VFS). Meanwhile for 45:55 wt. % of SLS to SBE, the sintering type involved was liquid phase sintering (LPS). The GCC was then formed using uniaxial dry pressing method with pressure at 2.5 tonnes for square mold and at 8 tonnes for rectangular mold. In the first part, the GCC sample was sintered at  $700^\circ\text{C}$  with heating rate of 2, 4, 6, and  $8^\circ\text{C}/\text{min}$ . Observation on crystalline phases showed that main crystalline phases appeared were quartz ( $\text{SiO}_2$ ) and wollastonite ( $\text{CaSiO}_3$ ). Increasing the heating rate did not give remarkable results of physical properties on green GCC. Microstructure observation revealed the presence of pores at minimal quantity. In this parameter,  $2^\circ\text{C}/\text{min}$  was chosen as an optimum heating rate. In the second part, the GCC was sintered at different sintering temperature of  $750^\circ\text{C}$  and  $850^\circ\text{C}$ . The results showed distinctive differences in physical properties, phases, and microstructure. This indicates that the composite was strongly influence by the sintering temperature. Increasing the sintering temperature showed high percentage of shrinkage, low percentage of porosity, and water absorption. The main phases appeared were quartz ( $\text{SiO}_2$ ), wollastonite ( $\text{CaSiO}_3$ ), cristobalite ( $\text{SiO}_2$ ), and carneigeite ( $\text{NaAlSiO}_4$ ). Crystalline phases showed high intensity and additional of new phases when the sintering temperature increased. Observation on microstructure disclosed more uniform and densified microstructure. An optimised green GCC of 45 wt. % SBE was successfully fabricated at sintering temperature of  $850^\circ\text{C}$  and heating rate of  $2^\circ\text{C}/\text{min}$ . The mechanical properties of hardness was 0.12 GPa and flexural strength of 9.50 MPa. A good prospect of addition some binder should be further explored on the fabrication of glass ceramic composite with high loading of SBE.

## ABSTRAK

Kajian bertujuan untuk menyediakan maklumat asas dalam pembuatan komposit seramik kaca (GCC) daripada kaca soda kapur silikat (SLS) dikitar semula dengan komposisi tertinggi dari tanah peluntur terpakai (SBE). Tujuan kajian ini adalah untuk mengkaji kesan kadar pemanasan dan suhu sinter yang berbeza terhadap sifat fizikal, mikrostruktur dan sifat mekanik GCC pada komposisi SBE yang berbeza. Kaca SLS yang dikitar semula ditapis untuk mendapatkan saiz serbuk <45 mikron manakala SBE telah melalui proses sonifikasi untuk membuang kandungan minyak. Taburan saiz zarah ditentukan menggunakan Penganalisa Zarah Laser (LPA). Elemen utama kaca SLS dikitar semula adalah silikon oksida ( $\text{SiO}_2$ ), dan kalsium oksida ( $\text{CaO}$ ) manakala unsur utama SBE adalah  $\text{SiO}_2$  dan Aluminium oksida ( $\text{Al}_2\text{O}_3$ ) melalui analisis XRF. Suhu peralihan kaca ( $T_g$ ) kaca SLS dikitar semula adalah  $514\text{ }^\circ\text{C}$ . Komposisi dipilih berdasarkan kepada kandungan SBE yang tinggi iaitu SLS kepada SBE; 60:40, 55:45, 45:55 wt. %. Jenis pensinteran yang terlibat untuk 60:40 dan 55:45 wt. % kaca SLS terhadap SBE adalah pembakaran aliran likat (VFS). Sementara 45:55 wt. % SLS terhadap SBE, jenis pembakaran yang terlibat adalah cecair fasa pensinteran (LPS). GCC kemudiannya dibentuk menggunakan kaedah mampatan kering ekapaksi pada tekanan 2.5 tan untuk acuan segi empat sama dan 8 tan untuk acuan segi empat tepat. Bahagian pertama, sampel GCC disinter pada  $700\text{ }^\circ\text{C}$  dengan kadar pemanasan 2, 4, 6 dan  $8\text{ }^\circ\text{C}/\text{min}$ . Pemerhatian pada fasa hablur menunjukkan bahawa fasa hablur utama adalah quarza ( $\text{SiO}_2$ ) dan wollastonite ( $\text{CaSiO}_3$ ). Peningkatan kadar pensinteran tidak memberi kesan yang terbaik pada ciri-ciri fizikal GCC. Pemerhatian terhadap mikrostruktur menunjukkan kuantiti liang berkurang. Walau bagaimanapun, permukaan kasar diperolehi dan kuantiti lubang berbentuk terhasil secara bertaburan. Dalam parameter ini,  $2\text{ }^\circ\text{C}/\text{min}$  dipilih sebagai kadar pemanasan yang optimum. Pada bahagian kedua, GCC disinter pada suhu pensinteran berbeza iaitu  $750\text{ }^\circ\text{C}$  dan  $850\text{ }^\circ\text{C}$ . Perubahan ketara pada sifat fizikal, fasa dan mikrostruktur diperhatikan. Ini menunjukkan komposit ini amat dipengaruhi oleh suhu pensinteran. Peningkatan suhu pensinteran menunjukkan peratusan yang tinggi terhadap pengecutan. Peratusan yang rendah pada keliangan dan penyerapan air. Fasa utama adalah kuarza ( $\text{SiO}_2$ ), wollastonite ( $\text{CaSiO}_3$ ), cristobalite ( $\text{SiO}_2$ ) dan carneigeite ( $\text{NaAlSiO}_4$ ). Kristaliniti menunjukkan fasa intensiti yang tinggi dan penambahan fasa baru apabila suhu pensinteran bertambah. Pemerhatian pada mikrostruktur mendedahkan keseragaman dan kemampatan. GCC hijau yang optimum adalah 45 wt. % SBE, berjaya direka pada suhu pembakaran  $850\text{ }^\circ\text{C}$  dan kadar pensinteran  $2\text{ }^\circ\text{C}/\text{min}$ . Sifat mekanik, ujian kekerasan micro adalah 0.12 GPa dan kekuatan lenturan adalah 9.50 MPa. Prospek yang lebih baik perlu dikaji terhadap penambahan beberapa bahan pengikat terhadap penghasilan komposit seramik kaca yang mengandungi kandungan SBE yang tinggi.

