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## Effects of Bentonite and Eggshell as Fillers on Thermal-Physical Properties of Sintered Glass Composite

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### ABSTRACT

As the number of waste increases rapidly, the need to recycle it too becomes increasingly important. The lack of proper recycling leads to negative effects like climate change, the need for many landfills and high costs of recovery. This study focuses on the recycling of eggshell (ES), soda lime silicate glass (SLSG), borosilicate glass (BSG) and spent bleach earth (SBE). We investigated the effect of bentonite and eggshell as fillers on the properties of sintered glass composite based on four types of sintering temperature, namely 750, 800, 850 and 900°C. The composites were prepared using bentonite and ES as fillers and SLSG, SBE and BSG as matrix. The particle size of powders was approximately 40 µm. The samples were formed using uniaxial dry pressing at different weight percentages (wt.%) of ES and bentonite loading such as 2, 5, 10, 15 and 20 wt.%. The samples were sintered at the four sintering temperatures at a constant heating rate of 2°C/min for 1 hour. Then, we analysed the composite in terms of its physical properties using X-ray diffraction (XRD), for identifying its crystalline phases, and scanning electron microscopy (SEM), for identifying the relationship between the physical properties and microstructures. XRD studies showed that quartz initially precipitated, and when the SBE loading increased to 25 wt.%, phases coesite, cristobolite and carnegite were also precipitated. The effect of bentonite on the hardness of these composites was characterised by Vickers, and the results revealed a significant increase in hardness of the fully crystallised system. High loading of ES sintered at 900°C produced the lowest water absorption of 2.48%, accompanied by an apparent porosity of 6.25% and a bulk density of 2.52 g/cm<sup>3</sup>. SEM observations revealed that the decrease of wt.% of the filler leads to the major occurrence of pores. The study concludes that suitable compositions of ES and bentonite filler loading can be formed using alternative materials for structural applications such as tiles production.

#### Keywords:

Bentonite; recycle glass composite;  
sintering; spent bleach earth

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## 1. Introduction

In recent years, special attention has been devoted to industrial sectors that are sources of environmental pollution [1]. The industry produces large volumes of solid wastes, which can

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negatively impact the environment, especially rivers and lakes. Waste disposal is an important problem as it can negatively affect public health as well. In term of waste materials, shortage of landfills sites leads to worldwide research to develop the recycling technology and to reduce the solid waste as much as possible. The landfill in Malaysia indeed needs solution due to the increase in waste dramatically [2]. One of the best solutions is to recycle and turn the waste into a beneficial and environment-friendly product [3].

In the glass-manufacturing industry, there are several types of waste glasses, with a majority consisting of borosilicate glass (BSG) and soda lime glass (SLG). The term 'waste' refers also to natural resources. In Malaysia, there are several types of such natural resources like spent bleach earth (SBE) from the oil refinery industry. SBE can be defined as an industrial waste that is mainly generated from the edible oil processing. SBE is commonly disposed of in landfill without any pretreatment and usually contains 20-40 wt.% of oil. During the refining process, 0.5%-1.0% of activated clay or fresh bleaching earth (BE) is used where all BE will convert into waste as SBE. Therefore, it is estimated that the production of SBE waste around the world is 600,000 tons. As the second largest producer of crude palm oil in the world, Malaysia alone produces more than 100,000 tons of SBE yearly which contains a high percentage of oil. For this reason, SBE is the potential to be used as a renewable raw material for the production of green glass ceramic [4]. All the natural wastes are safe to be used further and are not hazardous. The waste from palm oil processing can also be recycled into a new product. A recent study reported that SBE powder improves the hardness of the composite [5].

Eggshells (ES) are natural waste products of eggs that have been used as foods and foodstuffs. Unfortunately, ES contributes to environmental pollution, as they support microbial actions. ES are sources of calcium carbonate ( $\text{CaCO}_3$ ), known as calcite, a stable polymorph at room temperature. The eggshells represent approximately 11% of the total weight of eggs, which is approximately 60 grams [6]. This study aims to recycle ES in  $\text{CaCO}_3$  to develop useful filler in composites. It will reduce the need to manage ES waste without damaging the environment. A study has already reported that additional fillers such as bentonite improve the composite's colour appearance [7]. By reusing wastes like BSG, soda lime silicate glass (SLSG), and SBE, the problem of environmental pollution can be tackled. This study shows that ES can be used as fillers in recycled glass. Bentonite has also been used as filler in this project. The project aims to turn the waste into a beneficial product. The purposes of using waste are twofold: first, for protecting the environment, and second, for saving the cost of manufacturing a new product.

## 2. Methodology

SLSG waste was collected from household waste. SLSG and ES were prepared using a crusher machine and planetary ball mill. Then, the powders were sieved using a vibratory sieve shaker with a size of  $45\ \mu\text{m}$  to get fine powders ( $<40\ \mu\text{m}$ ). SBE was supplied by Universiti Malaysia Pahang (UMP). The supplied SBE was unprocessed, and the sonication method was used to extract oil. The raw SBE was cleaned before extracting the oil using the sonication process, which was followed by filtration and drying in an oven until a powdered form of SBE was obtained.

The SBE powder was then mixed with ethanol in a beaker and again sonicated in an ultrasonic machine for 20 minutes before drying at ambient temperature. Bentonite powder was purchased from PolyScientific Sdn. Bhd.

