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Ceramics International 39 (2013) 2423–2430

CERAMICS INTERNATIONAL

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Glass foam of macroporosity using glass waste and sodium hydroxide as the foaming agent

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Received 2 July 2012; received in revised form 30 August 2012; accepted 1 September 2012
Available online 10 September 2012

Abstract

Glass foams using float glass waste and sodium hydroxide were produced. The influence of the sodium hydroxide amount in the foam formulation was studied. Titanium dioxide was used as a strengthening agent. The variations of temperature, heating rate and sintering time were investigated during the synthesis process. Open porosity was estimated using mercury porosimetry. The morphology of the glass foams was evaluated using scanning electron microscopy, phase formation was studied using X-ray diffraction, and chemical composition was estimated using X-ray fluorescence. As a result, glass foams with macroporosity were obtained. Since the glass foams used glass waste as reactant, the results suggest the development of an alternative route for glass recycling.

Keywords: B. Porosity; D. Glass; Waste treatment; Sodium hydroxide

1. Introduction

Nowadays, the progressive increase in the consumption of manufactured items and environmental concerns, due to either the reduction of natural resources or the increasing in solid wastes, has made the recycling of materials a fundamental aspect to be considered. For example, according to The European Container Glass Federation (FEVE), about 20.8 million tons of glass was produced as solid waste in 2010 in Europe; about 67% of this glass is recycled. On the other hand, as stated by the United States Environmental Protection Agency (US EPA), in the United States, 11.5 million tons of glass in the municipal solid waste stream was generated in 2010, and only about 27% of the glass was recovered for recycling.

A great amount of this solid glass waste is in the form of crushed solids in a large range of particle size, and different colors. Moreover, the glass can be contaminated with metallic and non-metallic fragments such as iron, aluminum, paper, ceramics, polymers, and other organic substances. The costs involved in the removal of impurities and color classification are relatively high, particularly if shares of the glass have a very fine particle size; therefore, in many situations, the production of items using recycled glass is a low profit activity [1]. Consequently, the search for alternative ways for recycling glass is still arousing interest. Furthermore, the recycling has been considered an important environmental issue due to a decrease in global natural resources and the constant increasing in the generation of solid wastes [2].

One of the possibilities of glass recycling is the production of porous ceramics [2–4]. The porous ceramics are associated with some characteristic properties such as, high surface area, high permeability and high stability at elevated temperatures, low density and low thermal conductivity [5]. Consequently, porous ceramics can be used to manufacture several items in diverse fields, including filters for molten metals and hot gases, low-density structural components, electrodes, sensors, reactors, catalyst carriers,

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http://dx.doi.org/10.1016/j.ceramint.2012.09.002

radiant burners, and porous implants in the area of biomaterials [5]. Because of their particular properties the porous ceramics are also used as insulator materials in roofs and walls [6].

In general, porous ceramic can be divided into two principal types: reticulated and foams. Reticulated porous ceramics consist of interconnected voids surrounded by a standing ceramic net [7,8] while foam is a gas dispersed in the form of bubbles in a ceramic suspension [9]. The ceramic foam is a tridimensional matrix of polyhedral cells with linear average dimensions of 10 μm–7 mm [9,10]; it is constituted of a foaming agent and, occasionally, other minerals that release gas inside of a pyroplastic mass when heated [1]. This pyroplastic mass is obtained through a viscous flow sintering mechanism at a temperature higher than the glass transition temperature (Tg) [11,12]. Some oxides and compounds are employed to improve the mechanical properties of glass; for example, Al_2O_3 platelets were used as a strengthening agent in a glass matrix [13]. Hollow glass microspheres are also employed as strengthening agents [14].

There are several materials that are used as foaming agents; as examples, the following materials can be cited: glycerin [15], $CaCO_3$ or Na_2CO_3, pure carbon, SiC, Si_3N_4, sugar, starch or organic wastes [9,16]. On the other hand, reports of the use of sodium hydroxide as a foaming agent were not found in the specialized literature.

Several foams have been produced using glass as a precursor [6,17]. These foams have many applications; for example, they can be used as thermal insulators, and as protectives of structural components against fire, moisture and erosion [18].

