Effect of Sintering Temperatures and Foaming Agent Content to the Physical and Structural Properties of Wollastonite Based Foam Glass-Ceramics

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Abstract:
This study aims to fabricate low-cost foam glass-ceramic using soda-lime-silica (SLS) glass waste and clamshells (CS) as foaming agent in content between 1 and 12 wt.% by conventional powder processing method. The samples were undergoing sintering process between 700 and 1000 °C with holding time of 30 minutes and characterized according to the physical and structural properties. Samples containing 3 wt.% CS treated at 800 °C show the greatest size of porosity. As the sintering temperature increased, the samples tend to become less dense. However, for the samples sintered at 900 and 1000 °C, the trend of the density changes because of the excess CO₂ gases generated during the heat treatment process promotes an increase in internal pressure, which results in the rupture of the pore walls. For linear expansion, for samples with a sintering temperature of 800 °C and higher, the increment of the temperature will lead to the decrement of linear expansion (%). As the sintering temperature increases from 700 to 800 °C, the water absorption (%) increases. However, the percentage of water absorption decreases with the further increment of sintering temperature. The XRD characterization showed the formation of wollastonite phase (CaSiO₃) and further revealed the formation of greater peaks of CaSiO₃ at the higher sintering temperatures. The results of compressive mechanical strength between 0.15 and 1.50 MPa indicate that the obtained glass-ceramic foams have potential for building material applications.

Keywords: Foam glass-ceramic; Wollastonite; Clamshell; Glass waste; Sintering.

1. Introduction

Solid commercial waste management from daily home usage and factory has become one of the crucial environmental issues to humankind nowadays [1]. Hence, authorities around the globe are encouraged to promote the recycling process to tackle the issue including the usage of waste disposal methods such as composition and sanitary landfills. This idea sparked as the natural resources become lesser besides the increasing solid waste produced

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from the industry’s activities every single day. Among the materials that contribute to the increasing industry solid waste, glasses stand as one of the materials which intriguing large interest in the recycling approach [2-4]. Thus, in many developed as well as developing nations, they enforced a lot of efficient community organizations to reduce and reuse solid waste materials.

In the recent years, building industries have been aware of the sustainability in their areas so as to lessen the impacts of construction process on the globe and to concern on the usage of natural resources in the processes, hence, they have been constantly showing efforts to enhance the construction’s products’ sustainability by using and adding natural resources as well natural wastes in the production of their products [5]. Recent studies had been conducted to deal with these problems and the invention of foam glass-ceramic which is a porous material formed from two different phases; gaseous phase and solid phase has been seen as the solution for these problems [6-8]. However, the cost of raw materials for foam glass-ceramic has become the biggest obstacles in the massive usage of foam glass-ceramic in the construction industry, thus, there are a lot of foam glass-ceramic production plants where the raw material for their foam glass-ceramic is approximately 98 % from post-consumer waste glass especially soda-lime-silica (SLS) glass as these material have an idealistic glass-forming nature compared to other several conventional glass systems. [9].

Besides, a clamshell which can be found abundantly by the seashore, will act as the agent for foam formation in the foam glass-ceramic which will help in reducing the production cost of foam glass-ceramic considering that clamshell contains a large amount of CaO [10]. Besides possesses a higher mechanical, concoction and warm solidness than the polymeric foams, foam glass-ceramic also have an exceptional arrangement of properties such as being lightweight, unbending, solidify tolerantly, non-flammable thus, foam glass-ceramics are seen to be a more suitable solution for thermal and acoustic insulation [11]. Moreover, foam glass-ceramic helps to facilitate the construction process apart from having low vehicle costs as it is simpler to deal with, cutting, and penetrating, in addition to it is promptly joined with cement.

A large amount of SLS waste was thrown away occupying landfill without realizing the recycling potential of SLS, therefore, foam glass-ceramic has been proposed as a promising recycling method. Besides, these works also investigate the chang es various heat treatment temperatures and composition of CS will bring onto the physical and structural properties of foam glass-ceramics.

