Manufacturing of wollastonite-based glass from cement dust: Physical and mechanical properties

A.A. Francis and M.K. Abdel Rahman

Cogent Engineering (2016), 3: 1170750
Manufacturing of wollastonite-based glass from cement dust: Physical and mechanical properties

A.A. Francis1* and M.K. Abdel Rahman1

Abstract: By-pass cement dust is considered as a source of environmental pollution. Wollastonite-based glass foams are made by adding glass waste and SiC to the cement dust. XRD on samples indicated that the main crystalline phase after heat treatment at 850–1,000°C is wollastonite. Empirical models were developed to derive conclusion on the impact of SiC and temperature on the physical and mechanical properties of the products. The optimum sintering temperature was found to be at 900°C for 60 min, at which crushing strength was about 15 MPa and was the best uniform. Such wollastonite-based glass foam could be very attractive for thermal and acoustic applications.

Subjects: Engineering & Technology; Environment & Agriculture; Physical Sciences

Keywords: by-pass cement dust; crushing strength; microstructure; wollastonite-based glass; industrial wastes

ABOUT THE AUTHOR
A.A. Francis, Phd, is currently based at the Central Metallurgical Research and Development Institute. His research is broad and encompasses a number of the various areas that fall under the heading of Materials Science and engineering. His research interests are centred on the relationships between the processing, microstructure and functional properties of brittle materials, such as ceramics and glasses. This includes work on electrical, mechanical and magnetic properties of ceramics, development of new or improved products of glass and/or glass-ceramics from industrial waste materials. Another important research area is the mechanical and magnetic behaviour of porous ceramics, such as foams prepared from industrial wastes. Other research interests include the re-employment of the waste (in particular from metallurgical and chemical industries) as raw material to produce inert and usable material which can find wide range of applications in several fields.

PUBLIC INTEREST STATEMENT
The re-use of materials gained from industrial solid wastes contributes to the conservation of resources and provides economically favourable solutions as well as creates jobs. In this context, developing calcium silicate (wollastonite)-based foam materials from the combination of by-pass cement dust (BCD) and waste glass demonstrates the advantage of combining various remarkable properties in one material. BCD is a by-product from the manufacture of Portland cement. Wollastonite-based foam is not only useful in a range of applications including catalyst supports, but also it contributes considerably in the environmental protection while being used as substitute for asbestos in thermal insulation application. Empirical models were developed to derive conclusions on the impact of foaming agents and temperatures on the physical and mechanical properties of the produced wollastonite-based glass foams. The powder technology and sintering route was the effective approach to safely use the above-mentioned wastes and produce application-oriented products.
1. Introduction
In developing countries the majority of wastes end up in landfills, in uncontrolled sites or in other inappropriate places, as landfilling is still the cheapest and most common method for the disposal of industrial and municipal wastes. If most of the wastes could be diverted for material and resource recovery, then a lower environmental impact could be achieved and the recovered material could be utilized to fund an economically and environmentally solid waste management. By-pass cement dust (BCD) is a by-product of the manufacture of Portland cement. It is a fine-grained material that is collected from exhaust gases by electrostatic precipitators during the calcination process. Every year, the cement sector in Egypt generates 2.4 million metric tons of by-pass dust. Discarded window glass constitutes also a significant component of the municipal and industrial solid waste stream in every country. Although several solutions have been suggested for the recycling and safe disposal of such wastes, the production of glass or glass-ceramic foams by re-use of industrial wastes appears to be a promising development for a variety of engineering applications such as adsorbents, light weight structural components, filters and in thermal protection systems, etc. (Chinnam, Francis, Will, Bernardo, & Boccaccini, 2013; Francis & Abdel Rahman, 2013; Francis, Abdel Rahman, & Daoud, 2013).

