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# Bulk density decreasing of lightweight silicate based composite

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**Abstract.** The newly developed silicate composite combines advantages of two types of bulk density decreasing methods. Direct bulk density decreasing method is based on introduction of pores in the structure of binder and indirect bulk density decreasing is achieved by means of using fine grained porous aggregate. This combination makes it possible to achieve maximal decrease of bulk density of the mix. Material of this type shows very low bulk density, approx.  $300 \text{ kg}\cdot\text{m}^{-3}$  and appropriate application would be for non-structural, filling material or insulating material. Achieved results will serve as base for further modification of mix-design of aerated concrete utilizing secondary raw materials.

## 1. Introduction

The main target of the research was verification of possibilities of preparation of material based on aerated concrete without the autoclaving process. Lightened materials based on silicates (aerated concrete, perlite concrete and foam concrete (light weight cellular concrete) present a very good material base for filling materials in all respects. Their production is not costly. Production processes can be modified so that secondary raw materials can be used.

Classic autoclaved aerated concrete is prepared as a mix of cement, lime and gypsum used as binding elements, fine silicates, aerating powder and water. Because of chemical reaction, the mix expands and volume of the mix doubles and creates highly porous structure. On average, 80% of volume of the material is created by pores, 50% of air pores and 30% of micro-pores. Microstructure of autoclaved aerated concrete is created mainly by tobermorite. [1] [2]

Macroscopic pores are formed thanks to expansion of the material and caused by aeration; micro-pores are formed in the walls between macroscopic pores. Addition of fly ash enhances porous structure and inter-facial transition zones between pores and matrix. N. Narayaan a K. Ramamurthy in their work describe changes of strength of aerated concrete depending on the way of curing, in particular impact on compressive strength and shrinkage. X-ray diffraction of K. Ramamurthy shows that the main reaction products of aerated concrete are those of group of tobermorite and calcium silicate hydrates (C-S-H). The reaction goes as follows: C-S-H rich in calcium  $\rightarrow$  C-S-H  $\rightarrow$   $11,3\text{\AA}$  tobermorite. The product of the reaction is a mix of crystalline, semi-crystalline and nearly amorphous phase of tobermorite. Crystalline structure of tobermorite is variable and it is characterized by proportion of tobermorite to C-S-H. [3][4]



R. Demirboğa and R. Gül described in their paper from 2003 reduction of bulk density and heat conductivity of samples by addition of silica fumes and fly ash. Thus they followed K. Lu-Shu, who described and experimentally formulated dependence between bulk density and thermal conductivity: thermal conductivity reduces with decreasing bulk density. [5]

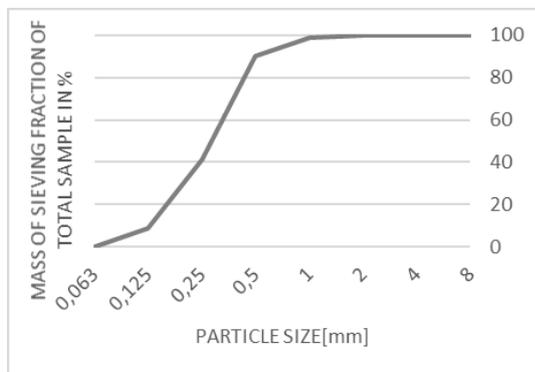
Y. Xia et al. dedicates one of his research to comparison of properties of autoclaved and non-autoclaved aerated concrete. This non-autoclaved material has a substantial advantage in facilitating the manufacturing process and reducing the cost of products. When Y. Xia and studied the microstructure of non-autoclaved concrete, he identified tiny Aft crystals and amorphous C-S-H gels. [6]