Particle sizes of SLSG, BSG, SBE bentonite, and ES were determined using particle size analyser, Mastersizer 2000 (Malvern Instrument Ltd model). Bentonite and ES acted as fillers in this project. The whole batch is mixed using a ball mill machine. The powder was compacted using a uniaxial die pressing machine at  $1.5\text{g}$  (for circle mould) and  $3.5\text{g}$  (for square mould) at 40 MPa (3.5 tonnes). Table

1 and 2 show the composition of the final composites. The green bodies were sintered using a laboratory electric furnace Carbolite (1300 model) at 750, 800, 850 and 900°C at a constant heating rate of 2°C/min for 1 hour. Physical analyses were conducted according to ASTM C373. The analyses involved were water absorption, bulk density, and apparent porosity.

Microstructures of the samples were observed through SEM, using an SEM EVO 50 (Carl Zeiss SMT, UK) at accelerated 10 kV. Surfaces of the samples were coated with gold layers to ensure that the samples were conductive using the SC-7620 mini sputter. The phases of sintered samples were analysed based on X-ray diffraction (XRD), while hardness was analysed using Vicker's hardness.

**Table 1**

Ratio of bentonite as filler content in green glass ceramic

Sample code	SLSG (wt.%)	BSG (wt.%)	SBE (wt.%)	Bentonite (wt.%)
GB1	32.5	32.5	35	0
GB2	32.5	32.5	33	2
GC1	32.5	32.5	30	5
GC2	32.5	32.5	25	10
GC3	32.5	32.5	20	15
GC4	32.5	32.5	15	20

**Table 2**

Ratio of eggshell as filler content in green glass ceramic

Sintering temperature, °C	Sample code	SLSG (wt.%)	SBE (wt.%)	ES (wt.%)
800	A1	47.5	47.5	5
	A2	45.0	45.0	10
	A3	42.5	42.5	15
	A4	40.0	40.0	20
850	D1	47.5	47.5	5
	D2	45.0	45.0	10
	D3	42.5	42.5	15
	D4	40.0	40.0	20
900	E1	47.5	47.5	5
	E2	45.0	45.0	10
	E3	42.5	42.5	15
	E4	40.0	40.0	20

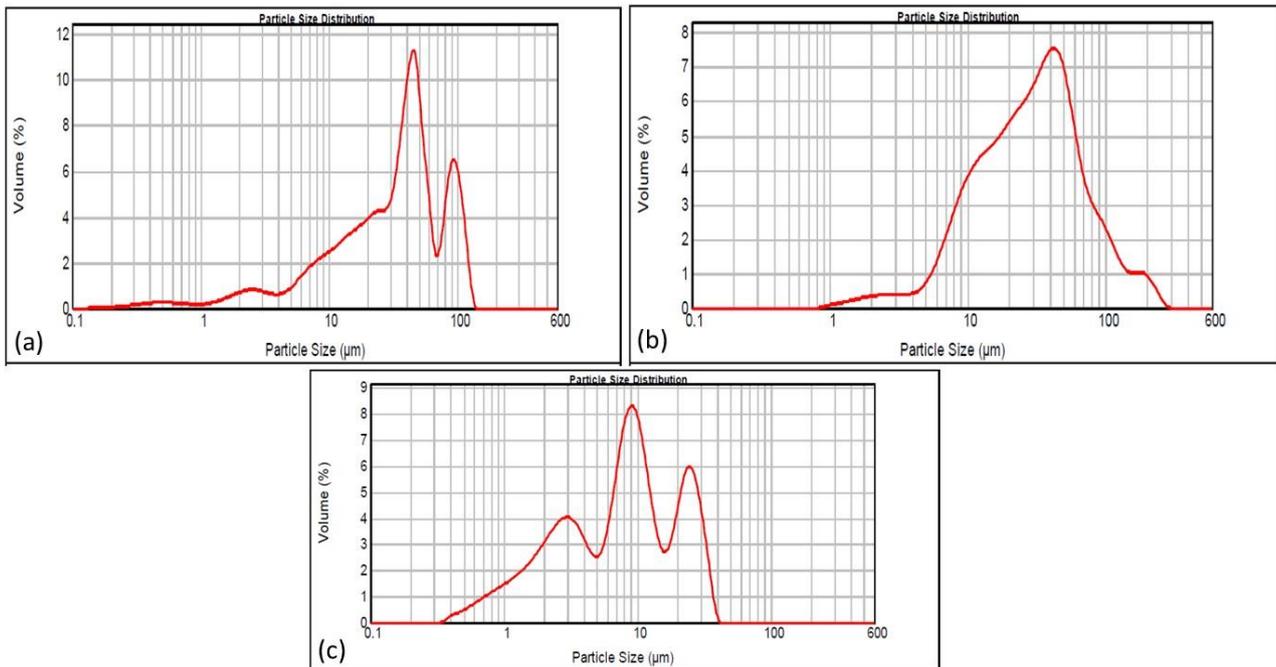
### 3. Results

#### 3.1 Particle Size Distribution Analysis

The analysis of frequency distribution and particle size indicates the amounts of that particular particle size exist in respective particle size intervals. In ceramic, the information on particle size is one of the important factors that affects the whole processing until achieving the properties of the final product. Finer particle size required shorter and lower sintering temperatures [8]. The higher specific surface area of the finer particle is also able to improve powder flow ability and improve forming reactive material [10]. Thus, more densified glass ceramic can be obtained. The effect of particle size will influence the sintering mechanism.

Figure 1(a) shows the distribution of particle size of soda lime silicate glass (SLSG). The dominant size of SLSG is in the range between 31.53 µm to 40.24 µm. This particle size is in the minimum range of commonly used size. The particle size of spent bleach earth (SBE) presented in Figure 1(b). A figure shows SBE powder having a particle size of < 45 µm in the range of 9.30 µm to 35.05 µm. About 97%

of particle size is within the size  $< 45 \mu\text{m}$  with maximum distribution is in the range between  $25.0 - 29.96 \mu\text{m}$ . These  $< 45 \mu\text{m}$  of particle size is also still in the range of commonly used size. Figure 1(c) shows the distribution of particle size of the eggshell powder. The dominant size of the eggshell powder is in the range between  $7.29 \mu\text{m}$  to  $11.87 \mu\text{m}$ . The maximum distribution is in the range between  $8.06 - 10.62 \mu\text{m}$ . These particle sizes are in the minimum range of commonly used size.