The development of cellular cement, consisting of Portland cement paste or mortar with a homogeneous void or cell structure created by introducing air or gas during the mixing process, has been the subject of many investigations (Laukaitis & Fiks, 2006; Tonyan & Gibson, 1992). Cement kiln dust (CKD) has been mixed with Portland cement to be used as filler in asphalt concrete mixtures (Krazowski & Emery, 1981); or with fly ash in road construction (Miller & Zaman, 2000). A similar approach was applied by Pavia and Regan (2010), who produced masonry mortar by combining mixtures of CKD with a non- hydraulic binder of high available-lime content. Various potential applications of CKD have been considered for the construction industry, e.g. in formulations for masonry and concrete blocks (Ravindrarajah, 1982); as well as in stabilizing highly expansive clay soils (Zaman, Laguros, & Sayah, 1992). Carlson, Sariosseiri, and Muhunthan (2011) demonstrated that lower and higher percentages of CKD can be used for soil modification/stabilization purposes in geotechnical construction. They found that the addition of a few percentages of CKD (5-20%) to the soils in the state of Washington showed significant improvement in drying rate and unconfined compressive strength of the CKD treated specimens as the percentage of CKD increased. On the other hand, it is also important to exploit CKD as a source material of CaO for carbon capture and mitigate the possible adverse health and environmental affects posed by improper CKD disposal (Huijgen & Comans, 2003, 2005). A recent study (Sanna, Dri, Hall, & Maroto-Valer, 2012) highlighted the advantages and disadvantages of using mineral wastes for carbon capture and storage. In this study, the physical and mechanical measurements of the final product indicate the potential of the sintering route to fabricate wollastonite-based foam materials from the combination of BCD and waste glass. However, to the best of the authors’ knowledge, the crushing strength of wollastonite-based glass foams has not been so much considered.

2. Experimental procedure
The BCD brought from National Cement Co., Egypt, has the following composition (weight %) CaO 44.24%, P₂O₅ 6.35%, Fe₂O₃ 1.92%, Cl 4.93%, MgO 1.45%, K₂O 3.5%, SO₃ 2.9%, Al₂O₃ 4.7%, SiO₂ 10.54%, Na₂O 2%, loss of ignition 17.47%. BCD consists of significant amounts of silica, alumina and phosphorous oxide; and small amounts of sulphur, Cl and alkalis. The chemical composition of the investigated BCD was conducted by X-ray fluorescence spectroscopy (XRF) (PANalyticalXRF-advanced AXIOS). Crystalline phase identification was conducted by X-ray diffraction (Bruker D8 X-ray diffractometer). The microstructure of polished samples was studied by means of scanning electron microscopy (SEM) coupled with energy dispersive X-ray analysis (Jeol–Japan). As a result of preliminary experiments, the mixture containing 30%
BCD and 70 wt.% soda-lime glass was selected for this study. This mixture and various amounts of SiC as foaming agent within the range 1-10 wt.% were mixed and pressed uniaxially, with the addition of a small amount of a sugar solution as binder at a pressure of 50 MPa, into cylindrical shapes. The powder compacts were sintered in an electric furnace in air. The heating rate was 5°C/min. Sintering temperatures were between 850 and 1,000°C and the sintering time was for 1 h. In this research, many repeated experiments were conducted to determine the empirical models generated by the sigma plot software. The data was fit with a 3D using SigmaPlot version 11, from Systat Software, Inc., San Jose California USA. Wollastonite-based foams with various SiC dosages were characterized in terms of density, water absorption capacity and crushing tests. In order to characterize the thermal behaviour of the sample, differential scanning calorimetry (DSC) was carried out in a Pt crucible up to 1,000°C with a 10°C/min heating ramp in air using a Setaram Setsys apparatus. The Archimedes method was employed to measure water absorption after the foams had been placed in boiling water for 3 h. The water absorption was calculated as follows: water absorption percentage = \((W_s - W_d) / W_d\) × 100, where \(W_d\) is the dry weight of the calcined foams, \(W_s\) is the 3 h saturated surface-dry weight. A universal testing machine (Shimadzu-UH-F1000 kNI) was used for testing the wollastonite samples. The cross-head displacement rate was 0.5 mm/minute in all tests. The output of the data acquisition system was used to make strength-displacement plots. The strength in MPa was calculated by dividing the applied load by the original specimen area to obtain the applied nominal stress.