Several process variables must be considered during the synthesis of glass foams, and these variables can alter the product formation. These variables include the heating rate, the final temperature of sintering, the amount of foaming agent in the foam composition, and the sintering time [12,19].

The processing route also leads to foam products with different characteristics. Silica foams prepared using two different routes (surfactant-stabilized foams and particle-stabilized foams) showed differences in their structures and, as a consequence, the foam final properties were also unlike. For example, the particle-stabilized foams presented a compression strength and Young's modulus of almost one third of that presented in the surfactant-stabilized foams; on the other hand, particle-stabilized foams showed a higher Weibull modulus [20].

The average pore size analysis is described for a system constituted by powders of waste cathode ray tube glasses mixed with reducer agents (SiC or TiN) varying the sintering time at constant temperatures between 750 and 950 °C; an increase in the temperature leads to an increase in the average pore diameter [19].

The variation of sintering temperature in the SiO_2–Al_2O_3–B_2O_3 system mixed with different amounts of saccharose allowed to identify the degree of dependency of expansion volume of glass foams, which suffered an expansion of about eight times [21].

The soda-lime and $CaO–Al_2O_3–2SiO_2$ glass system mixed with B_2O_3, TiO_2 and ZrO_2 showed a remarkable variation in the average pore volume in response to different heating rates from 5 to 15 °C/min; the higher the heating rate, the greater the amount of released gas and there is a superior activation energy of the crystallization process [12,22].

Based on the environmental issues and on the properties of glass foams, the aim of this work is to investigate the microstructural properties of glass foams synthesized using float glass waste, sodium hydroxide (foaming agent) and titanium dioxide (strengthening agent) at several heating rates and sintering times. NaOH was chosen because reports of its use as a foaming agent, as already mentioned, were not found in literature.

2. Materials and methods

The ceramic foams were prepared using float glass waste milled in a conventional ball mill for 8 h. The milled glass was mixed with titanium dioxide (99%, Synth) and sodium hydroxide (99%, CRQ). The compositions consisted of 20% (wt%) of titanium dioxide varying the amount of sodium hydroxide as 3%, 5%, 7%, 9%, 11%, 13%, 15% and 17% (wt%). The powders were mechanically mixed with the addition of sodium hydroxide dissolved in about 1 g/ml of water. The compositions were dried in a laboratory kiln at 60 °C for 24 h.

After the drying process, samples were sintered in a muffle furnace at 800 °C. The sintering times were 15, 30, 45, 60, 90, and 120 min. The heating rate was also varied as 5, 10, 15, 20, 25 and 30 °C/min.

Open porosity of the bulk specimens was investigated using mercury porosimetry technique (Micromeritics Autopore IV 9500); the pressure range varied from 0.007 MPa to 207 MPa. Geometric densities of samples were also measured. The microstructure of samples was characterized using scanning electron microscopy (Jeol JSM N-7500F-FEG).

After sintering, samples were milled in an agate mortar and sieved in a 400 mesh sieve. Afterwards, samples were

Table 1
Density and open porosity measured by mercury porosimetry and geometric density for samples with different amounts of NaOH.

NaOH content (wt%)	**Density at 0.0031 MPa (g/cm^3)**	**Open porosity (%)**	**Geometric density (g/cm^3)**
3	2.20 ± 0.02	1.8 ± 0.02	3.50 ± 0.77
5	0.83 ± 0.01	70.7 ± 0.71	0.83 ± 0.12
7	0.67 ± 0.01	53.1 ± 0.53	0.64 ± 0.07
9	0.59 ± 0.01	87.8 ± 0.88	0.69 ± 0.08
11	0.53 ± 0.01	80.7 ± 0.81	0.60 ± 0.06
13	0.56 ± 0.01	86.7 ± 0.87	0.39 ± 0.04
15	0.56 ± 0.01	83.1 ± 0.83	0.31 ± 0.04
17	0.86 ± 0.01	44.2 ± 0.44	0.33 ± 0.04

characterized using X-ray diffraction (Shimadzu XRD6000) using Cu Kα radiation at 40 kV and 30 mA with the patterns recorded in the 15°–90° 2θ measuring range using a step of 0.02° with a measuring time of 0.6 s. Milled samples were also stabilized in distilled water (175:1 water-to-powder ratio, in mass) for 24 h to perform pH measurements using a pH meter NT PHM. Likewise, a bulk sample containing 17% of NaOH was stabilized in distilled water and the pH was measured.

