



A Sustainable Building Material Developed from Low-Temperature Sintering of Mining Waste with an Alkali-Silicate Solution

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Keywords	Abstract
Glass-Ceramic Foam	The development of glass-ceramic foam has received significant attention in building and construction, given its potential for sustainability. This study investigated the low-temperature route of fabricating glass-ceramic foams from mining waste. The feasibility of one-step or chemical-aided sintering of glass-ceramic foams using granite powder, a naturally sourced mining waste, rather than using already heat-treated wastes such as glass and fly ash has been explored in this study. Glass-ceramic foam samples were synthesized from a homogenous blend of constant percentage by weight of granite-clay mix with varying amounts of alkali-silicate solution. The influence of the alkali-silicate solution on the physicomechanical and microstructural properties of the synthesized samples sintered at 850°C was investigated. The results showed water absorption of 9.5-33.3%, apparent porosity of 18.2-56.7%, bulk density of 1.7-1.91 g/cm ³ , and compressive strength of 20.7-26.3MPa. The glass-ceramic foam developed in this research can be suitably used for the thermal insulation of buildings.
Sustainable Building Material	
Physciomechanical Properties	
Microstructure	
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1. INTRODUCTION

Glass-ceramic foams are gaining significant research attention given their exciting properties, including low density, high perviousness, and appropriate moisture absorption (Khamidulina et al., 2017). Glass-ceramic foams are porous materials with many valuable applications as thermal and sound insulators, architectural panels, filters, gas sensors, absorbers, lightweight concrete aggregates, and more (Dragoescu et al., 2018). Unlike polymeric foams such as polystyrene and polyurethane, glass-ceramic foams are nonflammable, non-toxic, and chemically inert; thus, they substitute for polymeric foams as building insulation materials owing to their ability to provide aid in reducing damages during a fire outbreak (Dragoescu et al., 2018). Apart from the unique features of glass-ceramic foams that qualify it as a suitable thermal insulation material in numerous applications, its cost-effectiveness is another area of interest. In this regard, current research efforts on glass-ceramic foams are channeled toward two essential areas: waste valorization and low-cost fabrication routes.

Zhang et al. (2022), Yu (2022), Hujova et al. (2020), Paunescu et al. (2020), Hisham et al. (2021), and Ma et al. (2018), prepared glass-ceramic foams based on industrial wastes showing the possibility of converting wastes into valuable materials at a low cost. Glass-ceramic foam production is commonly based on secondary wastes obtained from manufacturing industries, including waste glass, bottom ash, and fly ash. However, in developing countries with limited industries, using these industrial wastes for glass-ceramic foams' mass production requires much work. For instance, the extensive use of glass cullet is stunted by their insufficient amounts and the variation of the chemical composition (Ivanov, 2018). Given waste glass's inadequate

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availability, alternative raw materials, such as naturally-sourced minerals for glass-ceramic foam production, are necessary (Sedlačík et al., 2022). Hence, the utilization of primary or naturally sourced industrial wastes such as granite powder, which are abundantly obtainable and suitable for producing glass-ceramic foams, is crucial. Accumulating granite powder as mining waste can cause environmental problems (Arivumangai & Felixkala, 2014). Reducing environmental impact is, therefore, a significant issue for granite industries (Ayodele et al., 2014). This necessitated the use of granite powder in this research.

Different techniques have been used to fabricate porous glass ceramics, including gas foaming, phase separation, sol-gel, and sintering (M. H. Ibrahim et al., 2022). Among other methods, fabricating glass-ceramic by sintering technique is widely used since it is convenient and cheap (M. H. Ibrahim et al., 2022). Foaming by the sintering technique of glass ceramic foam production can be achieved through either two-step sintering or one-step sintering. The two-step method involves double heat treatments. The two-step sintering method of glass-ceramic foam production entails the preparation of base glass material at a higher temperature, followed by foaming at a much lower temperature (Kim et al., 2017).