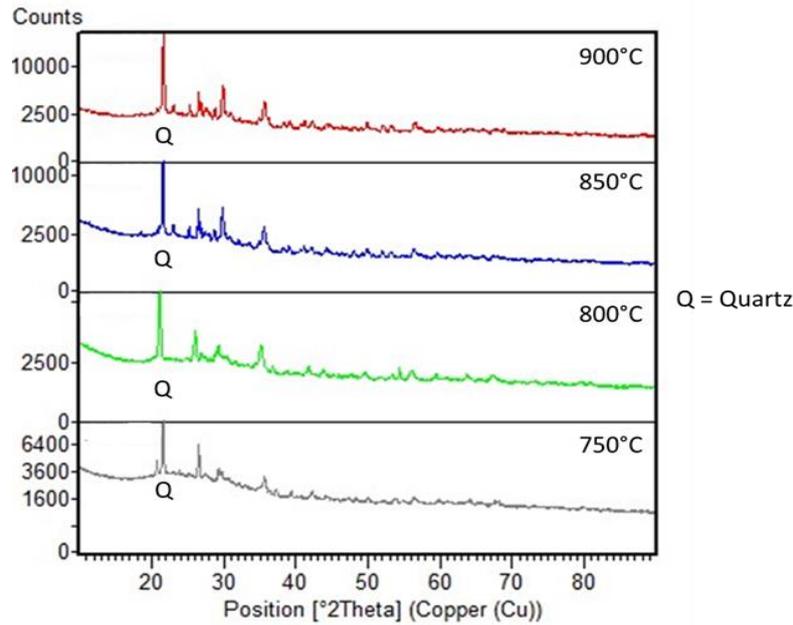


**Fig. 1.** Particle size of (a) soda lime silicate glass (SLSG), (b) spent bleach earth (SBE), (c) eggshell

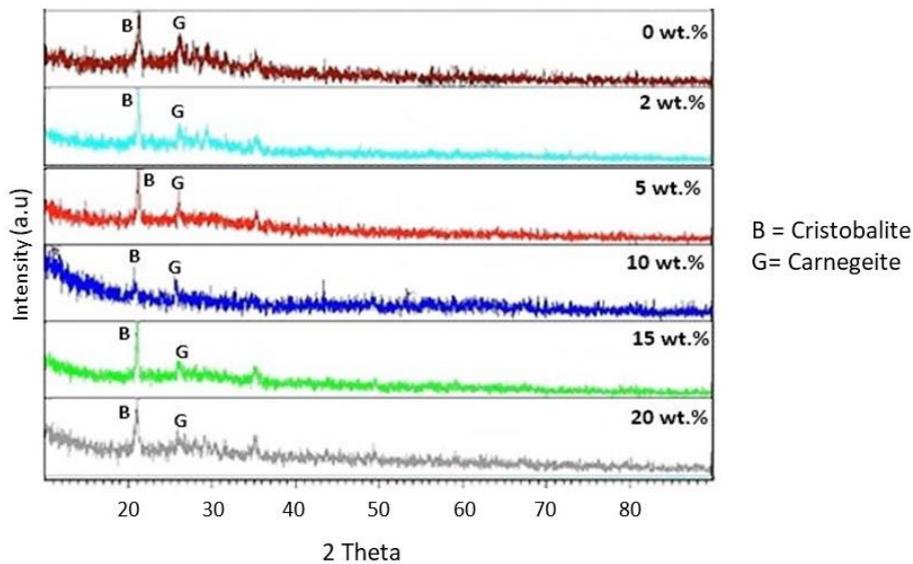
### 3.2 Phase Analysis

The crystalline phases presented in the sintered composite were identified by XRD. Figure 2 shows the XRD results for green glass ceramic without filler at 55 wt.% of SLSG and 45 wt.% of SBE. Most of the samples have 2–4 peaks for each sample, and the compounds are on the same peak. The compounds in samples are quartz and cristobalite. Cristobalite came from the soda lime silicate glass (SLSG). Figure 2 shows that the sample at  $900^\circ\text{C}$  had the highest intensity at a peak of  $21.3$  at position ( $^\circ 2\theta$ ), which is the  $\text{SiO}_2$  phase. Based on Figure 2, it is shown that by increasing sintering temperature, the higher the crystalline peak appears from the samples. The main crystal phase at that peak is quartz ( $\text{SiO}_2$ ). However, from the XRD results, it can be seen that not all samples consist of the quartz compound. This is because of the loss of ignition (LOI). The other elements in these samples are silicon oxide ( $\text{SiO}_2$ ).

The crystalline phases presented in the sintered composite were identified by XRD. Figure 3 shows the XRD results for the bentonite samples GB1, GB2, GC1, GC2, GC3, and GC4. Most of the samples have 2–5 peaks for each sample, and the compounds are on the same peak. The compounds in samples are cristobalite, coesite, anorthite, and carnegite. Cristobalite came from the SLG and BSG glasses. Figure 3 shows that GC3 had the highest intensity at a peak of  $21.6$  at position ( $^\circ 2\theta$ ), which is the  $\text{SiO}_2$  phase. The main crystal phase at that peak is cristobalite ( $\text{SiO}_2$ ). However, from the XRD results, it can be seen that not all samples consist of the cristobalite compound. This is because of the loss of ignition (LOI). The other elements in these samples are carnegite ( $\text{NaAlSiO}_4$ ), silicon oxide ( $\text{SiO}_2$ ), and coesite ( $\text{SiO}_2$ ).