Compression tests (Shimadzu Autograph AG-I 10 kN universal testing machine) were conducted in samples with and without TiO_2; samples with 11% of NaOH, and calcined at a heating rate of 15 °C/min were used in these tests.

3. Results and discussion

The open porosity and density of glass foams calcined at 800 °C for 60 min at a heating rate of 15 °C/min were measured using mercury porosimetry; these results are shown in Table 1, which also presents the geometric density for each sample. The calcination temperature of 800 °C is used

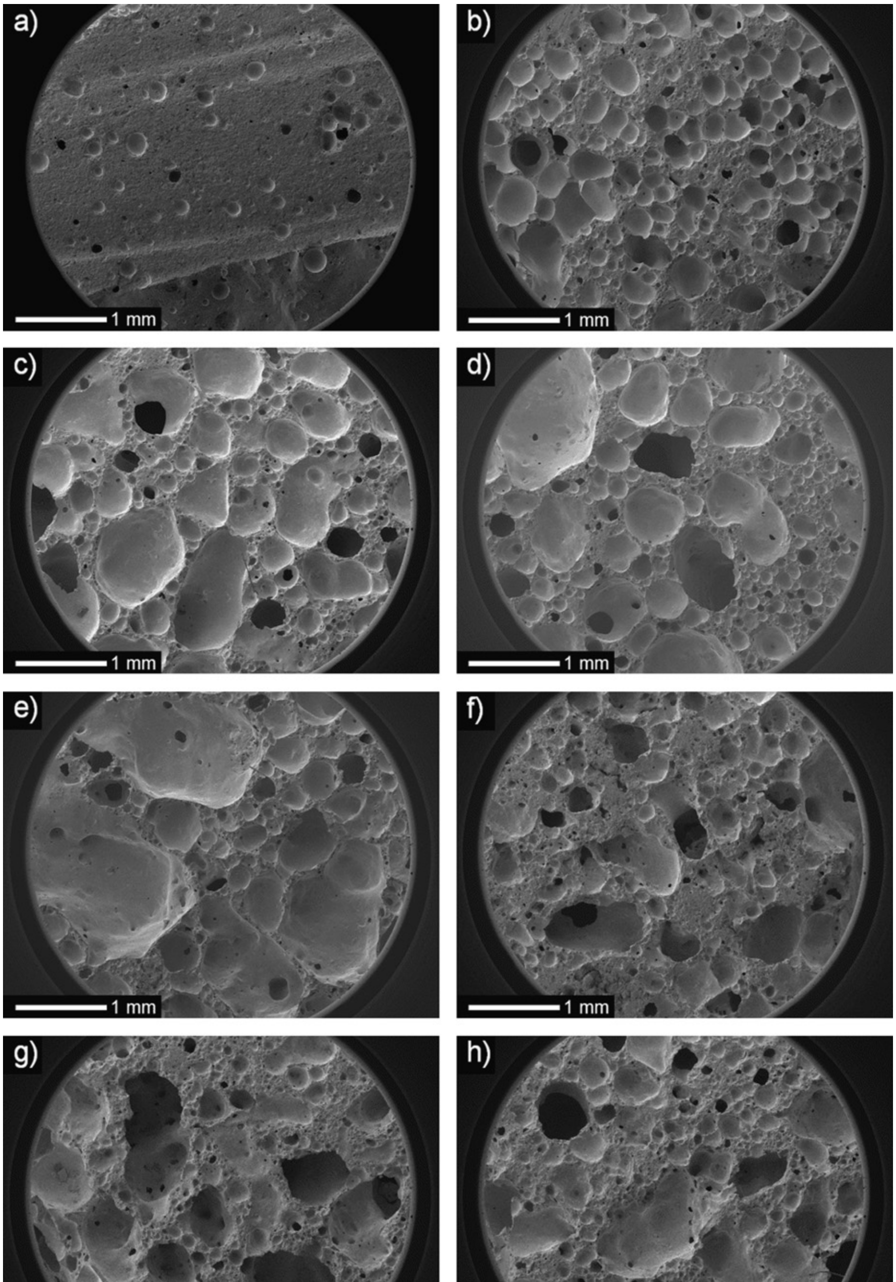

Fig. 1. Micrographs of glass foams sintered at 800 °C for 60 min at a heating rate of 15 °C/min with several NaOH amounts: (a) 3%, (b) 5%, (c) 7%, (d) 9%, (e) 11%, (f) 13%, (g) 15% and (h) 17%.