## ACKNOWLEDGEMENTS

All praises to Allah the Almighty and blessing be upon the final messenger Prophet Muhammad SWT. I am deeply thankful to Allah for giving me the strength to complete my master journey. First and foremost, I would like to convey my gratitude to my supervisor, Dr. Zurina binti Shamsudin for her guidance and encouragement throughout the duration of this project. The brilliant comments to raise the quality of my research are highly appreciated. I also like to thank my co-supervisor Profesor Madya Dr. Jariah Muhamad Juoi for her constant support, useful inputs and knowledge. Secondly, I gratefully acknowledge UTeM for PJP/2013/FKP (9A)/S01190 and KPM through MyBrain15 for financial support. I further extend my special appreciation to FKP staff and fellow postgraduate students for their help. Last but not least, I would like to express my thankfulness especially to my greatest blessing from Allah, Mohd Rasyuyadi bin Salleh for his sacrifice, unconditioned support and understanding during periods of my study. To my intelligent Nur Raisya and Muhammad Rasydan, both of you teach me a lots about time management and inspired me to smartly overcome the difficulties throughout the study. To my mother-in-law, Hjh. Aishah binti Daud, my father, Hj. Salleh bin Maidin and my mother, Hjh. Fatimah binti Muda, your prayer for me was what sustained me thus far, also a special thanks are dedicated to all my siblings and close friends who always motivate me to strike towards my goal.



## TABLE OF CONTENTS

	<b>PAGE</b>
<b>DECLARATION</b>	
<b>DEDICATION</b>	
<b>ABSTRACT</b>	i
<b>ABSTRAK</b>	ii
<b>ACKNOWLEDGEMENT</b>	iii
<b>TABLE OF CONTENTS</b>	iv
<b>LIST OF TABLES</b>	vii
<b>LIST OF FIGURES</b>	ix
<b>LIST OF ABBREVIATIONS</b>	xiii
<b>LIST OF PUBLICATIONS</b>	xv
<b>CHAPTER</b>	
<b>1. INTRODUCTION</b>	<b>1</b>
1.1 Background of study	1
1.2 Problem statement	3
1.3 Objectives	5
1.4 Scope of study	5
<b>2. LITERATURE REVIEW</b>	<b>7</b>
2.1 Introduction	7
2.2 Spent bleach earth	7
2.2.1 Composition and properties of spent bleach earth	9
2.2.2 Recycling of SBE	12
2.3 Incorporation of waste into soda lime silicate (SLS) glass	14
2.3.1 Types of waste	15
2.3.2 Processing of glass ceramic composite (GCC)	16
2.4 Sintering process	19
2.4.1 Theory of sintering	21
2.4.2 Types of sintering	23
2.4.3 Parameters that govern sintering	29
2.5 Sintering parameter affecting the properties and microstructure of green glass ceramic composite	32
2.5.1 Effect of heating rate	33
2.5.2 Effect of sintering temperature	37
2.6 Mechanical properties	42
<b>3. METHODOLOGY</b>	<b>46</b>
3.1 Introduction	46
3.2 Raw material	50
3.2.1 Spent bleach earth	50
3.2.2 Recycled soda lime silicate (SLS) glass	50
3.3 Powder preparation	51
3.3.1 Oil extraction	51