On the other hand, the one-stage or one-step sintering mechanism of obtaining glass-ceramic foam is achievable without needing a prior heat treatment to prepare the glassy base. If done this way, the crystallization and the foaming procedure are performed in one step. The two-step method effectively controls the glass ceramic foams' pore structure (Chen et al., 2017). However, significant energy input is required for developing a glassy base material, meaning that more substantial venture expenses are involved than in the single-step method (Chen et al., 2017). Merging the two steps into one operation by mixing the raw materials and heating directly to form the foamed material is more economically satisfactory since the glassy phase development and its foaming is carried out in a one-step process (Ivanov, 2018). Therefore, using one-step sintering to synthesize glass-ceramic foam helps reduce production costs (Yuan et al., 2018).

One-step sintering can be classified into two types: (i) indirect one-step sintering and (ii) direct one-step sintering. Indirect one-step sintering involves the using waste glass powder with or without other wastes that are by-products of high-temperature industrial activities or combustion processes, such as fly ash and slag, and with or without foaming agent addition. Mustaffar and Mahmud (2018) prepared glass-ceramic foams from a mixture of waste glass, fly ash, and SiC (foaming agent). Ma et al. (2018) obtained glass-ceramic foam from coal fly ash and calcium carbonate (CaCO_3) sintered at four varying temperatures of 1100°C, 1150°C, 1200°C, and 1250°C. Lardizábal-G et al. (2020) also fabricated glass-ceramic foam from glass waste, 5-15% pumice, and 5% limestone sintered at different temperatures of 700°C, 750°C, 800°C, and 850°C, respectively. Hisham et al. (2021) obtained foam glass-ceramic from waste glass and ark clamshell sintered at temperatures of 700°C, 800°C, and 900°C, respectively. Zhang et al. (2022) developed glass-ceramic foam from a mixture of fly ash, bottom ash, and pickling sludge, with borax (fluxing agent) and CaCO_3 (foaming agent), sintered at 1180°C and annealed at 500°C.

However, direct one-step sintering of glass-ceramic foams involves the using natural inorganic materials such as mining wastes, amorphous silica, or other rock minerals as base glass material with the addition of a foaming agent. Tian et al. (2016) developed thermal insulating foams at 650°C to 1200°C using shale as a glass-former, feldspar (fluxing agent), SiC (foaming agent), and walnut shell (pore-forming or internal combustion agent). Chemical-aided fabrication involves using chemicals (such as NaOH and Na_2SiO_3) to facilitate the one-step sintering of glass-ceramic foams. Previous studies reviewed in this respect are presented as follows. Ivanov (2018) manufactured glass-ceramic foams from diatomite and 40% NaOH solution (foaming agent), constantly heated at 775°C. da Silva et al. (2019) prepared foam glass from glass waste using NaOH as the foaming agent. Owoseye et al. (2020) prepared glass foam from waste glasses using 15wt-% Na_2SiO_3 as the foaming agent. Sedlačík et al. (2022) developed glass-ceramic foams from diatomaceous earth (clay-rich waste) through a hydrate mechanism using 50 wt-% NaOH solution as the foaming agent. J. E. F. M. Ibrahim et al. (2022) fabricated glass-ceramic foams from zeolite-rich clay and sawdust based on alkali activation using 15wt-% NaOH.

To the best of the knowledge of the author of this study through literature review, only Odewole (2022) has used a mix of NaOH and Na_2SiO_3 as a foaming agent. However, Odewole (2022) used an agricultural waste (maize cob) as a pore-forming agent. The novelty of this study is based on evaluating the effect of an alkali-

silicate solution (obtained by mixing NaOH and Na₂SiO₃) on mining waste (granite powder) without adding any carbonaceous or combustible material as a pore-forming agent.

2. MATERIAL AND METHOD

2.1. Sourcing of Starting Raw Materials

Granitic powder, ball clay, sodium silicate (Na₂SiO₃), and caustic soda (NaOH) were used in this study. Dotmond Quarry, Ita-Ogbolu, Ondo State, Nigeria, provided the granite powder used as the primary glassy material. Ire Clay Products Limited, Ire Ekiti, Ekiti State, Nigeria, provided the ball clay used as the binder. Qualikems Fine Chemicals Pvt. Ltd. supplied the sodium silicate (Na₂SiO₃), which contained about 12% Na₂O and 30% SiO₂. May & Baker Ltd., Dagenham, England, supplied the sodium hydroxide (NaOH) pellets with a minimum assay of 98.9%. This study used the mixture of Na₂SiO₃ and NaOH to form an alkaline silicate solution as the pore-forming agent.