Table 2
Chemical analysis using X-ray fluorescence for reactants (NaOH, float glass, and TiO_2) and a glass foam with 17% of NaOH sintered at 800 °C for 60 min using a heating rate of 15 °C/min.

	NaOH	Na_2CO_3	TiO_2	SiO_2	CaO	Na_2O	MgO	Al_2O_3	Other
NaOH	99% min	1% max							
Float glass				57%	25.1%	11.1%	3.2%	0.8%	2.8%
TiO_2			99.4% min						0.6% max
Glass foam			53.4%	28.0%	12.5%		1.7%	2.0%	2.4%

because at this temperature viscous flow sintering occurs as a result of the generation of a large amount of liquid phase with lower high-temperature viscosity [23]. Moreover, the softening temperatures of compounds used in the experiment are close to 800 °C, and this calcination temperature was used in several reports on glass foams [24–27].

According to the results of Table 1 it can be seen that the geometric density of samples decreases with the increasing of the NaOH content; this fact is due to the foaming process of samples. Moreover, it can be suggested that the amount of total porosity is proportional to the increase of NaOH content. The differences between the density values measured by porosimetry and the geometric method can be attributed to the fact that mercury porosimetry measures only accessible porosity (open porosity). The sample containing 3% of NaOH shows a small amount of pores with predominance of closed pores, explaining the low value of sample porosity. At higher NaOH contents the presence of open pores is significant which can be verified by analyzing the open porosity values. Moreover, the NaOH content influences the amount of open porosity of the foam; the maximum open porosity was found in the range of NaOH content from 9% to 15%. The interpretation of the results of Table 1 becomes more evident by analyzing the samples micrographs shown in Fig. 1.

In comparison to the other NaOH amounts, the micrograph of Fig. 1(a) shows a small fraction of pores which is dispersed in a dense matrix of sintered oxides. As the NaOH content increases to 5% and 7%, Fig. 1(b) and (c), respectively, closed pores still predominate in the microstructure. The presence of these closed pores in samples containing 5% and 7% of NaOH explains the fact that the latter showed smaller open porosity.

The use of NaOH as a foaming agent becomes effective only for NaOH contents higher than 7%. For example, on increasing the NaOH content to 9% (Fig. 1(d)) the increase in pore size continues to be verified, but open pores are already visible. For NaOH amounts higher than 9%, formation of macroporosity can be observed in the system according to the International Union of Pure and Applied Chemistry (IUPAC) [28]. The increase of pore size is due to the effect of grain coalescence [29] and in samples with 11% of NaOH, shown in Fig. 1(e), the pore size continues to increase and presents a remarkably misshapen morphology; also, the presence of holes inside the pores can be noticed. For samples with 13% of NaOH, shown in Fig. 1(f), it can be observed that the attack to the silicate phase is intensified due to the increase in alkali content, which is predominant in the viscous flow sintering process of the system. Consequently, the pore size diminishes and there is an increase in the number of interconnected pores, as can be seen in Fig. 1(g) and (h), and it is possible to observe an incipient coalescence among the cells. Coalescence leads to a decrease in the thickness of the cell walls as a result of coarsening of gas bubbles in the softened glass [25]. Samples with 17% of NaOH showed a smaller amount of open pores (Table 1) and this fact is also confirmed by analyzing Fig. 1(h). Based on these facts, the use of NaOH as a foaming agent leads to adequate porous materials when used in the range of 9–15%.

Table 2 shows chemical analysis using X-ray fluorescence for the reactants (sodium hydroxide, glass waste, and titanium oxide), and for a sample of glass foam with 17% of NaOH sintered at 800 °C for 60 min using a heating rate of 15 °C/min. The amount of elements in the sintered products indicates the transformation process of the materials, and chemical analysis was used to identify the reflections in the X-ray pattern of this sintered sample (Fig. 2).