2. Materials and Experimental Procedures

2.1 Materials

SLS glass waste was obtained by collecting the same Life brand sauce bottles from the recyclable waste bank located at Sri Serdang, Selangor, Malaysia and CS waste was gathered from cafes around Sri Serdang, Selangor, Malaysia. After the waste sauce bottles and CS were cleanly washed, they were left to dry under the Sun, then, those glass bottles and CS were crushed to small pieces using a stainless steel plunger. Next, by using a ball milling jar, the glass pieces were milling 50 rotations per minute (rpm) for an hour to obtain smaller glass and CS pieces. After that, using pestle and mortar, those pieces were finely grounded to obtain fine SLS glass and CS powder. Next, by using a 45 µm sieve, the fine powder was sieved to obtain the size of less than 45 µm (≤ 45 µm).

2.2 Sample preparation

The 45 µm sizes of raw materials which are SLS glass powder and CS powder were mixed homogeneously using ball milling jar with weight percent of CS; 1 wt.%, 3 wt.%, 6
wt.%, 9 wt.% and 12 wt.%. The milling process with 50 rotations per minute (rpm) took place for around 2 hours to ensure both materials were mixed well. Then, the mixture was taken out and weighed for 1 gram using an analytical balance. Next, the 1-gram mixture was inserted into the hydraulic press for the pelletizing process. For each pellet, 5 ton of load was pressed onto it for 10-15 minutes, which will then produce a pellet with the diameter of 13 mm. At the end of the pelleting process, there are 5 different batches of pellets according to raw material composition; 99 wt.% SLS + 1 wt.% CS, 97 wt.% SLS + 3 wt.% CS, 94 wt.% SLS + 6 wt.% CS, 91 wt.% SLS + 9 wt.% CS and 88 wt.% SLS + 12 wt.% CS. After that, the samples were taken for sintering process with 4 different sintering temperatures from 700-1000 °C. During the treatment, foam glass-ceramic were left to cooling constantly at certain temperatures for 30 minutes after reaching the required temperatures.

2.3 Characterization

X-ray fluorescence (XRF) was used to analyze SLS and CS powder in order to identify chemical composition in the both raw materials. Sizes of the foam glass-ceramic samples were observed after the sintering process. The samples are being cut into half, presenting the cross-section of the samples, so that, the presence of porosity in each sample can be observed and studied. Archimedes principle was applied in order to calculate the densities of the glass and glass ceramics. Essentially, the masses of the sample can be calculated either by using the weighing machine in air or immersion into the water. The differences between the real mass measured in air and the mass that had been displaced by the sample in water is equivalent to the apparent mass of the sample. As indicated by Archimedes principle, the water dislodged by the sample is proportionate to the mass of the example. In beginning stage, all the samples were weighed by using the weighing machine in the air giving their weight in air ($W_a$). Then, submerse the sample into liquid (water), to obtain the weight of displaced fluid. Then, the density of the sample (g/cm$^3$) can be calculated by the formula below:

$$\rho = \frac{W_a}{W_a-W_b} \rho_b, \quad (1)$$

where $\rho$ is the sample’s density, $W_a$ is the heaviness of sample in air, $W_b$ is the weight of displaced fluid after immersion of sample and $\rho_b$ is the water’s density which is 1 g/cm$^3$. Gram per centimeter cubic (g/cm$^3$) is the unit used for density.

The extension of materials cause by heat treatment procedure is identified with the pace of vitality transferred. The linear expansion percentage of the treated foam glass-ceramic samples has been determined using the Vernier caliper by measuring the diameter of the foam glass-ceramic before and after heat treatment so as to investigate the physical impact of heat treatment process on foam glass-ceramic, by means of the following formula:

$$\%\text{ linear expansion} = \frac{D_f - D_i}{D_i} \times 100\%, \quad (2)$$

where $D_i$ is diameter before heat treatment and $D_f$ is diameter after the sintering process. The samples are dried in an oven (heat treated) at 80 °C for an hour before left to cool down to prepare for water absorption test. Next, the samples are gauged and immersed in water at following conditions, 23 °C for a day. Samples were taken out of the water after 24 hours, patted dry with a cloth before weighed. Water absorption is defined as the increase in weight percent (%) and is measured using the formula:

$$\%\text{ water absorption} = \frac{\text{wet weight} - \text{dry weight}}{\text{dry weight}} \times 100\% \quad (3)$$
In this study, the foam glass-ceramic samples were squashed into fine powder using pestle and mortar after the heat treatment process. Packets of 1% and 3% clamshell composition of foam glass-ceramic samples’ powder were then sent for the XRD test. The selected range of 2θ angles (degree) for the XRD analysis was specified within Bragg angle (20-80°). By using the XRD machine from MPD X’PERT PRO PANalytical (Philips, PW 3040/60) model with copper, Cu Kα radiation at 40 kV and 30 mA of the input current, the crystalline phase of the samples was analyzed. The results were examined using PANalytical X’Pert High Score software. To determine the mechanical strength of the samples, compression tests using Universal Testing Machine (INSTRON 5566 model) were performed on 20 disc samples (sample after sintering process) with loading speed of 1 mm/min.

3. Results and Discussion

Raw materials, SLS glass and CS were chemically analyzed by using Energy Dispersive X-ray Fluorescence (EDXRF). The elemental analysis was analyzed in oxide form as tabulated in Table I. The chemical composition of the SLS glass waste in this research is similar to the SLS glass bottles. The CS used as blowing agent mainly contained of calcium carbonate (CaCO₃). The main constituents of the precursor glass such as SiO₂, CaO and Na₂O account 95 wt.% of the composition while the remaining percentage of other oxide elements are Al₂O₃ and MgO of the total composition precursor glass.

Tab. I Chemical composition of SLS glass and CS.

| Raw materials | SiO₂  | CaO   | Na₂O  | Al₂O₃ | MgO   | Others |
|---------------|-------|-------|-------|-------|-------|--------|
| SLS           | 70.5±0.1 | 11.3±0.1 | 12.5±0.1 | 2.8±0.1 | 2.0±0.1 | 0.9±0.1 |
| ES            | -     | 54.9 (97.2)±0.1 | 0.4±0.1   | -     | 0.8±0.1 | 1.6±0.1 |

a The value in original represents the CaCO₃ content in the clamshell.

Based on Fig. 1a, sample consists of 1 wt.% of CS at sintering temperature of 800 °C gives out the largest size out of the entire samples. Meanwhile, the smallest size of the sample is the 6 wt.% CaCO₃ content sample with sintering temperature of 1000 °C. With 700 °C and 800 °C as the sintering temperatures, sizes of the foam glass-ceramic samples tend to increase regardless of the wt.% of the CS, where after that, the sizes of the samples started to shrink from 900 °C of sintering sample until 1000 °C. This pattern of samples’ sizes happen due to the carbon dioxide (CO₂) gas that were released during the foaming process for 700 and 800 °C sintered temperatures. During the sintering process at 900 and 1000 °C, the foaming process still occurs. However, because of drop in the glass consistency and optional impacts which is the pore coalescence and pore opening, the samples’ sizes will only decrease as the sintering temperature increases [12].

Observing from Fig. 1b, highest amount of presence of porosity can be discovered at the 800 °C heat treatment temperature’s pellet due to the high percentage of CO₂ from the clamshell getting released during the foaming process which produces the gas matrix within the foam glass-ceramic. At heat treatment temperature of 900 °C, the porosity decreases. As the glass viscosity at this temperature begins to drop, even though the foaming process still
occurs, the porosity of the sample will decrease [12]. However, at 1000 °C heat treatment temperature, the size of the foam glass-ceramic gets really small, the porosity for the pellets also hard to be found and the color of the sample becomes different; dark yellow color. This is because as the softening temperature becomes greater, the viscosity of the liquid glass will also become greater which affect the pores with small diameters to become moderately hard to grow up [13]. This pattern works for 1 wt.%, 3 wt.%, 9 wt.% and 12 wt.%. However, for 6 wt.% foam glass-ceramics, tiny size of pores can be detected for sample with 700 °C heat treatment temperature. However, only a slight change in the size of porosity can be detected for 800 °C sample. An amount of CaCO$_3$ in this pellet stays accessible for foaming on the surface, nonetheless, because of a high measure of gas formed from the carbonate disintegration, the interior becomes inhomogeneous, thus causing the porosity cannot be formed efficiently [12].