Many authors describe using porous aggregate in theirs research. [7][8][9] Expanded perlite has excellent thermo- and sound-insulating properties, it is volumetrically stabile, has low bulk density, is resistant to humidity, frost (-200 °C) and high temperatures (900 °C) and has excellent sorption properties. It is naturally occurring aluminosilicate volcanic stone that expands up to 20 times its volume when quickly heated to the softening temperature (900-1200 °C). [10] It is used as a filler for manufacture of thermo-insulating boards, backfills, filler for perlite plastering, mortars, lightened concretes, heat-resistant concrete, perlite mattresses, insulation of tanks for liquidized gases, filler in paints, filtration, in agriculture for lightening soil and regulation of soil humidity. Added into concrete, it enhances its properties, lightens it, reduces thermal conductivity, acoustic conductivity and increases resistance to shrinkage. [11][12][13]

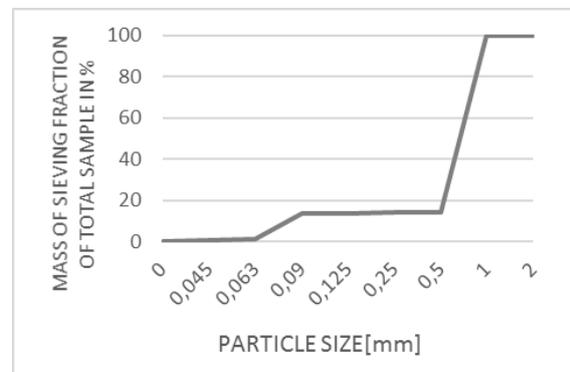
## 2. Materials and methods

Following input materials were used: expanded perlite EP 100, unslaked lime and cement CEM II 32.5 R, aluminum powder and fly ash for experimental partial replacement of binder.

Binders based on silicates like cement and unslaked lime have relatively high bulk density, which increases bulk density of resulting mix. To achieve density reduction, light-weight filler was used (expanded perlite) in combination with aluminum powder for direct decrease of bulk density.



**Figure 1.** Particle size distribution of expanded perlite.



**Figure 2.** Particle size distribution of used fly ash.

As lightweight aggregate the expanded perlite was used, its properties are in table 1.

**Table 1.** Properties of expanded perlite.

Bulk density	150	$\text{kg} \cdot \text{m}^{-3}$
Specific thermal capacity	0.85	$\text{kJ} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$
Heat conductivity coefficient	$0.16-3.92 \cdot 10^{-7}$	$\text{m}^2 \cdot \text{s}^{-1}$
Specific thermal conductivity	0.05	$\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$
Maximal temperatures for use	900	$^{\circ}\text{C}$

The fly ash was used as replacement of part of binder. Its chemical composition is stated in Table 2.

**Table 2.** Chemical composition of fly ash.

SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	FeO	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	annealing loss
43.2	0.7	19.5	3.3	1.1	<1.0	2	17.2	<1.0	1.1	5.31

Used aluminum powder is fine powder made of aluminum with purity of 99.7%, 89% of particles have dimension below 45  $\mu\text{m}$ , particles are spherical. It is a combustible solid, which releases combustible gases when in contact with water.

Physically-mechanical properties were determined on test specimens in the shape of a block with dimension 40×40×160 mm. Volumetric weight, compressive strength and flexural strength were determined. Mineralogical composition and microstructure by means of X-ray diffraction analysis was carried out (on the apparatus PANalytical Empyrean), electron microscopy (TESCAN MIRA 3 XMU) and optical microscopy (LEICA DM4000 M LED).

### 3. Discussion of results

Reference mix for this type of material was made of cement binder, filled with single fraction perlite EP100 in the amount 43% by weight. It seems as disadvantageous to measure expanded perlite by weight, but it is caused by low bulk density of expanded perlite. This proportion was determined on the basis of previous experiments, where it was found, that the mix is sufficiently workable, contains sufficient proportion of cement matrix which can be lightened with air pores. First, the reference samples with bulk density around 500  $\text{kg}\cdot\text{m}^{-3}$  were examined. Then, the influence of addition of various air entrainers was tested.

For example, calcium carbide at 0.1% by amount of cement had no influence on change of bulk density. At 5%, it destructed the whole structure. Moreover, at higher amount of calcium carbide, lot of acetylene is released, which blows up the mix, it is combustible and has a very unpleasant smell.