The X-ray pattern of the sintered sample (17% of NaOH sintered at 800 °C for 60 min using a heating rate of 15 °C/min) is shown in Fig. 2. The present phases identified according to the reflections of the X-ray pattern are summarized in Table 3. Analyzing Fig. 2 and Table 3 it can be noticed that no NaOH reflection, which is a precursor of the foam, was found after the sintering process. Moreover, the results suggest that the NaOH reacted with the other reactants, specially silicon, calcium and aluminum oxides, during the foam sintering process. It can be highlighted that the sodium hydroxide was an efficient foaming agent eliminating the need of adding carbon compounds in the foam composition to obtain good results. Moreover, according to Fig. 2, the presence of sodium oxide in crystalline phases is very limited suggesting that most of the oxide is in the residual glass phase.

To verify the influence of NaOH on structure of glass foam and verify its stability in acidic fluids and maybe even with moisture, the glass foam was milled and stabilized in distilled water; pH measurements of these suspensions are shown in Fig. 3. There is an increase in the pH of the suspensions as the NaOH content of samples increases; in this case, there is a significant variation in the pH value.

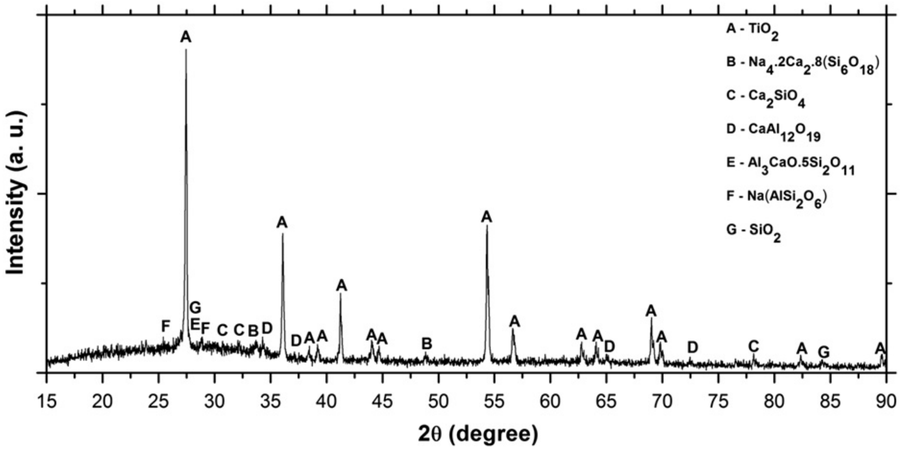

Fig. 2. X-ray pattern of a glass foam with 17% of NaOH, sintered at 800 °C for 60 min using a heating rate of 15 °C/min.

However, when a bulk sample is stabilized in distilled water the pH change (also shown in Fig. 3) is less significant. These results suggest that, besides the large amount of NaOH in green samples, the foams produced can be used in the filtration of certain types of fluids. For comparison, Fig. 3 also shows the pH of distilled water with no foam.

The influence of the heating rate and the sintering time at 800 °C in the foaming process were also evaluated. The heating rate needs to be accurately controlled to generate glass foams because the effect of the rate of heating on the bubble–melt interface area can be caused by the impact of rate of heating on bubble nucleation and on the mode of foaming [12]. This variable was studied using samples with 11% of NaOH, sintered at 800 °C for 60 min using several heating rates: 5, 10, 20, 25, and 30 °C/min. The micrographs of Fig. 4 present the behavior of these samples after sintering.

It can be noticed in Fig. 4 that low heating rates are not adequate for pore formation because the process becomes too slow. At high values of rate of heating, the melt becomes quickly oversaturated with gas, forcing the pre-existing bubbles to grow rapidly and become large [12]. Heating rates until 20 °C/min made the elimination of steam and gases more difficult, originating large macropores due to the sintering process with viscous flow, as can be seen in Fig. 4(a)–(d). On the other hand, heating rates of 25 and 30 °C/min favored the elimination of steam and gases during the viscous flow process and this effect can be observed in Fig. 4(e) and (f).