![Fig. 1. a) The green size of foam glass-ceramic pellets, b) The porosity inside foam glass-ceramic pellets.](image)

![Fig. 2a. Graph of density against CS composition (wt%) for various heat treatment temperatures (°C).](image)
As shown in Fig. 2a, due to the crystalline filled structure of samples, the density of the foam glass-ceramics tend to decrease after the heat treatment process at room temperature [14]. The densities of the pellets are also decrease with the addition of CaCO₃ content. This is because the high density of pure glass becomes lower with the addition of lower density of clamshell. Thus, higher addition of CaCO₃ will affect the density of the pellets to get lower. Density as can be observed from the Fig. 2b, increases from 1 % CS content’s sample to higher wt.% CS content. This is because the glass matrix of the pellet gets compacted. However, as the content of CaCO₃ becomes greater, the density of the sample tend to decrease as the increase of CaCO₃ addition which acts as the blowing agent in the samples control the quantity of carbon dioxide released during sintering process which then formed the porous effect in the structure [15]. The formation of porosity in the samples’ structure will affect the volume of samples as well as their density. Not to mention, the heat treatment temperature also supports the formation of the foam glass-ceramic. The density of samples tends to decrease by rising the temperature of heat treatment process and keeping the holding time constant [16]. By contrasting the structure of pellets at 800 and 700 °C with 30 minutes holding time, it is demonstrated that the foam glass-ceramic acquired at 800 °C presents an inhomogeneous structure with pores of large dimensions and an expansion of the quantity of channels interfacing the pores. With the softening temperature of the glass being at 826 °C, viscosity of the glass at 800 °C is lower compared to that of 700 °C as the temperature make it harder to control the expansion of the gas besides the fragmented decomposition of the foaming agents associated to a low viscosity of the glass matrix at 700 °C which causing higher density of the sample [16]. Contingent upon the sintering temperature and the amount of the foaming agent, the foam glasses may have various structures and sizes [14]. The glass surface region which in contact with the foaming agent diminished at low temperature which bringing about lower decomposition pace of foaming agent though, at higher temperature, the heat treatment procedure proceeds quicker and the glass respond promptly with the foaming agent [14]. During the foaming procedure, the calcium carbonate particles exist in the foaming agent break down to the oxide with the concurrent arrival of carbon dioxide (CO₂) [11]. The CO₂ gas will at that point be caught in the molten glass mass and force the molten glass mass to expand and while cooling, the molten glass mass is solidified and their cell or permeable structure is formed [17]. However, due to dropping of glass viscosity and presence of
secondary effects such as pore coalescence and pore opening, further in heat treatment (900-1000 °C) will cause an increase in density.

The linear expansion of the foam glass-ceramic samples against heat treatment temperature was shown in the Fig. 3. For 1 % CS composition, starting from 700 °C, the graph showed an increasing pattern in the linear expansion of the samples. This pattern lasts until 800 °C where the linear expansion begins to decrease. This is due to the porosity formed during the foaming process with heat treatment temperature of 700 °C until 800 °C caused by the CO₂ gas released during the sintering process. Thus, the samples tend to expand according to the viscous liquid phase formation. In the range of temperature approximately between 700 and 900 °C, the CO₂ gas released related to the calcium carbonate decomposition (in this study, from clamshell) is quite similar to the commercial calcium carbonate [18]. At temperature of 900°C and higher, the viscosity of the pellets begins to decrease which causing minimum porosity formed in the structure and the samples become compact. The glass viscosity is adequately low at 900 °C onwards for expansion to occur and consequently, for porosity to form. In this temperature, glass reaches a more noteworthy shrinkage and higher densification [18]. However, linear expansion of sample at 900 °C is greater than that at 700 °C. This pattern is exactly the same for samples with 6 wt.%, 9 wt.% and 12 wt.% of CS composition. However, for 3 wt.% CS composition, the pattern is different from the 1 wt.% CS composition, where sample at 700 °C heat treatment temperature shows the lowest percentage of linear expansion. Followed by sample at 1000 °C (second lowest percentage of linear expansion), sample at 900 °C and the largest percentage of linear expansion for 3 wt.% CS expansion is the sample at 800 °C.