**Fig. 2.** XRD pattern of green glass ceramic without filler at different sintering temperature



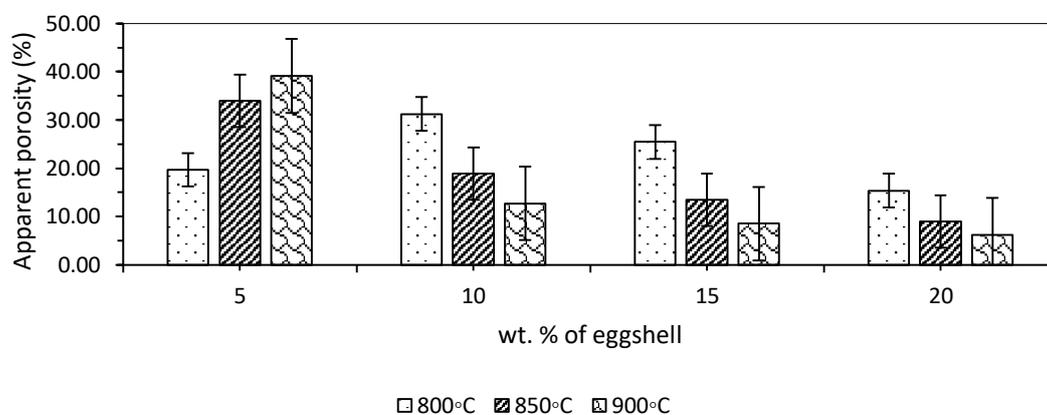
**Fig. 3.** XRD pattern of green glass ceramic with different bentonite content (wt.%)

### 3.3 Physical Properties

Figure 4-6 show apparent porosities, water absorptions, and bulk densities at different sintering temperatures and eggshells as filler loadings. Figure 7 shows bulk densities at different bentonite filler loadings. As shown in the figures, as the eggshell loading increased, the porosity and water absorption decreased for all sintering temperatures. As a study reported, the samples had high densities and were influenced by lower water absorption [10]. It was also found that the apparent porosity and water absorption results were inversely proportional to the bulk densities.

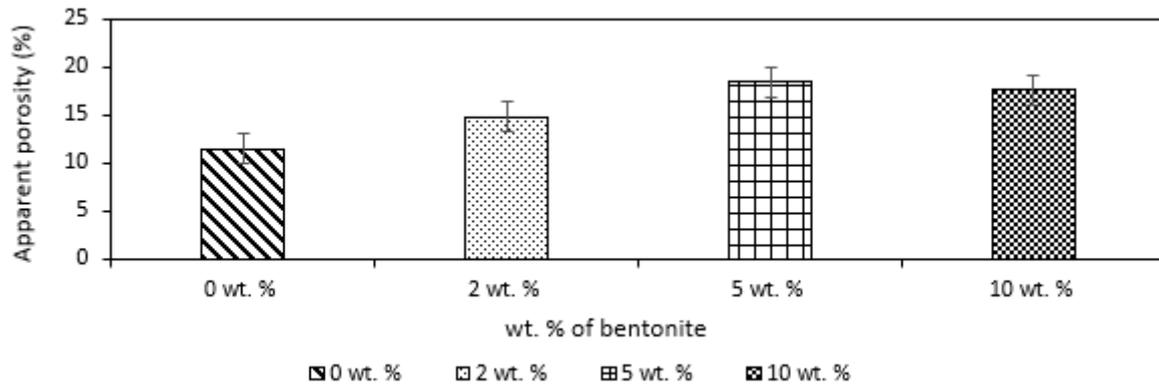
### 3.3.1 Apparent porosity

At higher sintering temperatures, the percentage of porosity on high weight percentage (wt.%) of filler was improved. As shown in Figure 4, the filler at 10, 15, and 20 wt.% contributed to the lower percentage of porosity when the sintering temperature increased from 800 to 900°C. The percentage of porosity of sample A2 and D2 with 10 wt.% of filler at 800 to 850°C decreased from 31.25% to 18.92% and further decreased to 12.73% at 900°C. The percentage of porosity of the sample with 15 wt.% of filler at 800°C and 850°C decreased from 25.45% to 13.46% and then to 8.51%. Meanwhile, at 20 wt.% of a filler, the percentage of porosity at 800°C and 850°C decreased from 15.38% to 8.96% and further decreased to 6.25% at 900°C. The lowest porosity of 6.25% was observed on the sample with 20 wt.% of filler at 900°C, while the highest porosity of 39.13% was seen on the sample with 5 wt.% at 800°C. Therefore, the sintering temperature and soaking time are important factors that affect densification due to low porosity [11].