The open porosity as a function of the heating rate was also analyzed using mercury porosimetry. Samples shown in Fig. 4(a) and (b) presented open porosity of 35%. In contrast, samples of Fig. 4(c) showed open porosity of about 80%. Finally, the relative porosity of samples prepared using heating rates of 20, 25 and 30 °C/min are 35%, 50%, and 45%, respectively. Since all these samples suffered the same calcination time, slower heating rates allow a longer exposition of samples in temperatures that the foaming process may occur in. On the other hand, higher heating rates may limit the foaming upon heating and the foaming process occurs during the holding time at the calcination temperature. These facts were first reported for glass foams from dismantled cathode ray tubes [3]. The heating rate of 15 °C/min favored the foaming process at the calcination time employed, without favoring excessive pore coalescence. When higher heating rates are employed the foaming upon heating was limited and the presence of open porosity is reduced.

Table 3
X-ray reflections shown in Fig. 2 identified according to the Powder Diffraction File (PDF).

Reflection from Fig. 2	Phase	PDF card
A	TiO_2	21-1276
B	$Na_4 \cdot 2Ca_2 \cdot 8(Si_6O_{18})$	78-1649
C	Ca_2SiO_4	87-1261
D	$Ca_2Al_{12}O_{19}$	33-0253
E	$Al_3CaO \cdot 5Si_2O_{11}$	46-0744
F	$CaAl_2(SiO_4)$	03-0559
G	$Na(AlSi_2O_6)$	80-1561
H	SiO_2	83-2473

The influence of sintering time was also evaluated, and these results are shown in Fig. 5 for sintering times of 15, 30, 45, 60, 90 and 120 min.

According to Fig. 5, samples showed similar behavior when sintering times of 15–60 min are used (Fig. 5(a)–(d)); however, pore size tends to increase when sintering times of 90 min (Fig. 4(e)) and 120 min (Fig. 4(f)) are employed. This occurs because there is a closing of interstitial holes promoted by an increasing in the surface tension of the liquid–gas interface when the system is heated at higher temperatures than the glass transition of the milled glass present in samples (Tg ≈ 550 °C). As expected, prolonged calcination times favored pore coalescence.

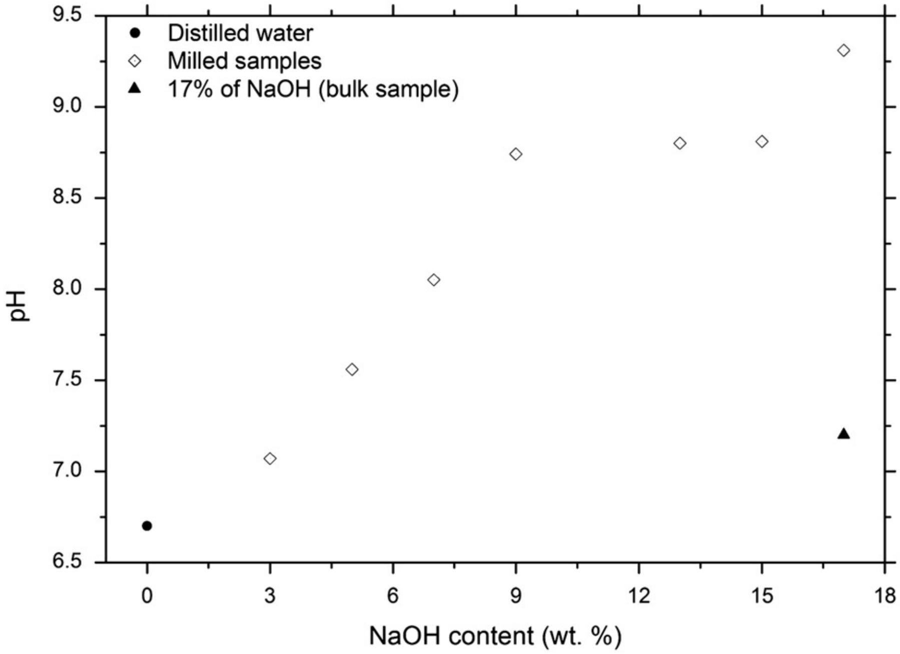

Fig. 3. pH of milled samples in distilled water as a function of the NaOH content. The figure also shows the pH of distilled water without milled foams and of a bulk sample containing 17% of NaOH (15 °C/min) in distilled water.

