Synergy Action of Glass Powder and Foaming Additive in Production of Lightweight Cement-Based Materials

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Abstract. Waste glass in the form of finely grounded powder can be, due to its assumed pozzolanic behaviour, used in cement and concrete industry as a Supplementary Cementing Material (SCM) helping to reduce the consumption of natural resources and emission of greenhouse gases. In this sense, the presented paper is focused on the use of waste borosilicate glass in production of cement-based lightweight composites. For both Portland cement and glass admixture, chemical composition, pozzolanic activity and basic physical characteristics were accessed. In composite mixes, cement binder was partially replaced with glass powder in the amount of 15 and 20 mass %. In order to produce lightweight materials, foaming agent in the amount of 0.58 ml/kg of blended binder was used. For the developed materials, bulk density, matrix density, total open porosity, mechanical resistance, water transport parameters and thermal properties were tested. The obtained experimental results revealed pozzolanic activity of micro scale milled waste glass that led to the decrease in porosity and thus improved mechanical resistance of the investigated composites. High porosity of foamed concretes significantly improves their thermal-insulation function and helps to save raw materials for their production.

1. Introduction
Concrete as well as other cement-based materials belong among worldwide spread man-made building materials used not only in 20th century but also to these days. About 800 million tons of concrete was consumed in the U.S. in 2007, and the world consumption was estimated at 11 billion tons. It approximately corresponds to 1.7 ton for every living human being [1]. Excessive cement and concrete production is closely tied with high nature resources consumption as well as energy claims. In addition, cement production as highly polluting process contributes about 5-8 % to global CO2 emissions [2]. The application of supplementary cementitious materials in cement and concrete production introduce a promising method for mitigating environmental impacts and in many cases providing economic benefits. In general, concrete practice utilizes a wide variety of SCMs as in the form of active mineral admixtures as well as filler materials. Active powders, so called pozzolans, are defined according to the standard EN 197-1 (2011) [3], compartmentalized into nature and technogenic (artificial) material sort. Among very often incorporated admixtures is considered fly ash, silica fume, diatomite powder, blast furnace slag, etc. which are consumed in huge amount [4], [5], [6].

Glass waste represents urgent environmental problem all over the world from the point of its nonbiodegradable origin. This unused commodity is left on spread landfills releasing pollutants influencing water and soil [7]. In 2007, the world total production of glass was estimated about 89.4 million tons. From this amount European Union contributes by 38.3 million tons, corresponding approximately to 30 % of the total world’s output. About 83 % of glass production is attributed to...
container and flat glass [8]. From a sustainable point of view recycling procedure provides a suitable solution in waste glass reduction, nevertheless, this process is limited for different types of glass and their sorted combinations. Glass contains significant amount of silica and as a finely milled waste material can be very interesting alternative to common SCMs. However, its properties, such as chemical composition and particle size, affect its pozzolanic character. In this respect some authors, such as Khmíri et al. [9] and Shi et al. [10], recommend to mill the glass material with the help of ball mill to micro size range with particle size below 100 µm.

Glass powder incorporation into cement based materials positively helps to improve workability of fresh mix [1], however, recommended dosage supporting compressive strength is usually between 10 up to 20 mass %. Nassar and Soroshian [11] examined a different percentage substitution of cement by glass powder in portion of 15; 20 and 23 mass %. The compressive strength results revealed higher strengths for concretes containing 15 and 20 mass % of waste glass compared with control samples. Nevertheless, ordinary produced concretes cannot always meet the specific requirements of civil engineers. In recent years, the trend of application of lightweight concretes in construction has rapidly increased. This building material excels in a low content of input substances for its manufacturing as well as due to its advanced properties, such as low weight and very good sound and thermal insulation function. Together with fire resistance are lightweight composites predetermined as sound and thermal insulation layers, material for production of lightweight blocks and an infill material for lightweight composite panels [12], [13].

This paper is aimed at application of waste borosilicate glass in the form of fine powder in production of lightweight concretes. Developed materials were characterized by their physical properties, mechanical and liquid water transport parameters as well as their thermal properties. Experimental investigation carried out on both ordinary fine-grained and foamed concretes revealed positive effect of glass powder that acted as a suitable cement replacing material and foaming technology enabled produce an environmentally sustainable material with thermal-insulation function.