2.2. Sample Preparation

The granite powder and ball clay used in this study were screened to pass a 300µm British standard sieve. The granite powder-clay mix prepared by mixing granite powder and ball clay in a ratio of 3:1 served as the starting material for the glassy phase. The addition of ball clay was mainly to serve as a binder since granite powder is a non-plastic material. The Alkali-silicate solution prepared from the mixture of Na₂SiO₃ and 10M NaOH solution in a ratio of 1:1 served as the foaming agent. 10M sodium hydroxide has been found to exhibit desirable effects when used to prepare alkali activators in the production of metakaolin geopolymers (Jaya et al., 2018; Vitola et al., 2020). Three different samples of glass-ceramic foam were formulated by adding 15vol%, 20vol%, and 25vol% of the alkali-silicate solution to 100g of granite powder-clay mix, labeled as samples 1-3. The prepared samples were carefully mixed. The homogenized blend was transferred into steel molds (5 x 5 x 5 cm) and pressed at 10MPa into cubic shapes. The formulated samples were oven-dried at 110°C for 6hr., heat-treated in a gas-fired kiln at 850°C for 3hr., and held for 2hr.

2.3. Materials and Samples' Characterization

X-ray fluorescence analysis done with Skyray Instrument, Model: EDX3600B, to obtain the chemical composition of the granite powder and ball clay used in this study are presented in Table 1. The sintered samples' apparent porosity, bulk density, and water absorption (physical properties) were estimated according to ASTM C20-00 (2022). The samples' compressive strength (mechanical properties) was measured according to ASTM C240-97 (2017) using Instron 3369 universal testing machine. The samples' microstructural properties were investigated using Nikon SMZ745T Stereomicroscope.

Table 1. Chemical composition of granite powder and ball clay

Raw Materials	Constituent (wt%)										
	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	Fe ₂ O ₃	SnO ₂	Sb ₂ O ₃	LOI
Granite Powder	12.82	59.72	0.44	0.82	5.74	5.67	0.35	11.29	1.14	1.10	0.91
Ball Clay	21.60	58.15	0.21	0.71	2.30	0.15	1.81	14.40	-	-	0.67

3. RESULTS AND DISCUSSION

3.1. Physical Observation of Samples after Sintering

Different properties were perceived in the glass-ceramic foams' sintered samples. These properties were noted to have been impacted by the varying amounts of the alkali solution added to the granite powder-clay mix to facilitate one-step foaming by sintering. From the physical appearance of the samples, as shown in Figure 1, it was noticed that the foaming of the samples appears to increase with rising amounts of the alkali-silicate solution.



Figure 1. Glass-ceramic foams sintered at 850°C with different amount of alkali-silicate solution; **a)** 15vol%; **b)** 20vol%; **c)** 25vol%

3.2. Physical, Mechanical, and Thermal Properties

Figures 2a, 2b and 2c reveal an increasing trend in the apparent porosity and water absorption and a decline in bulk density of the sintered samples from 1 to 3, respectively, with the rise in the amount of alkali-silicate solution (ASS). Water absorption rose by 98% from 9.5% in sample 1 containing 15vol% ASS to 18.8% in sample 2 containing 20vol% ASS and by 77% from 18.8% in sample 2 to 33.3% in sample 3 containing 25vol% ASS. The apparent porosity increased by 83% from 18.2% in sample 1 to 33.3% in sample 2 and by 70% from 33.3% in sample 2 to 56.7% in sample 3. The bulk density decreased by 68% from 1.91g/cm³ in sample 1 to 1.78 g/cm³ in sample 2 and by 45% from 1.78 g/cm³ in sample 2 to 1.70 g/cm³ in sample 3. This trend could result from the rise in NaOH content resulting from increased ASS added to the glass-ceramic foam samples as the foaming agent. da Silva et al. (2019) observed that increased NaOH content reduced the density of the produced glass-ceramic foam but increased its open (apparent) porosity.