3.3.2	Sieving	52
3.3.3	Powder processing	52
3.4	Forming of glass ceramic composite (GCC)	53
3.4.1	Uniaxial pressing method	53
3.5	Sintering	56
3.6	Characterization of raw material	58
3.6.1	Particle size analysis	58
3.6.2	Elemental analysis	58
3.6.3	Thermal behaviour analysis	58
3.7	Physical analysis	59
3.7.1	Linear Shrinkage	59
3.7.2	Apparent Porosity, Water Absorption and Bulk Density	60
3.8	Characterization of sintered GCC	62
3.8.1	Phase identification	62
3.8.2	Morphology observation	63
3.9	Mechanical properties analysis	64
3.9.1	Microhardness	65
3.9.2	Flexural strength (3 Point bending Test)	65
<b>4.</b>	<b>RESULT AND DISCUSSION</b>	<b>67</b>
4.1	Introduction	67
4.2	Powder characterization	67
4.2.1	Particle size analysis	67
4.2.1.1	Spent bleach earth (SBE)	68
4.2.1.2	Recycled soda lime silicate (SLS) glass	68
4.2.2	Chemical composition	69
4.2.2.1	Spent bleach earth (SBE)	69
4.2.2.2	Recycled soda lime silicate (SLS) glass	70
4.2.3	Thermal reaction	72
4.2.3.1	Spent bleach earth (SBE)	72
4.2.3.2	Recycled soda lime silicate (SLS) glass	74
4.3	The effect of different heating rate and SBE loading on sintered properties of glass ceramic composite (GCC)	75
4.3.1	Physical properties	76
4.3.1.1	Physical Observation	76
4.3.1.2	Linear Shrinkage	78
4.3.1.3	Apparent Porosity	80
4.3.1.4	Water Absorption	82
4.3.1.5	Bulk Density	84
4.3.2	Phase and microstructure	87
4.3.2.1	Phase identification using X-ray Diffraction (XRD)	87
4.3.2.2	Microstructure analysis Scanning Electron Microscopy (SEM)	89
4.4	Effect of sintering temperature and SBE loading on glass ceramic composite (GCC)	92
4.4.1	Physical properties	93
4.4.1.1	Physical Observation	93
4.4.1.2	Linear Shrinkage	95
4.4.1.3	Apparent Porosity	97

4.4.1.4	Water Absorption	100
4.4.1.5	Bulk Density	102
4.4.2	Phase and microstructure	105
4.4.2.1	Phase identification using X-ray Diffraction (XRD)	105
4.4.2.2	Microstructure analysis Scanning electron microscopy (SEM)	111
4.4.3	Mechanical properties analysis	116
4.4.3.1	Microhardness	116
4.4.3.2	Flexural Strength	118
<b>5.</b>	<b>CONCLUSION AND RECOMMENDATIONS FOR FUTURE RESEARCH</b>	<b>121</b>
	<b>REFERENCES</b>	<b>126</b>
	<b>APPENDICES</b>	<b>146</b>

## LIST OF TABLES

<b>TABLE</b>	<b>TITLE</b>	<b>PAGE</b>
2.1	Amount of SBE from vegetable oil refineries process per year	9
2.2	Chemical composition of spent bleach earth (SBE)	11
2.3	The utilization of SBE in various field	14
2.4	Types of waste incorporation with recycled glass	15
2.5	Stage of sintering	22
3.1	The ratio of SLS waste glass powder and SBE	53
3.2	Total sample required for testing	55
3.3	Batches of samples sintered at different heating rate	57
3.4	Batches of samples sintered at different sintering temperature	57
4.1	Chemical composition of spent bleach earth (SBE)	70
4.2	Chemical composition of recycled soda lime silicate (SLS) glass (Rosli,2013)	71
4.3	Summarize of thermal event of spent bleach earth (SBE)	73
4.4	Summarize of thermal event of recycled SLS glass	75