2. Experimental

2.1. Used materials

The composites preparation was done in two separate steps. At first, the basic reference mix containing Portland cement and fine silica aggregate was enriched by glass powder. Subsequently, the foaming additive was admixed into both reference and modified concrete mix. As a main binder substance, Portland cement 42.5 R produced by Heidelberg Cement Group Radotin, Czech Republic, was used. Part of cement was substituted by an artificially prepared borosilicate glass powder (GP) in the dosage of 15 and 20 mass %. Collected discarded glass vessels were washed in an ultrasonic bath, dried, manually crushed and milled in a disc mill. After milling, newly made waste glass powder disposed of an average particle size of 48.3 µm.

Chemical composition of applied materials is presented in Table 1.

| Table 1. Chemical composition of used powders. |
|-----------------------------------------------|
| Substance          | CEM 42.5 R (mass %) | GP (mass %) |
| SiO₂               | 19.00               | 78.5        |
| Al₂O₃              | 4.31                | 2.94        |
| Fe₂O₃              | 2.40                | 0.19        |
| CaO                | 62.90               | -           |
| MgO                | 1.80                | -           |
| K₂O                | 0.82                | 1.09        |
| Na₂O               | 0.14                | 4.55        |
| TiO₂               | 0.28                | -           |
| SO₃                | 3.24                | 10.50       |
| P₂O₅               | 0.16                | 0.16        |


The binders’ composition was accessed on the basis of classical chemical analysis. The particular materials were melted together with LiBO₂, and then dissolved into the solution. The solution composition was analysed with ICP spectrometer.

Basic material characteristics determined for both used binder materials are given in Table 2. The values of specific surfaces were measured according the standard EN 196-6 (2010) [14] with the help of Blaine apparatus. Obtained data revealed approximately 1.4 higher specific surface of glass powder compared to Portland cement. Pozzolanic activity of milled glass was determined by the modified Chapelle test (NF P 18-513:2012 [15]) in which reacts 1g of tested powder material with 2g CaO in water. In general, added mineral admixture is considered pozzolana active when 1 g of sample absorbs more than 650 mg of Ca(OH)₂.

| Material | Specific surface (m²kg⁻¹) | Density (kgm⁻³) | Loss on ignition at 1000 °C (%) | Pozzolanic activity (mgCa(OH)₂g⁻¹) |
|----------|--------------------------|----------------|-------------------------------|-------------------------------|
| CEM I    | 360                      | 3 129          | 0.40                          | -                             |
| GP       | 517                      | 2 345          | 0.02                          | 719                           |

2.2. Developed mixtures and sampling

All prepared mixes contained three fractions of silica sand 0.0/0.5; 0.5/1.0 and 1.0/2.0 (Filtrační písky, Ltd.) which were combined together in mass ratio 1/1/1. The amount of batch water was kept constant with water/binder ratio 0.5. In case of foamed lightweight composites, foaming additive (FA) MasterCell 285 produced by BASF Stavební hmoty, Ltd., Czech Republic was added to batch water in the amount of 0.58 ml/kg of binder. Chemical additive contained 30 mass % of a dry matter and had density of 1.12 kgm⁻³. Residual water of foaming agent was deducted from the total amount of batch water. Composition of all developed mixes is given in Table 3.

| Substance | Content in kg/m³ |
|-----------|------------------|
|           | Dense composites | Foamed composites |
|           | R-C   | GP 15-C | GP 20-C | R-F   | GP 15-F | GP 20-F |
| CEM I 14.25 | 496.2 | 421.8   | 397.0   | 320.8 | 272.7   | 256.6   |
| GP       | -     | 74.4    | 99.2    | -     | 48.1    | 64.2    |
| Sand 0.0/0.5 | 496.2 | 496.2   | 496.2   | 320.8 | 320.8   | 320.8   |
| Sand 0.5/1.0 | 496.2 | 496.2   | 496.2   | 320.8 | 320.8   | 320.8   |
| Sand 1.0/2.0 | 496.2 | 496.2   | 496.2   | 320.8 | 320.8   | 320.8   |
| FA       | -     | -       | -       | 0.22  | 0.22    | 0.22    |
| Water    | 248.1 | 248.1   | 248.1   | 160.2 | 160.2   | 160.2   |

From Table 3, there are evident savings in dosage of input raw materials for production of lightweight concretes. It was due to the use of foaming additive that allows increase of material volume compared to the reference concrete mix.

Mixing procedure included dosage of dry raw materials in a mixer with vertical axis Spar D 200-Band and their mixing at speed 96 rpm (1st speed regime) for 1 min. After that, batch water with or without liquid foaming agent was added and mixing procedure continued another 1 minute. Then, fresh mix was manually mixed in order to remove the scum from the bottom of mixing vessel. Materials without foaming were after that mixed in 2nd speed regime (196 rpm) for 2 min and their mixing was finished in 1st speed regime that was used for 1 min. Preparation of foamed mixes was finished in 2nd speed regime applied for 4 min.