Figure 2d shows that the samples' compressive strength increased by 12% from 20.7 MPa in sample 1 to 23.1 MPa in sample 2 and by 14% from 23.1 MPa in sample 2 to 26.3 MPa in sample 3. The increasing values of compressive strength of the samples were supposed to correspond to increasing bulk density and decreasing porosity, which is generally expected according to Zakaria et al. (2020). However, in this study, there is a contrastive decrease in bulk density with a rise in compressive strength even though there was an increase in apparent porosity. It is noteworthy that a similar occurrence of contrast between compressive strength and bulk density of foam glass was noted by Owoeye et al. (2020), in which the samples' compressive strength and apparent porosity increased while the bulk density decreased. Although Owoeye et al. (2020) attributed this compressive strength-bulk density anomaly to the decline in the average pore size coupled with the rise in the quantity of liquid glassy phase attained at increased temperature, it can be observed that temperature is kept constant in this study, while varying the amount of foaming agent. Therefore, the compressive strength-bulk density anomaly observed in the samples could be due to the increasing tendency of the samples to vitrify at 850°C, forming a glassy phase which is accounted for by the rising amount of the alkali-silicate solution used as a foaming agent.

The results of the prepared glass-ceramics foam by Odewole (2022), involving the use of an agricultural waste (maize cob powder) and alkali silicate solution (mixture of Na₂SiO₃ and NaOH) as the pore-forming agent revealed water absorption of 25.6–46.7%, apparent porosity of 43.5–75%, bulk density of 1.45–1.9 g/cm³, and compressive strength of 0.7–9.7 MPa, respectively. On the other hand, the results of this study conducted using an alkali silicate solution (mixture of Na₂SiO₃ and NaOH) only, without the addition of any agricultural waste as a pore-forming agent, showed water absorption of 9.5–33.3%, apparent porosity of 18.2–56.7%, bulk density of 1.7–1.91 g/cm³, and compressive strength of 20.7–26.3 MPa, respectively. Comparing these results, we deduced that glass-ceramic foams of higher compressive strengths were obtained without including any carbonaceous material as a pore-forming agent.

The compressive strengths of all three samples obtained in this study are significantly higher than the 0.8–3.5MPa recommended range for a typical foam glass (Khamidulina et al., 2017) but compare favourably with over 20 MPa of porous concrete obtained by Alemu et al (2021). Nevertheless, sample 3, produced by adding 25 vol% of the alkaline-silicate solution, is considered the optimum sample for this study, given that its

compressive strength is close to 28 MPa, specified as the minimum standard value for concrete (Alemu et al., 2021). Hence, this implies that the glass-ceramic foam obtained in this study can be used in building applications requiring a load-bearing material.