4.5	Color changes towards different heating rate	77
4.6	The SEM micrograph at different batch formulation and heating rate	90
4.7	Color changes towards sintering temperatures	94
4.8	Crytalline phases at different sintering temperature	110
4.9	The SEM micrograph at different batch formulations and sintering temperatures	112
4.10	Microhardness results of sample 45AC	117
4.11	Modulus of rupture (MOR) of 45AC	118

## LIST OF FIGURES

FIGURE	TITLE	PAGE
2.1	Spent bleach earth (Indiamart, n.d.)	9
2.2	Two stage of sintering profile (Rahaman, 2008)	20
2.3	One stage of sintering profile (Rahaman,2008)	20
2.4	Microstructure changes of powder particles during stages of sintering (Rahaman,2008)	22
2.5	Schematic on two possible path during solid state sintering (Park, 2007)	24
2.6	a) Changes on particles during liquid phase sintering and b) The mechanisme occurred with regards to shrinkage (Rahaman,2008)	26
2.7	SEM image of sintered silica particle on silicon wafer surface (Zink, 2008)	27
2.8	SEM microstructure of mullite ceramics sintered at (a) 1300 °C 5°C/min (b) 1600 5 °C/min (c) 1300 °C; 30 °C/min (d) 1600 °C/min 30 °C respectively for 5 h. (Sanad et al. , 2013)	34
2.9	SEM micrographs of high content of glass: (a) Fast heating rate, (b) Slow heating rate (Cicek et al., 2014)	35

2.10	SEM micrographs of low content of glass: (a) Fast heating rate, (b) Slow heating rate (Cicek et al., 2014)	36
2.11	SEM micrograph of glass ceramic composite (GCC) sintered at different sintering temperature a) 800 °C b) 880 °C c) 950 °C d) 1000 °C (Appendino et al., 2004)	39
2.12	SEM micrograph of glass ceramic from finer powder a) 0 hour b) 5hour (Bernard et al., 2007)	45
3.1	Flow diagram of heating rate methodology	48
3.2	Flow diagram of sintering temperature methodology	49
3.3	Spent bleach earth	50
3.4	Recycled soda lime silicate glass	51
3.5	Sonification process removing oil from SBE	52
3.6	Horizontal ball mill machine	53
3.7	Uniaxial dry pressing and the forming process	54
3.8	(a) square mold (b) rectangular mold	54
3.9	Schematic of sintering profile (a) Heating rate (b) Sintering temperature	56
3.10	Soaking the samples into boiling distilled water	62
3.11	Schematic illustration of cross section cut of square sample	64
3.12	Scanning electron microscopy	64
3.13	Schematic drawing of rectangular mold for flexural strength testing	66

3.14	Three-Point Fixture Configuration (ASTM C1161)	66
4.1	Particle size distribution of spent bleach earth (SBE)	68
4.2	Particle size distribution of recycled soda lime silicate (SLS) glass	69
4.3	DTA-TG curve of spent bleach earth (SBE)	73
4.4	DTA-TG curve of recycled soda lime silicate (SLS) glass	74
4.5	Linear shrinkage of green GCC at different heating rate and various SBE loading	78
4.6	Percentage of apparent porosity of green GCC at different heating rate and SBE loading	80
4.7	Percentage of water absorption of green GCC at different heating rate and SBE loading	83
4.8	Percentage of bulk density of green GCC at different heating rate and SBE loading	85
4.9	XRD pattern of green GCC on 40 wt. % SBE at different heating rate	88
4.10	Linear shrinkage at different sintering temperatures of green GCC on different SBE loading	95
4.11	Apparent porosity on different sintering temperature of green GCC at different SBE loading	97
4.12	Water absorption on different sintering temperature of green GCC at different SBE loading	101