Another examined mix was perlite concrete with aerating agents (urea and sodium hypochlorite 5% by weight). The reaction should release gaseous nitrogen which should aerate the mix. However, neither this reaction reduced bulk density. On the surface of the material, undesired crystals were formed.

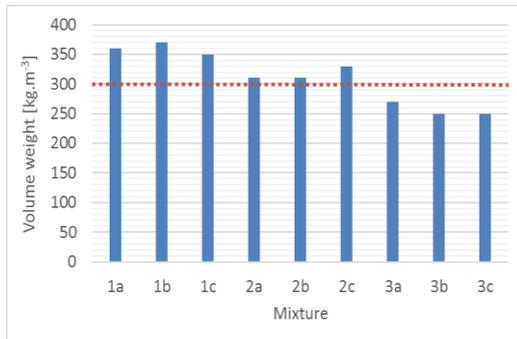
After these unsuccessful experiments, addition of Al powder was examined in subsequent stages. In the first stage of designing the material, the optimal amount and proportions of individual components of binder were experimentally determined so that lowest possible bulk density and highest strengths could be achieved (at least 0.1 MPa). The composition of mixtures is in the Table 3.

**Table 3.** The mixture composition.

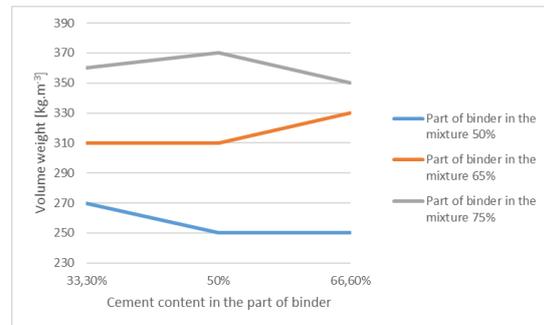
Mixture	Perlite EP100	Cement CEM II/B-M(S-L)32.5R	Lime CARMEUSE CL 90	Al-powder	Water/binder ratio
1a	25	50	25	0.8	2
1b	25	40	35	0.8	2
1c	25	25	50	0.8	2
2a	35	25	40	0.7	3
2b	35	35	30	0.7	3
2c	35	45	20	0.7	3
3a	50	15	35	0.5	6
3b	50	25	25	0.5	6
3c	50	35	15	0.5	6

### 3.1. Physical-mechanical properties

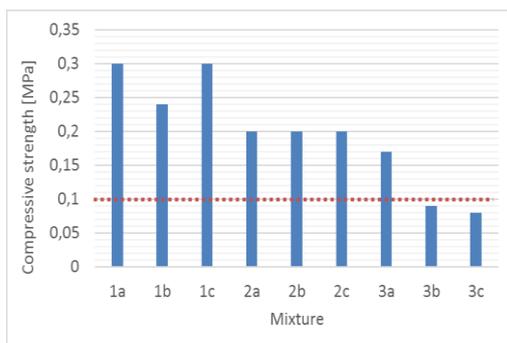
As the results imply, decreasing proportion of binder reduces not only bulk density but also strengths. The most significant drop of strength was observed in mix with 50% of binder and proportion of cement in binding component 50% by weight.



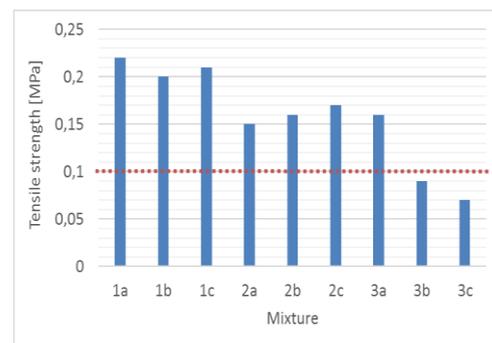
**Figure 3.** Bulk density, the maximal required value is  $300 \text{ kg}\cdot\text{m}^{-3}$ .