**Fig. 4.** Percentage of apparent porosity of sintered glass composite at different sintering temperatures and eggshell loadings

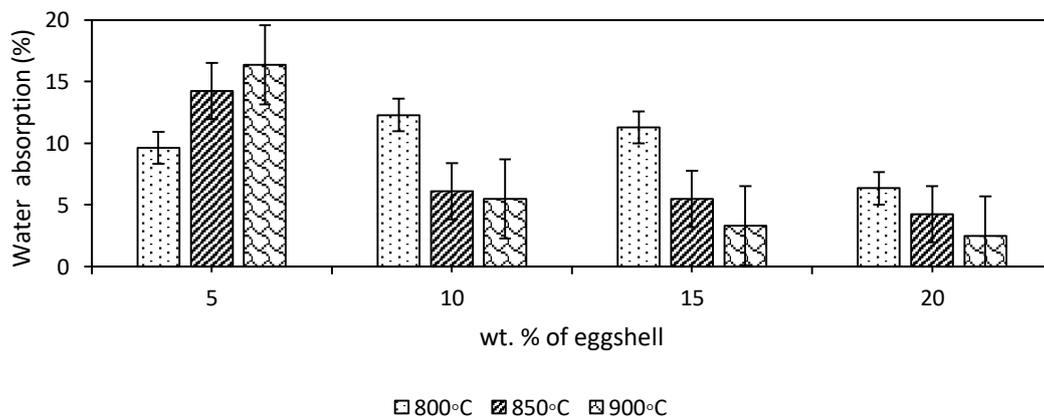
At higher weight percentage of bentonite, the percentage of porosity was decreased. As shown in Figure 5, the filler at 2, 5, and 10 wt.% contributed to the higher percentage of porosity when the weight percentage of bentonite increased from 2 to 10 wt.%. The percentage of porosity of sample GB2 with 2 wt.% of filler increased from 11.51% to 14.79% and further increased to 18.43 at 5 wt.% of bentonite filler. The percentage of porosity of the sample with 10 wt.% of filler decreased from 18.43% to 17.62%. The lowest porosity of 11.51% was observed on the sample with 0 wt.% of bentonite filler, while the highest porosity of 18.43% was seen on the sample with 5 wt.%. The influence of sintering temperature and soaking time contributed the densification where all samples show low porosity.



**Fig. 5.** Percentage of apparent porosity of sintered glass composite at different bentonite loadings

### 3.3.2 Water absorption

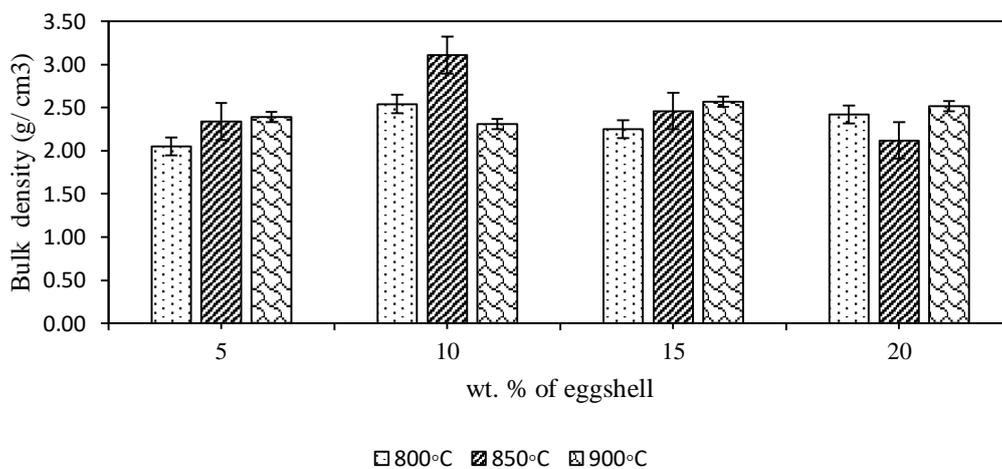
Figure 6 shows the percentage of water absorption. It can be seen in the figure that at high sintering temperatures, water absorption of sintered glass composite improved [12]. Water absorption and apparent porosity are related to each other. It was observed that the trends of apparent porosity and water absorption are similar. As the sintering temperature increased, the percentage of water absorption decreased at 10, 15, and 20 wt.% of filler, while at 5 wt.% of filler, the water absorption increased. The results of water absorption and porosity were parallel with each other. At 800°C, the water absorption increased when the filler loading increased from 5 to 10 wt.%. However, at 15 wt. %, the water absorption was slightly decreased. The water absorption percentage was 9.63% at 5 wt.% of filler loading, while it was 12.30% at 10 wt.% of filler. It slightly decreased to 11.29% at 15 wt.% of filler and to 6.35% at 20 wt.% of filler. At 800–900°C, when the filler loading increased, it decreased at 10, 15 and 20 wt.% of filler. The percentages of water absorption of samples at 5 wt.% of filler at 850 and 900°C were very high due to the high value of apparent porosity. The minimum percentage of water absorption of 2.48% was observed at 20 wt.% of filler at 900°C, while the maximum water absorption of 16.36% was observed at 5 wt.% of filler at 900°C. Therefore, if the percentage of eggshell powder added is high, the percentage of water absorption will be low, improving the appearance.



**Fig. 6.** Percentage of water absorption of sintered glass composite at different sintering temperatures and eggshell loadings