From fresh mixes were casted prisms having dimensions of 40 × 40 × 160 mm and cubes with side of 70 mm. Samples prepared from dense mixes were casted in two layers, whereas each layer was compacted on vibrating table for 30 s.
In case of foamed concrete specimens, every layer was compacted by 3 hits on a table. Casted specimens were left in laboratory conditions at temperature of 23 ± 1 °C and 45 ± 5 % of relative humidity. In order to prevent excessive evaporation of batch water and autogenous shrinkage, the specimens were covered with PE foil for 1 day. Then these were unmoulded and stored in water (dense concrete samples) or in laboratory conditions covered with PE foil and water sprayed ones per day (foamed concrete samples) for overall time of 28 days.

2.3. Measured properties

2.3.1. Particle size distribution. Except specific surface, cement and GP were also characterized by their particle size distribution. For the measurement, red and green laser equipped analyser Analysette 22 – Micro Tec Plus (Fritsch) was applied. This apparatus is able to detect grains with diameter from 2 nm up to 2 000 µm. In the measurement, spherical grains’ shape is assumed.

2.3.2. Penetration time. The influence of glass admixture as well as consequent effect of chemical additive on rheology of fresh concretes were characterized by the measurement of penetration time in accordance with the technical standard EN 1015-9 (1999) [16], where the value of compressive stress caused by penetration cylinder is supposed to reach 0.5 MPa. This stress is considered as a border value for suitable concrete processing. Experiments were performed at 23 ± 1 °C for minimum two samples of each studied concrete mix.

2.3.3. Basic physical properties. For dried hardened specimens cured for 28 days, basic physical parameters, such as bulk density, matrix density and total open porosity, were accessed. The bulk density measurements were performed with utilization of gravimetric method according to the standard EN 1015-10 (1999) [17]. The relative expanded uncertainty of the density test was 2.3 %. For determination of the matrix density, device Pycnomatic ATC (Thermo Scientific) working on a helium pycnometry principle, was used. The total open porosity was calculated on the basis of measured matrix and bulk densities. The relative expanded uncertainty of the total open porosity test was 3.6 %.

2.3.4. Strength parameters and dynamic moduli measurements. Mechanical resistance of the examined materials was studied by flexural and compressive strength tests that were performed according to the standard EN 1015 – 11 (1999) [18]. Three point bending test was realised on three prismatic samples with dimensions of 40 × 40 × 160 mm. On the rest of broken prisms, the compressive strength was measured. The expanded measuring uncertainty of the both strength tests was 2 % [19]. Among mechanical parameters testing, dynamic moduli measurement according to the standard EN 12504-4 was done. For this purpose, device Dio 562 NLF (Starman Electronics) was applied [20]. The relative expanded uncertainty of the dynamic modulus measurement was 2 %.

2.3.5. Liquid water transport properties. One dimensional free water uptake test was carried out according the procedure described in the standard EN 1015-18 (2002) [21]. Hardened cubic samples were previously water and vapour proof insulated by epoxy resin on all lateral sides. The face side of each sample was immersed 1-3 mm into water on the top of a water reservoir, allowing samples to suck water from free water surface. The sample mass was measured in the chosen times interval. The water absorption coefficient \( A \) (kgm\(^{-2}\)s\(^{-1/2}\)) was calculated from the linear part of the dependence of the increasing sample mass on the square root of suction time. From the known weight of dry and fully saturated sample, the saturated moisture content value \( w_{sat} \) (kgm\(^{-3}\)) was calculated. The apparent moisture diffusivity \( \kappa_{app} \) (m\(^2\)s\(^{-1}\)) was calculated based on \( w_{sat} \) and \( A \) data using procedure reported by Roels at al. [22].