**Figure 4.** Bulk density, the maximal required value is  $300 \text{ kg}\cdot\text{m}^{-3}$ .



**Figure 5.** Compressive strength, the minimal required value is 0.1 MPa.



**Figure 6.** Flexural strength, minimal required value is 0.1 MPa.

There was an effort to replace part of the binder content with fly ash. But in this case it was not beneficial. The 10 % replacement of binder caused increase of bulk density of 20%, which means  $320 \text{ kg}\cdot\text{m}^{-3}$  replacement of 20 % caused increase of 25 % (it means  $340 \text{ kg}\cdot\text{m}^{-3}$ ), and that caused, that the limit was exceeded. The bulk density should have the value under  $300 \text{ kg}\cdot\text{m}^{-3}$ .

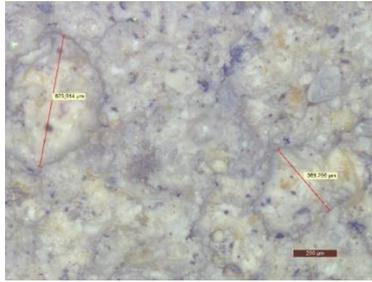
For this reason, mix with 75% of binders (without the replacement of binder with fly ash) was selected for further experiments. This material has bulk density  $270 \text{ kg}\cdot\text{m}^{-3}$ , compressive strength 0.17 MPa and flexural strength 0.16 MPa.

### 3.2. Microstructural analysis

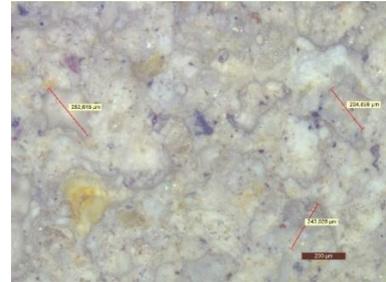
The Figure 5 shows that aluminum powder made the structure of the sample sufficiently porous and shape of individual pores is relatively regular, and their distribution is even in whole cross section. The size of pores in samples was from  $230 \mu\text{m}$  to  $630 \mu\text{m}$ . Figures 7-9 shows grains of filler (expanded perlite EP100) coated with cement-lime matrix.



**Figure 7.** Macroscopic structure of the sample.

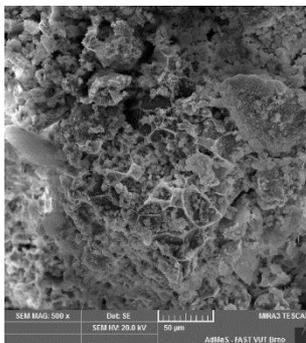


**Figure 8.** Porous structure of the sample determined with optical microscope.

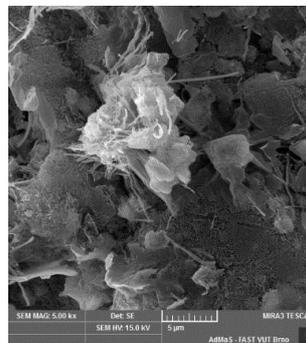


**Figure 9.** Porous structure of the sample determined with optical microscope.

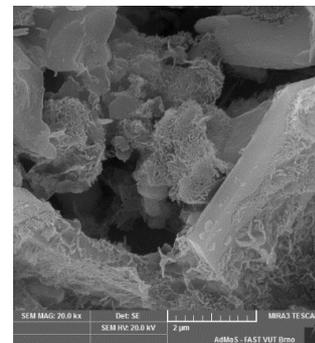
The image with 5000× magnification shows silicate composite exposed only to laboratory conditions, grains of portlandite, calcite and amorphous C–S–H gels in hydrated cement matrix are visible. This same concluded Xia, Y. in his work [6], too.



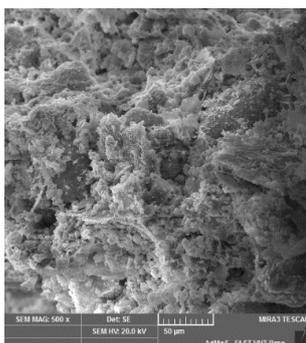
**Figure 10.** The image of the cement matrix, at 1 month age, at 500× magnification.