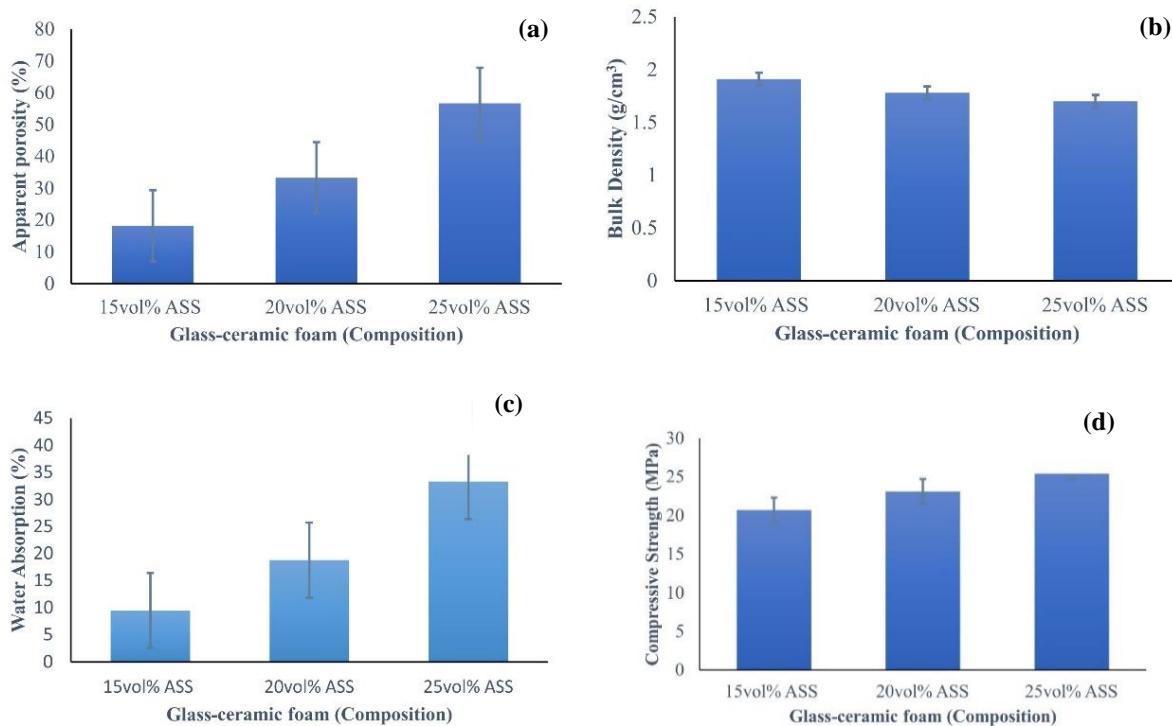


Figure 2. Physical and mechanical properties of the obtained glass-ceramic foam samples;
a) Apparent porosity; b) Bulk density; c) Water absorption; d) Compressive strength

3.3. Microstructural Properties

The glass ceramic foams' physicomechanical properties are substantially influenced by pore morphology (Zhang et al., 2022). In this regard, Figures 3a-c show the microstructural properties of the samples sintered at 850°C. The micrographs reveal varying morphological features of different degrees of agglomeration and porosity. The lighter areas illustrate the densification and aggregation of the samples' grains, showing the solid phase characteristic of glassy materials. The samples were observed to have a non-uniform pore size distribution and an open-celled morphology. The degree of open porosity and the morphology and dimensions of pores generated in glass-ceramic foams are influenced by the quantity of foaming agents introduced. This is supported by a study by Osfouri and Simon (2022), where varying amounts of SiC used as the foaming agent were added to a glass to produce foams with various pore characteristics. The results showed that increasing the volume of foaming agents resulted in higher porosity and larger pore sizes in the resulting foam. This finding is consistent with the research on ceramic foams conducted by Zakaria et al. (2020). Therefore, the amount of foaming agents utilized plays a substantial role in influencing the open porosity and pore characteristics of glass-ceramic foams.

According to Sazegaran and Nezhad (2021), pore morphologies considerably affect the mechanical properties of porous materials. The influence of pore morphology on the compressive strength of foamed materials revealed that their pore shapes had a higher impact on their mechanical properties than the pore size (Parveez et al., 2022). Therefore, the interconnected network of pores in the open-celled morphology and its ability to absorb energy under compressive loading could be responsible for the high compressive strength of the developed samples in this study.