4.13	Bulk density at different sintering temperature of green GCC at different SBE loading	102
4.14	XRD pattern of GCC sintered at 700 °C for different SBE loading	106
4.15	XRD pattern of GCC sintered at 750 °C for different SBE loading	107
4.16	XRD pattern of GCC sintered at 850 °C for different SBE loading	109
4.17	SEM micrograph and EDX result	115
4.18	Optical microstructure of fracture sample	119

## LISTS OF ABBREVIATIONS

$\text{Al}_2\text{O}_3$	Alumina
ASTM	American Society for Testing and Material
$\text{CaCO}_3$	Calcium Carbonate
CaO	Calcium Oxide
DTA	Differential Thermal Analysis
EDX	Energy Dispersion X-ray
EU	European Union
GCC	Glass ceramic composite
GPa	Giga Pascal
LOI	Loss of ignition
LPA	Laser Particle Size Analyzer
LPS	Liquid Phase Sintering
MPa	Mega Pascal
SBE	Spent Bleach Earth
SEM	Scanning Electron Microscopy
SFE	Spent Filtered Earth
$\text{SiO}_2$	Silica
SLS	Soda Lime Silicate
SSS	Solid State Sintering
$T_c$	Crystallization temperature
$T_g$	Glass transition temperature
$T_m$	Melting temperature
$T_n$	Nucleation temperature
$T_{ng}$	Nucleation and glass transition temperature
$T_s$	Softening temperature

VFS

Viscous Flow Sintering

XRF

X-ray Fluorescence

## LIST OF PUBLICATIONS

### **(i) Peer reviewed journals**

1. Shamsudin, Z., **Salleh, N.**, Juoi, J.M., Mustafa, Z., Zulkifli, M.R., 2016. The Effect of Spent Bleach Earth Loading on the Sintered Properties of Green Glass Ceramic Composite. Key Engineering Materials, 694, pp.179-183.
2. Shamsudin, Z., **Salleh, N.**, Mustafa, Z., Bakar, M.A.A., Hasan, R., 2016. Influence of Size Particles of SLS Glass on Properties of Sintered SBE Reinforced Glass Waste Composite. Proceedings of Mechanical Engineering Research Day, pp. 1-2.

### **(ii) Conference**

1. 24<sup>th</sup> Scientific conference of the Microscopy Society Malaysia (SCMSM) 2015 (Oral), Melaka, 2<sup>nd</sup> – 4<sup>th</sup> December 2015.
  - Title: The Effect of Spent Bleach Earth Loading on the Sintered Properties of Green Glass Ceramic Composite.
2. Mechanical Engineering Research Day (MERD' 16) 2016 (Poster), Melaka, 30<sup>th</sup> – 31<sup>st</sup> March 2016.
  - Title: Influence of Size Particles of SLS Glass on Properties of Sintered SBE Reinforced Glass Waste Composite.

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of study

Technological development, economic prosperity, cultural evolution, and urbanization have affected the Earth greatly in all aspects and this includes the increasing waste production. As urban population is increasing and consumption pattern is changing, solid waste management has been applied. In Asia, immediate attention towards waste management is required in all countries especially for developing countries including Malaysia. In the context of developing countries, the urgency is needed as health and environmental implication is always associated with solid waste management (Marshall and Farahbakhsh, 2013).

Recycling is considered as a very safe method and not expensive. In Kuala Lumpur, the government focuses on increasing the recycling rate, managing the costs of solid waste disposal, and deploying the use of technology in managing solid waste (Saeed et al., 2009). Due to this situation, studies have been done to protect and preserve the earth by implementing more reduce, reuse, and recycle (3R) alternatives. The 3R initiative is not only run in Malaysia. This is a common practice in the other developed and developing countries too. The usage of recycled soda lime silicate (SLS) glass is also an initiative that has been proposed by researchers who study the fabrication of glass ceramics or so-called glass ceramic composite with various applications (Ponsot and Bernardo, 2013). This has been recommended due to the unique behaviour of glass that can be recycled many times

without reducing its strength. It is also adaptable in different techniques and shapes of fabrication (Juoi et al., 2013). Besides reducing pollution, it also offers less energy in the processing of secondary materials (Chinnam et al., 2013).

The establishment of recycled SLS glass in the fabrication of glass ceramic composite (GCC) has inspired other researchers to integrate other natural waste materials into the recycled SLS glass. A combination of various types of natural waste was found improving the properties of glass ceramic composite (GCC). Spent bleach earth (SBE) is a natural waste from palm oil refineries. It is a clay type and has been used as an absorbance in palm oil refineries to absorb all the impurities including colours. It contains a major composition of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ , it is expected to have a possibility in improving the properties of GCC when integrated with recycled SLS glass. Boey et al. (2011a) stated that SBE has a potential to be used as a clay substitute in brick or tile manufacturing processes.