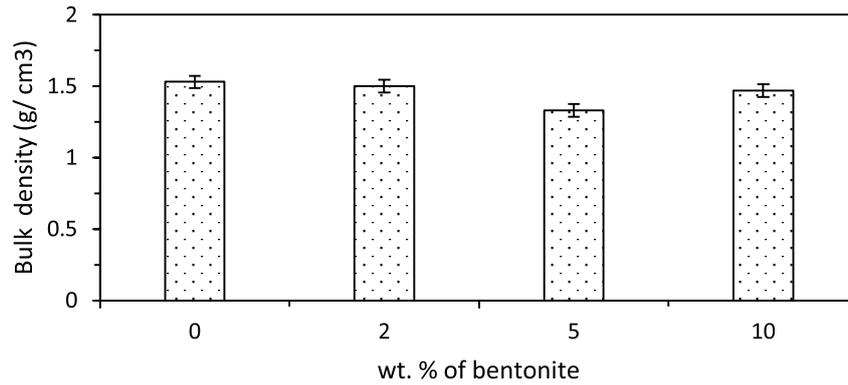
### 3.3.3 Bulk density

Figure 7 shows the percentage of bulk density at different sintering temperatures and eggshell loadings. The bulk density at 5 and 15 wt.% of eggshells as filler loading increased when the sintering temperature increased. At 20 wt.% of filler, at 900 °C, it was observed that lower porosity and water absorption contributed to the higher bulk density value of 2.52 g/cm<sup>3</sup>. However, the density at 10 and 20 wt.% of filler showed anomalous results – no uniform increase or decrease across sintering temperature. The average results at 10 wt.% of filler are between 2.31 and 3.11 g/cm<sup>3</sup>, while the results at 20 wt.% of filler are between 2.12 and 2.52 g/cm<sup>3</sup>. The results of the anomalous densities showed slight differences in porosity and water absorption. This could be because the liquid phase and its viscosity at sintering temperatures of 800, 850, and 900°C were not good enough to integrate the particles [13].



**Fig. 7.** Percentage of bulk density of sintered glass composite at different sintering temperatures and eggshell loadings

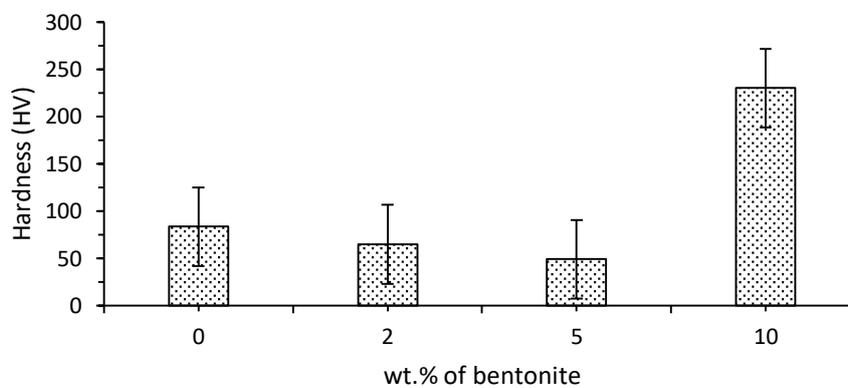
Figure 8 shows bulk density percentages for samples GB1, GB2, GC1, and GC2 with bentonite as filler loading. Sample GB1 has the highest density. It can be seen in the figure that with less percentage of bentonite, the samples had higher densities. One of the reasons might be the addition of the BSG and bentonite. SBE is expected to be denser than other materials in the compound, which tend to be evaporated. That is why the samples with high SBE percentage show high density where bentonite has a density of 0.593 mg/mm<sup>3</sup>. Besides that, the density of the material relies on the densities of its constituents. However, in the case of glass-ceramic, other factors also marginally influence the density of the final product [14].



**Fig. 8.** Bulk density of sintered glass composite at different bentonite contents (wt.%)

### 3.4 Microhardness Analysis

Samples with flatter surface and even dimension were chosen for microhardness analysis as shown in Figure 9. Glass composite with the 10 wt.% of bentonite composition has the highest mean value of hardness. One of the factors behind the low hardness value is the low pressure during the forming process. For samples GB1 and GB2, the compaction pressure was 17 MPa (1.5 tonnes), compared to 40 MPa (3.5 tonnes) for samples GC1 and GC2. High pressure during the forming process promotes bonding between the green pellets, which is expected to give a higher hardness to the composite. The presence of SLSG also in overall improves the hardness of glass composite in comparison to without it as reported in [7].

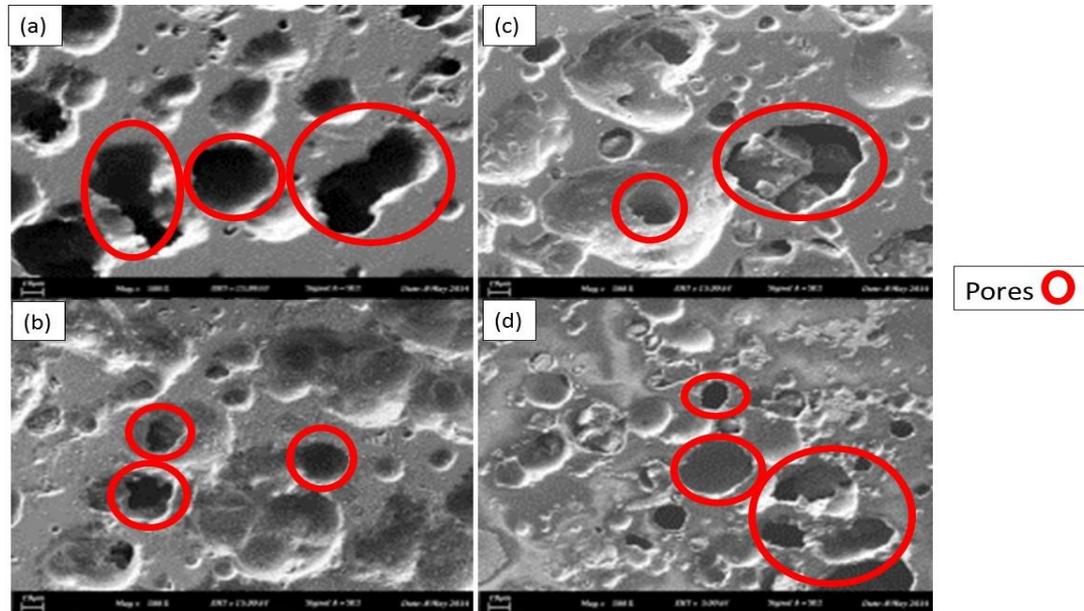


**Fig. 9.** Hardness for of sintered glass composite at different bentonite contents (wt.%)

### 3.5 Microstructure Analysis

Microstructure analysis was carried out to determine the presence of pores in the glass composite as well as surface condition of the sample with changes of their filler content and sintering temperatures (Figure 10-13). The presence of pores might contribute to the noises in the XRD graph as previously shown in Figure 2 and Figure 3, where the appearance of the peaks is not sharp and a good peak is hard to obtain. The noises present in the graphs show that the samples are amorphous. BSG and SBE are factors that might cause porosity. Based on [5], with the addition of SLSG weight