From Fig. 4, the percentage of water absorption becomes higher as the temperature increases from 700 to 800 °C. However, this trend changes when the sintering temperature keeps increasing where percentage of water absorption at 900 °C is lesser than that of 800 °C. Obviously, sample sintered for 1000 °C produces a much lower percentage of water absorption compared to 900 °C. The general decrease of water absorption percentage with increasing sintering temperature is quite normal, since the viscous flow of the fluid stage given by glass softening is relied upon to seal the open porosity, at the surface [19]. The

Fig. 3. Graph of LE (%) against sintering temperatures for different CS composition (wt %).
sealing impact ought to be to some degree supported by the adopted firing strategy (direct presentation of samples in the furnace at chosen temperatures), with the surfaces encountering a lot quicker heating [19]. The decrease in the pace of water absorption with expanding heating temperature (900-1000 °C) shows the event of local liquid stage sintering, which added to a diminishing in pore volume and the water absorption rate [20]. Open pores are the factor affecting the ability of water absorption of the samples. Samples containing lower CaCO\textsubscript{3} content will have a lower molten glass’ viscosity. The pores created were anything but difficult to move; in this manner, some neighboring pores effectively shaped into bigger pores, and the water absorption increased [13]. That is why water absorption percentage of 3 % CS sample is lower than that of 1 wt.% CS sample and so on.

![Graph of water absorption (%) against sintering temperature for selected composition of CS.](image)

**Fig. 4.** Graph of water absorption (%) against sintering temperature for selected composition of CS.

From Fig. 5a, XRD spectrum of room temperature the sample shows that most of the spectrum presents the amorphous nature of pellet with a sharp peak of calcium carbonate. The increment of the heat treatment temperature to 700 °C causing the peak of CaCO\textsubscript{3} to get higher and a lot of tiny peaks of CaCO\textsubscript{3} can be observed. This is due to the insufficient energy supplied by the heat treatment temperature for CO\textsubscript{2} to fully release. It is shown that as the sintering temperature increases to 800 °C, the samples become more crystallized. The peak intensity of CaCO\textsubscript{3} also seems to disappear as the heat treatment temperature has enough energy to trigger the reaction of CaCO\textsubscript{3} with glass (SiO\textsubscript{2}). In addition, the spectrum also shows the crystalline peak of SiO\textsubscript{2} (quartz), at 2θ = 22°. Small peaks of CaSiO\textsubscript{3} (wollastonite) can also be detected in the XRD spectrum for 800 °C. This indicates that the reaction between SiO\textsubscript{2} and CaO has taken place. Despite increasing the heat treatment temperature to 900 °C, the XRD spectrum still shows the sharp peak of SiO\textsubscript{2} (quartz), however, with a lower intensity of peak compared to that of 800 °C. The peaks of CaSiO\textsubscript{3} (wollastonite) also tend to increase in intensity as the sintering temperature increases.
Fig. 5a. X-ray diffraction pattern of 1 wt.% CS composition foam glass-ceramic pellets with different sintering temperatures.

Fig. 5b. X-ray diffraction pattern of 3 wt.% CS composition foam glass-ceramic pellets with different sintering temperatures.

The changes in trend are affected by the higher heat treatment temperature; more energy is supplied for the reaction to take place; more CO₂ was released and more products from CaO-SiO₂ reaction were produced, which lead to the reduction of the amount of SiO₂ in the pellet. And of course, 1000 °C of heat treatment will produce higher peaks of CaSiO₃, thanks to more energy supplied for the interaction between CaO-SiO₂ to happen. This pattern is exactly the same with 3 wt.% CS content foam glass-ceramic. However, by comparing the XRD
spectrum from Fig. 5a and 5b, it was concluded that the intensity of CaSiO$_3$ (wollastonite) peaks from 3 wt.% CS composition samples in Fig. 5b is greater than 1 wt.% CS content samples as shown in Fig. 5a, comparison is made by comparing between the samples with the same sintering temperature. This is because at a higher percentage of CaCO$_3$ addition, there are changes in the XRD analysis [12].