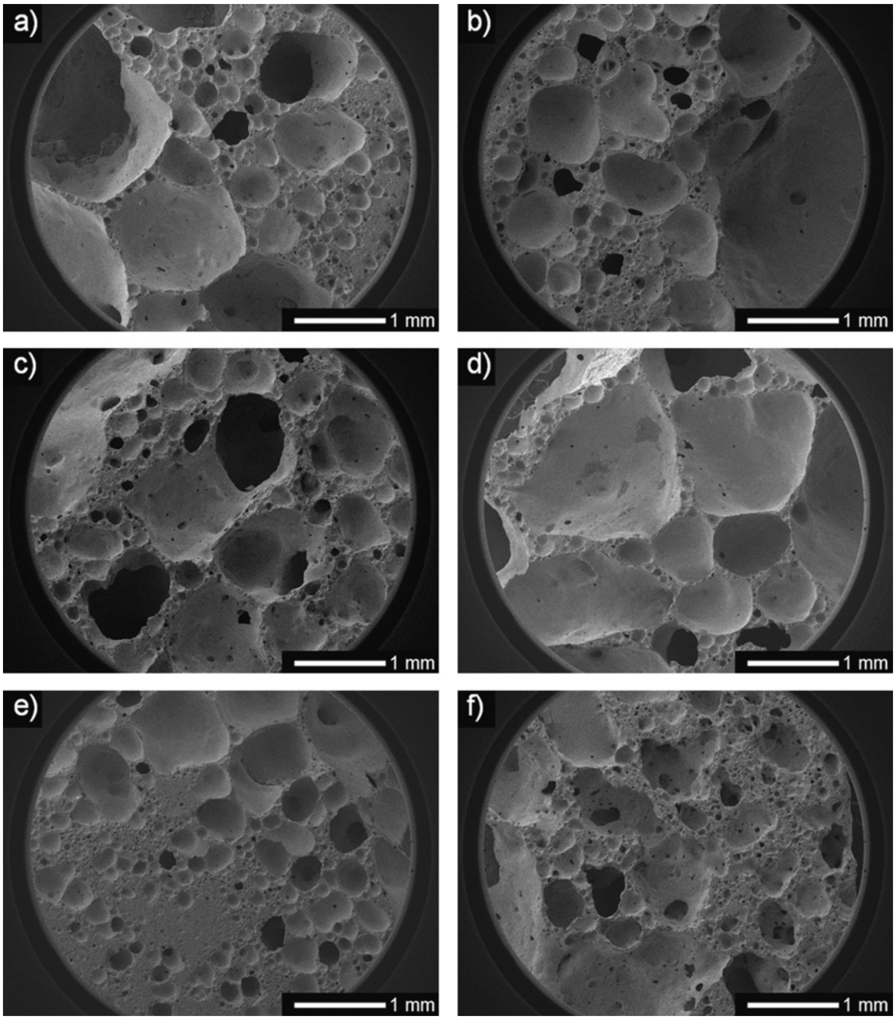

Fig. 4. Micrographs of glass foams with 11% of NaOH sintered at 800 °C for 60 min at several heating rates: (a) 5 °C/min, (b) 10 °C/min, (c) 15 °C/min, (d) 20 °C/min, (e) 25 °C/min and (f) 30 °C/min.