3. Results and discussion
The batch sample containing 30 wt.% BCD was selected for study on the basis of basic processing characteristics such as ease of sintering and maintaining the shape of the cylindrical samples. The thermal behaviour of BCD-glass mixture with incorporation of 5% SiC up to 1,000°C is displayed in Figure 1. The DSC plot revealed a well-defined glass transition at 686°C. This is followed by a broad shoulder exothermic reaction over the temperature range 800–900°C which is ascribed mainly to the
crystallization of wollastonite as confirmed by XRD. It should be noted that Müller et al. (2009) stated that wollastonite is firstly developed between 850 and 900°C. This is consistent with the DSC result which displays the broad shoulder zone at 800–900°C. Variation of density and water absorption percentage along with SiC content and sintering temperature are shown in Figures 2 and 3. It was reasonable to observe a decrease in density over the foaming range of 1–10% SiC from 1.816 to 1.568 g/cm³ at 850°C. It was mentioned in previous investigations (Bernardo & Albertini, 2006; Bernardo, Cedro, Florean, & Hreglich, 2007), that the non-progressively decrease in density with increasing foaming agent content is likely due to the occurrence of coalescence phenomena. It can be seen that the density decreases dramatically with increasing sintering temperature up to 1,000°C, reaching minimum values of 0.805 and 0.562 g/cm³ at 5 and 10% SiC, respectively, Figure 2. The decrease in density at higher temperature occurs due to the expansion of pores with the release of CO₂ and the low viscosity of samples. At 1,000°C, a shape distortion effect occurred and the samples did not retain their original cylindrical dimension, and that's explained why the sintering at 900°C was chosen as the best one (the cover image displayed the sintering of the powder compacts at 1,000°C for 60 minutes in the presence of 5% SiC). Figure 3 showed that the water absorption capacity at 850 and 900°C did not lead to major changes and followed the same trend as the 3D plot of the density. On increasing the temperature to 1,000°C, the water absorption went up to more than 90%, and this behaviour was mainly ascribed to the unstability (distortion) of the cylindrical shape.

Figure 2. Dependence of the density of wollastonite-based foams on the percentage of SiC and temperature.

Figure 3. Dependence of the water absorption capacity of wollastonite-based foams on the percentage of SiC and temperature.
Experimental data were analysed by multiple models to test the significance and suitability of the model. The paraboloid model adequacy was confirmed by a reasonable value of the correlation coefficient (R), which was determined to be 0.909 and 0.8943 for the density and water absorption of the wollastonite-based glass foam. These values suggest that the chosen model could predict ~ 90% of the variability in both responses. A paraboloid equation was developed to correlate the density ($\rho$) and water absorption (WA) as a function of processing temperature and amount of SiC. The evaluation is undertaken for 5% confidence level of significance.

\[
\text{Density} = -13.1655 + 0.037x - 0.1343y - 2.2489e^{-0.05}x^2 + 0.0082y^2
\]  

\[
\text{Water absorption} = 2336.44 - 5.1983x + 0.0419y + 0.0029x^2 + 0.2496y^2
\]

where $y = \text{SiC\%}$ and $x = \text{temperature}$.

The XRD patterns from samples sintered at different temperatures in the range 850–1,000°C for a constant time of 60 min are presented in Figure 4. The poor crystallinity at 1,000°C and the fewer diffraction peaks confirmed our findings regarding the partial melting of the sample that leads to the decrease or disappearance of many peaks belonging to the wollastonite phase. The XRD analysis of samples sintered at 900°C for different periods of time revealed that the peak intensity of wollastonite decreased slightly with time, Figure 5, where a partial melting occurred and the resulting material has a lower crystallinity.
The effect of sintering temperature on samples containing fixed amount of added SiC (5wt %) and treated for 1 h, is analysed by SEM in Figures 6 and 7. The sample showed a dispersed phase of wollastonite and exhibited a very rough strut surface with non-uniform crystals, which appear in light grey as shown in Figure 6. The cell structure of the foam consists of opened and closed cell pores. EDX revealed that the composition of the crystal has a predominant silicon (Si) content followed in abundance by oxygen (O), calcium (Ca), sodium (Na), magnesium (Mg) and potassium (K), Table 1. By increasing the sintering temperature to 1,000°C, Figure 7, the cell structure of the foam consists of opened and closed pores with a majority of larger portion of opened pores. Elements such as calcium, oxygen and silicon were only detected in the wollastonite-based glass foam, Table 2. The

**Figure 6.** SEM images of wollastonite-based foams obtained at 900°C using 5wt.% SiC. A representative EDX spectrum showing the elemental composition of the product.