FESEM revealed the microstructure of foam glass-ceramic at 800 and 900 °C of temperature as shown in Fig. 6. As we can see from Fig. 6a and 6b the pores are well formed at 800 °C and as the sintering temperature increase to 900 °C, the number of pores become reduced and it clearly show the densification of glass. It is agreed with the decompose rate of calcium carbonate as the result indicate that there is no reaction after 800 °C. Furthermore, the low in viscosity affect the pore formation because the pores tend to rise to the top of mold consequently cause inhomogeneous pore distribution. When low in temperature, the viscosity of melt glass is increase; making difficulty of gas expansion hence there is little changing the volume of the foam glass-ceramics.

Fig. 6. Microstructure of closed pore for 3 wt.% CS (a) 800 °C and (b) 900 °C.

Fig. 7. Compressive strength ($\delta_c$) of glass-ceramic foams with different contents of CS at different sintering temperatures.
Fig. 7 shows the value for compressive strength ($\delta_c$) test with different contents of CS at different sintering temperature. Generally, mechanical strength of the foam glass-ceramics is greatly influenced by the density and the porosity inside the samples. Normally, the glass-ceramics sample with lower density and higher porosity will have thinner pore walls. From Fig. 7, the sample with the lower density and higher porosity (1 wt% CS) sintered at 800 °C presented the lowest compressive strength value ($\sim$0.32 MPa) and the foam glass-ceramic sample with one of the highest density and lowest porosity values (6 wt% CS) sintered at 1000 °C showed the highest mechanical resistance ($\sim$1.2 MPa).

4. Conclusion

Foam glass-ceramics has been successfully fabricated from waste material which made up from SLS glass and clamshell by solid state process as a promising material to be used in construction industry. According to physical appearance, Archimedes technique, linear expansion measurement, water absorption test and XRD analysis, sintering temperature and percentage of CS content indeed played a huge role in obtaining the best foam glass-ceramic. The FESEM of the glass-ceramic foams showed glass structures with partially open porosity (interconnected pores) and the mechanical strength of the foam glass-ceramics is greatly influenced by the density and the porosity of the glass-ceramic samples. After thorough observation, it is concluded 1 wt.% CS content sintered at 800 °C is the optimum condition for producing a less dense, great water absorption percentage and large porous foam glass-ceramic.

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Сажетак: Циљ овог рада био је добијање јефтине пене стакло-керамика употребом сода-лајм-силике као стакленог отпада и шкољки садржаја између 1 и 12 теж. % као агента за прањење пене, конвенционалном методом процесирања праха. Узорци су синтеровани између 700 и 1000 °C са временом задржавања од 30 минута и охарактерисани су физичка и структурна својства. Узорци са 3 теж. % шкољки синтеровани на 800 °C показују највећу порозност. Са порастом температуре синтеровања, узорци као да показују мању густину. Међутим, за узорке синтероване на 900 и 1000 °C, тренд мењања густине се мења, услед више CO₂ гаса који се генерише током процеса синтеровања и повећава унутрашњи притисак у узорку, што резултује руптуром зидова пора. За узорке синтероване на 800 °C и више, пораст температуре доводи до смањења линеарне експанзије. Са порастом температуре од 700 до 800 °C, апсорпција воде расте. Проценат апсорбоване вода опада са даљим порастом температуре синтеровања. XRD указује на формирање воластонита.
(CaSiO₃) и на пораст интензитета пикова воластонита са порастом температуре синтеровања. Резултати отпорности између 0,15 и 1,50 MPa указују на то да добијена пена стакло-керамика може да се примењује као материјал у грађевинарству.

Кључне речи: пена стакло-керамика, воластонит, шкољка, стаклени отпад, синтеровање.

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