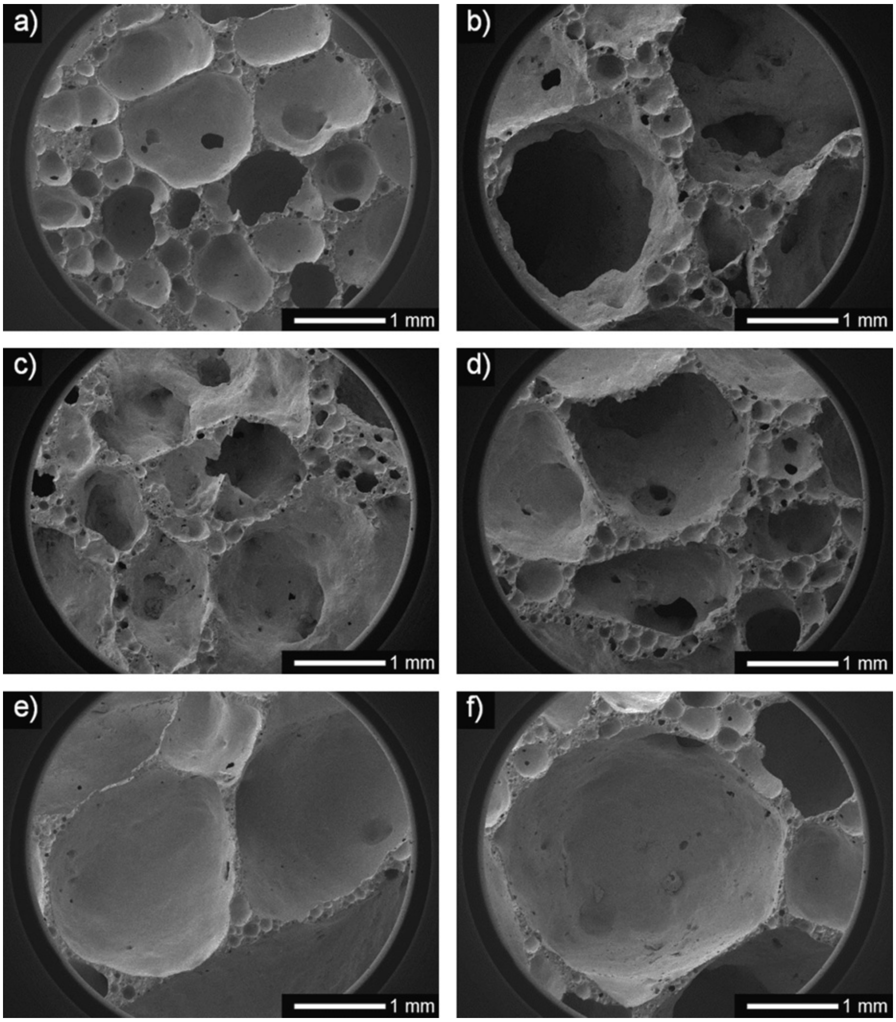

Fig. 5. Micrographs of glass foams sintered at 800 °C with 11% of NaOH at several sintering times, using a heating rate of 15 °C/min. (a) 15 min, (b) 30 min, (c) 45 min, (d) 60 min, (e) 90 min and (f) 120 min.

In general, for all studied samples in this work, the presence of macropores, which means pores greater than 50 nm, were predominant [30].

The use of TiO_2 is justified because this oxide works as a strengthening agent to the glass foams produced in this work. This action of TiO_2 was verified by performing compression tests in samples containing 11% of NaOH with and without TiO_2. The maximum compressive stress values were 3.40 MPa and 0.48 MPa for samples with and without TiO_2, respectively. In other words, the maximum compressive stress of samples containing TiO_2 is about seven times greater than the stress of samples containing no TiO_2.

The experiments performed here used waste of float glass as raw material; however, the application of the methodology and the use of sodium hydroxide as a foaming agent can be tested with other kinds of glass waste, which would open an alternative route for glass recycling. Moreover, this work focused on the development of a methodology to produce glass foams and on the characterization of their microstructure, and further research should be conducted to characterize other foam properties, such as mechanical strength and thermal conductivity.

4. Conclusions

Sodium hydroxide was satisfactorily employed as a foaming agent to produce ceramic foams based on float glass waste and titanium dioxide (strengthening agent). Elevated open porosity, in the order of 80%, was found in the range of 9–15% of NaOH (wt%).

The different sintering parameters of glass foam studied (the amount of sodium hydroxide, heating rate, and sintering time) allowed the obtaining of macroporous glass foams predominantly. These sintering parameters interfere in the porosity and interconnection of pores in the foams.

The synthesis process is controlled by viscous flow during the sintering of the system because the sintering process occurs at higher temperatures than the glass transition temperature of the float glass.

Acknowledgments

The authors acknowledge the Brazilian research funding agencies Capes, CNPq (Grant 558595/2008-1), and Finep (Grant 3903/06) for their financial support.

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