Sintering process is a controlled heat treatment used to fabricate GCC. This process is very common in which it can produce strong and dense-glass ceramics from waste at very low sintering temperature (Bernardo et al., 2007; Fernandes et al., 2012). Properties and densification of GCC can be adjusted by controlling the sintering profile (Pascual et al., 2002). Different parameters such as method, composition, temperature, and heating rate give advantages in producing glass ceramics (Juoi et al., 2013) as it influences the types of sintering occur during sintering process.

This sintering profile of composition, heating rate and sintering temperature need to control to achieve a good properties of GCC with low porosity, low water absorption, and high density. The formation of phases and development on the microstructure is expected to show correlation between the selection of parameters with the physical and mechanical properties of GCC.

## 1.2 Problem Statement

In Malaysia, recycled soda lime silicate glass is easily accessible. Besides that, it has a good bending strength and chemical stability (Bernardo et al., 2007). Also, it improves water absorption of ceramic tiles (Pontikes et al., 2007). In general process, recycling soda lime silicate glass was seen as a solution of landfilled space (Saeed et al., 2009).

Spent bleach earth (SBE) is well known as one of vegetable industrial waste has dramatically increased. It is used as an absorbance in the vegetable refineries process including from palm oil industries. In Malaysia, over 150,000 tonnes of SBE is estimated to be produced annually with more than 17 million tonnes of palm oil production (Boey et al., 2011a). Generally, it is predicted that the production of SBE waste around the world is 600,000 tonnes (Suhartini et al., 2011). The current practice to dispose the high volume of SBE is through landfill. However, this disposal method is problematic due to the oil on surface area of the SBE particles which exposed to oxygen from the environment. Thus, producing rapid oxidation and adequate heat to ignite the oil. Hence, the disposal activity need to be done immediately to avoid rapid oxidation. This disposal method has been banned by European Union (EU) landfill directive (Beshara and Cheeseman, 2014). A consequence of this disposal method also was very costly and need to be paid by refineries (Loh et al., 2013). Hence, alternative is extensively needed to control consequence mentioned. Recycling method can be used extensive in maintaining good levels in disposal of waste from refineries especially SBE.

SBE was utilized by regenerating the SBE with acid or alkaline to recover it. This regenerated SBE will be used again as an absorbance to absorb other unwanted matter such as fluoride (Malakootian et al., 2011), basic dye (Mana et al., 2006) and herbicide paraquat

(Tsai and Lai, 2006). Further studies on properties of SBE has led to a discovery of new technology on bio-organic fertilizer and briquette made from SBE. In bio-organic fertilizer, the adequate amounts of valuable mineral elements from co-composed of SBE with some agricultural and palm oil milling by-products has a positive impact on soil physical. Meanwhile, the SBE was used as briquette due to the catalytic partial oxidation's (CPO) hydrocarbon component and high calorific value in it (Suhartini et al., 2011; Loh et al., 2013).

Besides that, the potential of SBE to be used as a clay substitute in the production of brick and tile manufacturing also has been highlighted by many researchers (Boey et al., 2011a; Beshara and Cheeseman et al., 2014). Recent research on the formation of pore forming brick by Eliche-Quesada and Corpas-Iglesias (2014) reported that only 10 wt. % of SBE was added into clay brick formulation that showed an optimum result of low bulk density, mechanical strength and thermal conductivity with higher total porosity and water absorption with respect to the pure clay brick. It was sintered at sintering temperature of 950 °C and heating rate of 3 °C/min. Increase the SBE loading higher than 10 wt. % result in high mechanical strength which was 30–25 MPa. However, higher water absorption value was obtained.

In theory, sintering profile at various heating rate, sintering temperature and holding time can affect the end properties of sintered GCC (Guo et al., 2010). However, the holding time favour less significant effect to the properties as compared to heating rate and sintering temperature (Bernardo et al., 2007). Therefore, by differentiate the heating rate and sintering temperature, it will be expected to produce a good properties of GCC with maximum used of weight percentage SBE between 40 to 55 wt. % in one GCC batch. Moreover, the effect of these parameters on physical properties, microstructure and phases