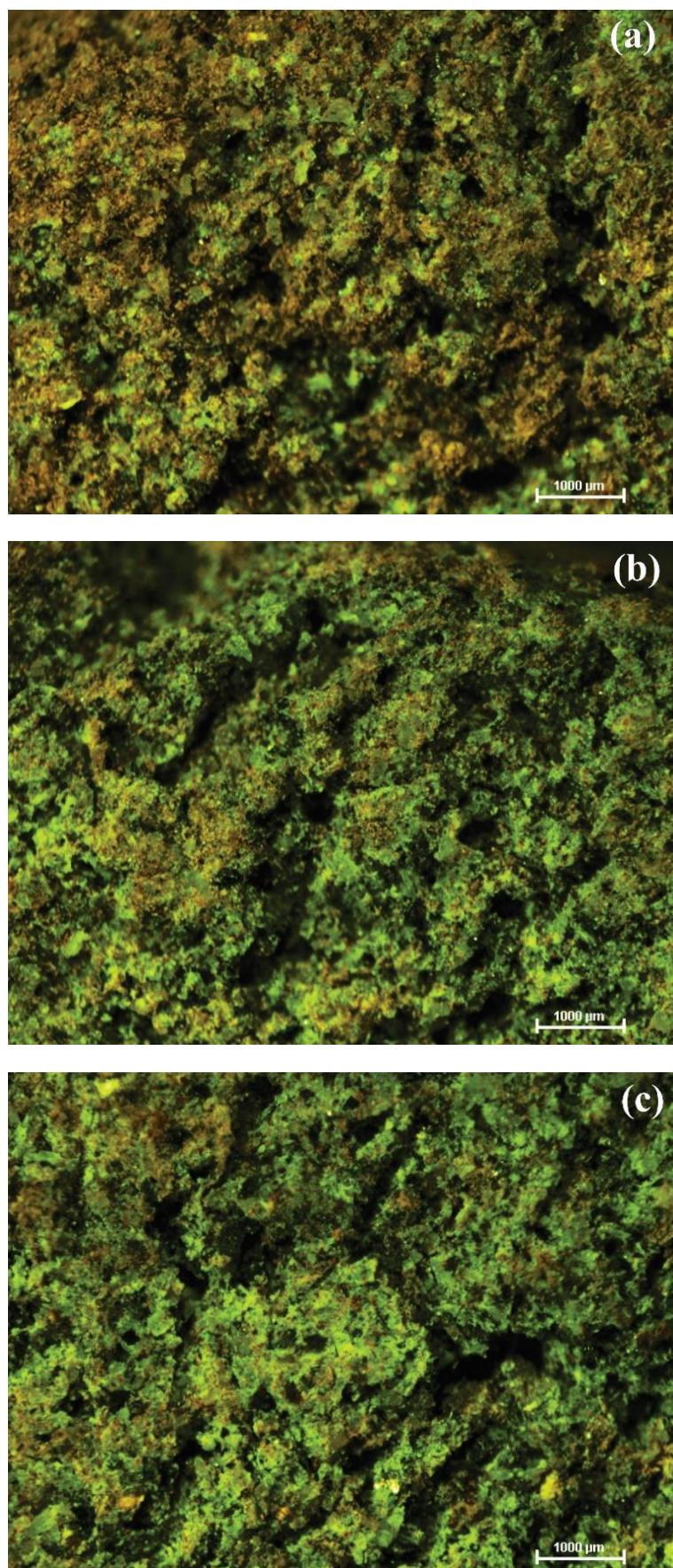


Figure 3. Micrographs of the prepared glass-ceramic foam samples sintered at 850°C with varying quantities of alkali-silicate solution; **a)** 15vol%; **b)** 20vol%; **c)** 25vol% (each at 1000 μm)

4. CONCLUSION

The viability of synthesizing glass-ceramic foams from a granite powder-clay mixture by one-step sintering has been explored in this study. The variation in the performance properties of the developed glass-ceramic foam samples was observed to have been due to the varying amounts of alkali-silicate solution used as the foaming agent. It was observed that with the rise in the foaming agent addition, water absorption increased linearly with apparent porosity values in the obtained glass-ceramic foam samples. In contrast, bulk density decreased linearly with a contrastive rise in the samples' compressive strength, which was traceable to the growing tendencies of the samples to vitrify at 850°C. The result obtained in this study and other pertinent studies showed that chemical reagents could serve as a sintering aid, foaming agent, or both, as the case may be when fabricating glass-ceramic foam from mining wastes, and provides an alternative cost-effective production route. According to Yu (2022), glass-ceramic foams have found practical applications in traditional buildings' thermal insulation. Therefore, the obtained glass-ceramic foam samples in this research can be suitable as a sustainable material in the thermal insulation of buildings. The results of the physicomechanical tests conducted on the glass-ceramic foams gotten in this study showed water absorption of 9.5-33.3%, apparent porosity of 18.2-56.7%, bulk density of 1.7-1.91 g/cm³, and compressive strength of 20.7-26.3MPa. Sample 3, produced by adding 25 vol% of the alkaline-silicate solution, is considered the optimum sample for this study, given that its compressive strength is close to 28 MPa, specified as the minimum standard value for concrete, and hence, can be used in building applications requiring a load-bearing material.

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CONFLICT OF INTEREST

The author declares no conflict of interest.

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