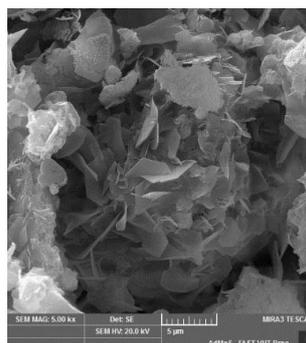
**Figure 11.** In the image of sample at 5000× magnification, crystals of portlandite in hydrated cement matrix are apparent, age 1 month.



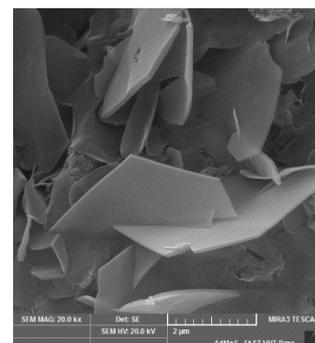
**Figure 12.** In the image at 20 000× magnification, hydrated cement matrix is visible, age 1 month.



**Figure 13.** The image of the cement matrix, at 1 year age, at 500× magnification.

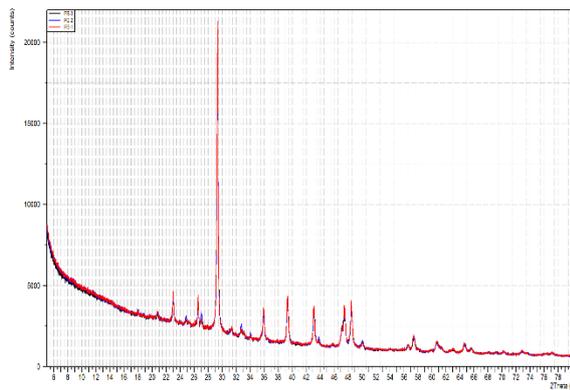


**Figure 14.** In the image of sample at 5000× magnification, crystals of portlandite in hydrated cement matrix are apparent, age 1 year.

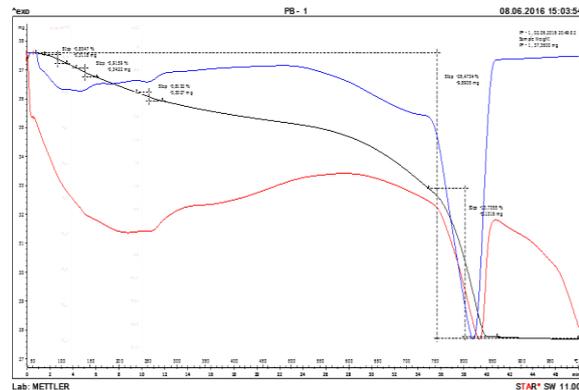


**Figure 15.** In the image at 20 000× magnification, hydrated cement matrix is visible, age 1 year.

X-ray diffraction analysis and differential thermal analysis implies that samples contain  $\beta$ -silica, calcite and portlandite, which was confirmed also by microscopic analyses.



**Figure 16.** X-ray diffraction analysis.



**Figure 17.** Differential thermal analysis.

#### 4. Conclusion

The properties of non-autoclaved aerated concrete, created by mix of unslaked lime, cement CEM II/B-M (S-L) 32.5 R, fine grained perlite EP 100 and aluminum powder, were experimentally determined.

The results imply that it is advantageous to use material with direct and indirect lightening of the structure. It is possible to achieve bulk density lower than  $300 \text{ kg}\cdot\text{m}^{-3}$ . Without the process of autoclaving, it is possible to achieve compressive strength about 0.1 MPa. This material could be used as material with several ways of use, for example as filling material with low bulk density or insulating material, its preparation could be done in-situ. The achieved results will serve as base for further modifications of mix-design of autoclaved aerated concrete with secondary raw materials, in the first stage with waste perlite.

#### Acknowledgments

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