percent ratio, the sample had better surface finishing with less porosity. Their crystallisation and rigidity are higher when the sample surface is smooth due to the presence of Al and Na elements. These two elements caused the crystallisation of the glass composites. However, as the weight percent ratio of bentonite increased, the porosity reduced, as shown in Figure 10. This is because bentonite acted as a filler and occupies the pores as well as strengthens the bonding within the sample [15].

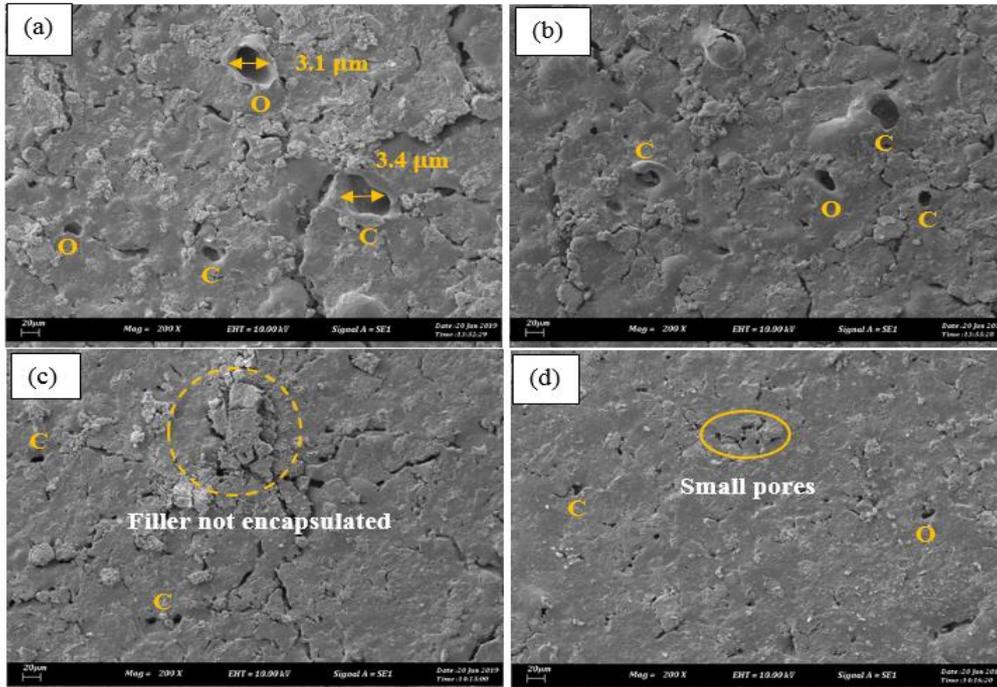


**Fig. 10.** SEM analysis of sintered glass composite filled with different bentonite content (a) 0 wt.% (b) 2 wt.% (c) 5 wt.% (d) 10 wt.%

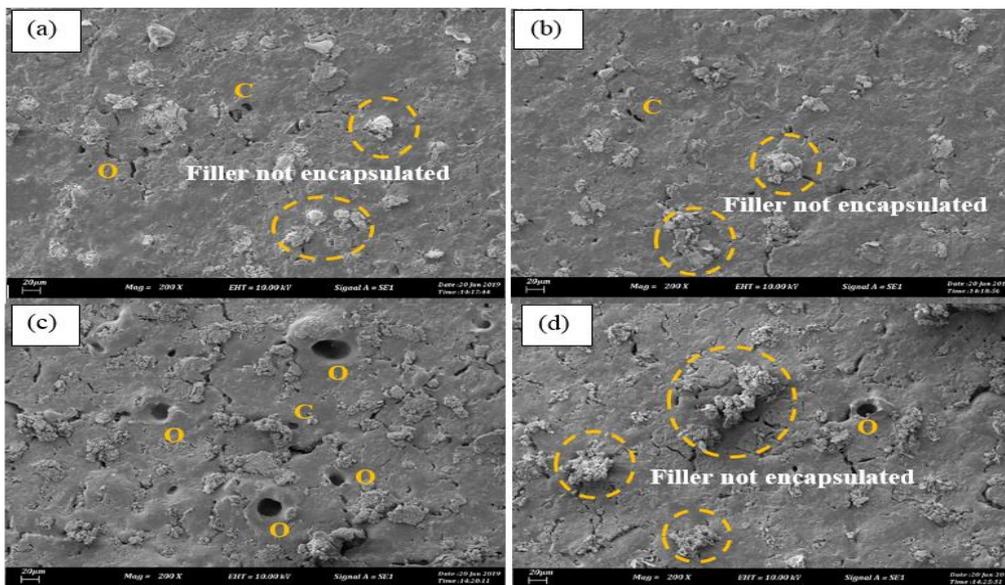
Figure 11 to Figure 13 show the SEM micrograph of sintered glass composite produced using different ES loading and sintering temperatures. When the filler loading increased at different sintering temperatures, the microstructures obtained were less uniform and had higher densities. Irregularly shaped pores were also observed. As illustrated in the figures, at 5 wt.% of a filler, the micrograph shows the formation of pores from 3.1 to 3.2  $\mu\text{m}$ , when the sintering temperatures increased from 800 to 900°C. This indicated that a microstructure failure due to the entrapment of glasses occurred on the samples [10]. In fact, the eggshells contain a significant amount of gas exchange pores [1].