**Table 1.** Quantitative elemental composition of the crystals

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>33.24</td>
<td>49.7</td>
</tr>
<tr>
<td>Si</td>
<td>35.25</td>
<td>30.02</td>
</tr>
<tr>
<td>Ca</td>
<td>26.03</td>
<td>15.54</td>
</tr>
<tr>
<td>Na</td>
<td>2.43</td>
<td>2.53</td>
</tr>
<tr>
<td>Mg</td>
<td>1.1</td>
<td>1.09</td>
</tr>
<tr>
<td>K</td>
<td>0.63</td>
<td>0.26</td>
</tr>
<tr>
<td>Fe</td>
<td>0.68</td>
<td>0.29</td>
</tr>
</tbody>
</table>

**Table 2.** Quantitative elemental composition of the crystals

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>65.12</td>
<td>78.89</td>
</tr>
<tr>
<td>Si</td>
<td>20.52</td>
<td>14.16</td>
</tr>
<tr>
<td>Ca</td>
<td>14.36</td>
<td>6.95</td>
</tr>
</tbody>
</table>
effect of foaming agent percentage and sintering temperature on the crushing strength is depicted in Figure 8. By observing the dependency of the crushing strength on the process variables, it can be concluded that the sintering at 900°C is the most preferable. It is obvious from the plot that as the sintering temperature increases, the crushing strength decreases to 1.8 MPa and that is due to the expansion of pores resulted from the partial melting of the sample. The $R^2$ was found equal to 0.9011. A Gaussian equation was developed to correlate the crushing strength as a function of processing temperature and amount of SiC. It is worth mentioning that the control of temperature is significant for achieving uniform structure and realizing reasonable density and water absorption.

\[
\text{Crushing strength} = 19.5213 \times \exp\left(-0.5\times\left(\frac{(x-902.0518)}{48.7841}\right)^2 + \left(\frac{(y-6.8174)}{4.4412}\right)^2\right)
\]

(3)
The influence of temperature on crushing strength (strength–displacement behaviour) of wollastonite-based glass foams is illustrated in Figure 9. In the initial stage and especially at 850 and 900°C the material is linear elastic up to the peak stress, at which point the stress drops dramatically leading to failure of pore walls and thus crushing of the sample up to a large strain. By increasing the strain, serration is observed and the stair-like deformation of foam cells occurred layer by layer from either the top or bottom layers in the direction of the compression load (Gibson & Ashby, 1997). The crushing strengths were all higher than ≥1 MPa, and the densities were in the range of 0.5621–1.816 g/cm³.

Figure 9. Crushing strength–displacement curves of wollastonite-based foams prepared at 850, 900 and 1,000°C containing 10% SiC (a,b,c), respectively.
These results indicate that the increase in the sintering temperature leads to the decrease in both the density and the crushing strength of samples. Felipé-Sesé, Eliche-Quesada, and Corpas-Iglesias (2011) mentioned that the compressive strength of ceramic materials derived from different industrial residues covered the range of 29.8–59.3 MPa. Wollastonite-based foam is not only useful in a range of applications including catalyst supports, but also it contributes considerably in the environmental protection while being used as substitute for asbestos in thermal insulation application.

4. Conclusion

The use of industrial by-product materials as raw materials is an interesting and profitable alternative from both the economic and the environmental perspective. BCD is considered as a source of environmental pollution. Wollastonite-based glass foams are made by adding waste glass and SiC to the by-pass cement dust. The strength–displacement curve is typical of brittle foam. The deformation of foam cells occurred layer by layer from either the top or bottom layers in the direction of the compression load. Wollastonite-based glass foams derived from industrial wastes is an important substance in the ceramic and cement industries, which is able to compete with current materials for construction, insulation and other specialized applications.

Funding
The authors received no direct funding for this research.

Author details
A.A. Francis
E-mail: adel_francis@hotmail.com
ORCID ID: http://orcid.org/0000-0001-8542-2912
M.K. Abdel Rahman
E-mail: mkamal7@hotmail.com
1 Central Metallurgical Research and Development Institute- CMRDI, P.O. box 87 Helwan, Cairo, Egypt.

Citation information
Cite this article as: Manufacturing of wollastonite-based glass from cement dust: Physical and mechanical properties, A.A. Francis & M.K. Abdel Rahman, Cogent Engineering (2016), 3: 1170750.

Cover image
Source: Authors.

References