2.3.6. Thermo-physical properties. Among the thermal properties, thermal conductivity \( \lambda \) (Wm\(^{-1}\)K\(^{-1}\)), thermal diffusivity \( \alpha \) (m\(^2\)s\(^{-1}\)) and volumetric heat capacity \( c \) (Jm\(^{-3}\)K\(^{-1}\)) were measured with the help of commercially produced device ISOMET 2114 (Applied Precision) working on a dynamic
measurement principle [23]. The measuring range of the thermal conductivity is from 0.015 to 6 Wm$^{-1}$K$^{-1}$. The accuracy is 5 % of reading + 0.001 Wm$^{-1}$K$^{-1}$. The measurement reproducibility is 3 % of reading in operation temperature range from 0 to 40°C. The measuring range of the volumetric heat capacity is $4 \times 10^4 - 4 \times 10^6$ Jm$^{-3}$K$^{-1}$ with accuracy 15 % of reading + $1 \times 10^3$ Jm$^{-3}$K$^{-1}$. The reproducibility of the volumetric heat capacity measurement is 3 % of reading + $1 \times 10^3$ Jm$^{-3}$K$^{-1}$.

3. Results and discussion

Particle size distribution curves of cement and glass powder are graphed in Figure 1. The both measured distribution curves exhibit bimodal character with two local maxima. In case of Portland cement, curve peaks correspond to the particle diameter 8 µm and 30.2 µm respectively. Curve characterising glass admixture is more spread with local maxima at 5 µm and 80.4 µm. In comparison with cement binder, used mineral admixture contains a considerable amount of coarser particles that may also bring positive filler effect. On the other hand, higher volume of coarser grains is generally linked with a lower pozzolanic activity of SCMs [24].

![Figure 1. Particle size distribution of cement and glass powder.](image)

Basic physical properties measured on hardened samples cured for 28 days in a laboratory or in water are summarized in Table 4. Portland cement substitution by milled glass brought reduction in the total open porosity especially in case of dense fine-grained concretes. The observed differences in porosity were 8.1 % for GP 15-C and 10.6 % for GP 20-C related to the reference material (R-C). This behaviour is attributed to the pozzolanic activity of glass and additionally to the partial filler effect due to the presence of coarser GP grains. On the other hand, combined action of mineral admixture and foaming agent used in production of lightweight samples caused only slight decrease in porosity that varied in the relative expanded uncertainty of the applied testing method. In comparison with hardened concretes, foamed materials reached approximately three times higher porosity values. This data points to the significant weight decrease of foamed materials compared to the reference concrete. Based on that, better thermal-insulation function of lightweight concretes can be anticipated.

| Material | Bulk density (kgm$^{-3}$) | Matrix density (kgm$^{-3}$) | Total open porosity (%) |
|----------|--------------------------|----------------------------|-------------------------|
| R-C      | 2.107                    | 2.511                      | 16.1                    |
| GP 15-C  | 2.105                    | 2.471                      | 14.8                    |
| GP 20-C  | 2.110                    | 2.465                      | 14.4                    |
| R-F      | 1.336                    | 2.495                      | 46.5                    |
| GP 15-F  | 1.337                    | 2.491                      | 46.3                    |
| GP 20-F  | 1.331                    | 2.485                      | 46.4                    |
Table 5 shows mechanical parameters and penetration time data measured for examined concrete samples. Obtained mechanical parameters, represented by the flexural strength, compressive strength and dynamic moduli exhibited distinctly increasing character for dense concrete samples modified with GP. Similar improvement in mechanical resistance we observed also for lightweight concretes, however, the differences compared to the reference foamed concrete were not such remarkable. In general, the measured mechanical parameters correspond with the total open porosity data. From the quantitative point of view the lightweight materials reached low strength, nevertheless, they are worth because of their specific advanced properties. Incorporation of GP into fresh concrete mixes has no negative impact on workability when the values of spreading oscillated between 160/160 mm and 180/180 mm. However, significant changes in workability were recorded from the point of view of measured penetration times. One can see a gradual shortening of penetration time with the increasing dosage of GP in the dense concrete mix. On the other hand, penetration times measured for foamed mixes were distinctively higher compared to the dense concrete mixes. The prolongation of penetration time was a consequence of encased air bubbles network uniformly dispersed in a fresh mix. Certain effect may be also attributed to foaming agent, which as a chemical additive might possibly affect hydration process of Portland cement.

| Material | Flexural strength (MPa) | Compressive strength (MPa) | Dynamic moduli (GPa) | Penetration time (min) |
|----------|-------------------------|---------------------------|----------------------|------------------------|
| R-C      | 9.1                     | 54.1                      | 32.2                 | 145                    |
| GP 15-C  | 11.2                    | 59.2                      | 34.3                 | 130                    |
| GP 20-C  | 11.5                    | 61.2                      | 35.4                 | 115                    |
| R-F      | 1.7                     | 4.7                       | 4.6                  | 355                    |
| GP 15-F  | 2.0                     | 4.9                       | 4.8                  | 480                    |
| GP 20-F  | 1.8                     | 4.7                       | 4.6                  | 460                    |