As it can be observed in Figure 11–13, at 10 wt.% of ES loading, smaller pores, and dense surface were seen when compared to 5 wt.% of filler. At 20 wt.% of filler, fewer pores were observed when the sintering temperature increased. The microstructure supported the lower apparent porosity and a lower percentage of water absorption, as shown in Figure 4 and Figure 6. The micrographs of samples at 15 wt.% of filler at 800°C and at 5, 10, and 20 wt.% of filler at 850°C, as shown as dotted circle in the figures, revealed that filler particles were not fully encapsulated, and it looked like they were chipped out from the sintered glass composite. This situation contributed to the apparent porosity and water absorption of the samples. Besides that, open pores observed on the surface were due to incomplete viscous glass phase [16] and the decomposition of organic matter during the elevated temperature process. Eggshells are porous in nature. Therefore, the pores allow gas transfer from the inner side to the outer side of the eggshell [17]. This study reveals that a high sintering temperature (900°C) is required to have a uniform and densified microstructure with fewer pores. A good surface condition among the samples with no big pores was observed at 20 wt.% of filler at 900°C. A study has reported that the morphology was depicted to be smoother on the surface, as the

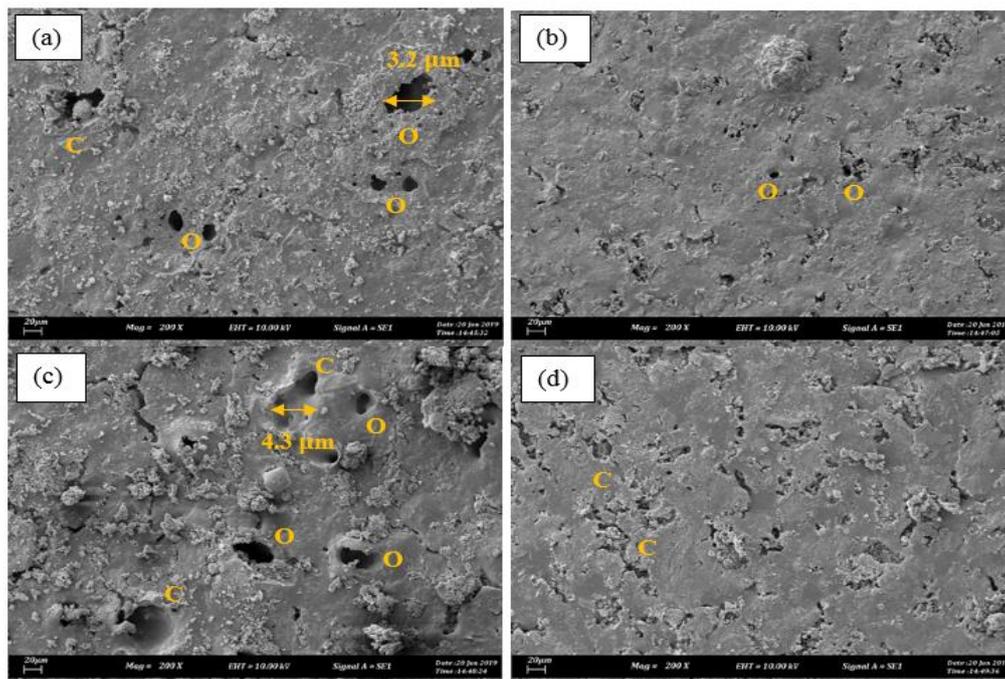
sintering temperature increased [17]. It can be seen that an increase in sintering temperatures and wt.% of filler loading influenced the microstructure of sintered glass ceramic. The results showed that the densification of the eggshell improves with the increasing sintering temperature [18]. Densification is also a result of the crystallization process [14].



**Fig. 11.** SEM micrographs of glass composite sintered at 800°C with different egg shell content (a) 5 wt.% (b) 10 wt.% (c) 15 wt.% (d) 20 wt.% [ indicator: O – Open pore; C – closed pore]



**Fig. 12.** SEM micrographs of glass composite sintered at 850°C with different eggshell content (a) 5 wt.% (b) 10 wt.% (c) 15 wt.% (d) 20 wt.% [ indicator: O – Open pore; C – closed pore]



**Fig. 13.** SEM micrographs of glass composite sintered at 900°C with different eggshell content (a) 5 wt.% (b) 10 wt.% (c) 15 wt.% (d) 20 wt.% [ indicator: O – Open pore; C – closed pore]

#### 4. Conclusions

It was observed from XRD studies that cristobalite initially precipitated, and when the loading SBE increased to 25 wt.%, phases coesite, cristobalite, and carnegite were precipitated. The effect of bentonite on the density and hardness of the composite was characterised by Vickers's, and the results revealed a significant increase in density and hardness of the fully crystallised system. SEM observation revealed that with more weight percent ratio of bentonite, the porosity was reduced. The effect of eggshell loading in sintered glass ceramic was successfully studied. The sample with 20 wt.% of ES loading, sintered at 900°C, produced the minimum water absorption of 2.48%, accompanied by an apparent porosity of 6.25% and a bulk density 2.52 g/cm<sup>3</sup>. Observations showed that the microstructure of the sintered glass composite varied significantly with the changes of ES loading at various sintering temperatures. This shows that sintered glass composite produced with a considerably high amount of waste loading can obtain acceptable physical durability. These findings encourage further investigations into the mechanical properties and standardised parameters for processing sintered glass composite.

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