Material parameters characterizing the liquid water transport are expressed in Table 6. Results obtained for dense concretes with GP showed a slight decrease in the water absorption coefficient and thus sorptivity. This was accompanied by the drop in the capillary moisture content when the lowest values were recorded for concrete with 20 mass % of GP. On the contrary, foamed concretes exhibited practically unchanged water transport characteristics regarding to their initial state. As evident from different structural nature, lightweight samples, as more porous materials, are able to easily transport liquid water which clarifies their increased apparent moisture diffusivity.

| Material | A (kgm\(^{-2}\)s\(^{1/2}\)) | \(w_{eq}\) (kgm\(^{-3}\)) | \(\kappa_{app}\) (m\(^{-1}\)s\(^{1}\)) | \(S\) (m\(^{-1}\)s\(^{1/2}\)) |
|----------|-------------------------------|-----------------------------|----------------------------------|--------------------------|
| R-C      | 0.018                         | 174.4                       | 1.09 \times 10^8                 | 1.82 \times 10^{-5}     |
| GP 15-C  | 0.017                         | 172.2                       | 9.74 \times 10^9                 | 1.70 \times 10^{-5}     |
| GP 20-C  | 0.016                         | 169.9                       | 8.92 \times 10^9                 | 1.61 \times 10^{-5}     |
| R-F      | 0.025                         | 455.2                       | 3.04 \times 10^9                 | 2.52 \times 10^{-5}     |
| GP 15-F  | 0.024                         | 450.0                       | 2.87 \times 10^9                 | 2.41 \times 10^{-5}     |
| GP 20-F  | 0.025                         | 453.0                       | 3.14 \times 10^9                 | 2.55 \times 10^{-5}     |

Thermal properties measured on dried cubic samples are summarized in Table 7. Compared to the reference material R-C, a gradual increase in both the thermal conductivity and diffusivity with increasing amount of GP in dense concretes was observed. This is in agreement with results presented above and clearly corresponds with porosity data. Moreover, the explanation of heat transport parameters may be provided by comparison of the thermal conductivities measured for all dry input substances. The particular raw materials were placed into glass cylinder and vibrated for 10 sec.
Obtained data revealed that the average value of the thermal conductivity for borosilicate glass powder was 0.84 Wm⁻¹K⁻¹; 0.10 Wm⁻¹K⁻¹ for cement, and 0.56 Wm⁻¹K⁻¹ in case of silica sand mix. As was expected on the basis of measured open porosities, the thermal conductivity values accessed for foamed samples were three times lower contrary to the dense concretes. On this account, lightweight composites are able to provide much better thermal-insulation function than ordinary concretes. For all analysed concrete samples, the volumetric heat capacity corresponded to the porosity data and other investigated material characteristics.

### Table 7. Thermo-physical properties of tested composites.

| Material | λ (Wm⁻¹K⁻¹) | a (× 10⁻⁶ m²s⁻¹) | c (× 10⁶ Jm⁻³K⁻¹) |
|----------|-------------|------------------|------------------|
| R-C      | 2.10        | 1.14             | 1.84             |
| GP 15-C  | 2.15        | 1.16             | 1.85             |
| GP 20-C  | 2.5         | 1.40             | 1.86             |
| R-F      | 0.76        | 0.48             | 1.59             |
| GP 15-F  | 0.81        | 0.50             | 1.60             |
| GP 20-F  | 0.76        | 0.47             | 1.59             |

### 4. Conclusions
A combined effect of waste borosilicate glass powder and foaming additive on properties of both fresh and hardened concretes was examined. Tests performed on fresh non-foamed mixtures showed a gradual shortening of penetration times with increasing portion of glass admixture attributed to its pozzolanic activity. On the other hand, foamed mixes reached desired penetration pressure much later due to presence of encased bubbles network. Observed decreasing porosity values corresponding with the better mechanical resistance determined for dense concrete samples indicated positive filler effect and pozzolanic activity of glass powder. Three times higher porosity of foamed concretes led to the significant reduction in strength characteristics, however, on the other hand to the enhanced thermal-insulation function. Lightweight foamed composites disposing with advanced properties may provide a possible material sort applicable for specific construction requests.

Our future work will be aimed at the possible use of different kinds of industrial by-products as mineral pozzolanic admixtures for production of foamed lightweight composites and research on the synergistic performance of pozzolanic materials and foaming agents of different origin.

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