The effect of mineral additives on foam concrete porosity

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Abstract. Non-autoclave foam concrete is a promising material for low-rise house construction. It has been found that the addition of mineral additives allows to regulate the porous structure of non-autoclave foam concrete, at the same time the coefficient of thermal conductivity is reduced, density is reduced, and frost resistance is improved. The introduction of dispersed wollastonite and diopsid promotes the formation of conditionally closed pores, their volume increases by 47-49%. The introduction of wollastonite reduces the pore volume with a diameter greater than 0.1 µm by 2.34 times, the introduction of diopsid reduces it by 8.2%. It is noted that the addition of the examined additives enables to obtain closed pores with a glossy surface; it improves the performance characteristics of natural hardening foam concrete. According to the results of DTA (Differential Thermal Analysis) and XRFA (X-ray fluorescence analysis), a more complete hydration of cement is recorded, this fact can indicate the structural and modifying influence of additives as crystallization centers for newgrowths.

1. Introduction
With the increasing volume of low-rise house construction non-autoclave foam concrete draws much attention as it has got low density, thermal conductivity, and relatively low cost [1-4]. However, this material has got high shrinkage deformations that lead to the increase of its density, deterioration of porous structure and heat insulation properties [5-6].

It is known that it is possible to improve the properties of cement materials by controlling porosity using various additives: mineral, chemical, nanomodified, etc. [7-10]. The sizes of pores are conditionally divided into five groups in a cement stone: large pores (over 5000 nm), macropores (1000-5000 nm), mesopores (500-1000 nanometers), micropores (100-500 nm) and nanopores (<100 nm).

The reduction of pore diameter can help to increase frost resistance of the material [11-12]. Closed pores with the dimensions less than 2 mm prevent convective heat exchange in foam concrete [13]. The research results of other authors also allowed establishing that fine-porous homogeneous structure will have higher density [14-16].

Natural calcium silicate mineral additives were earlier introduced to improve the quality of foam concrete, optimal compositions were determined, lower shrinkage of samples was noted, heat conductivity was reduced, and total porosity of the material was increased [17]. This paper evaluated the effect of natural wollastonite and diopsid on the porosity of natural hardening foam concrete.

The objective of the research is to study the influence of calcium silicate mineral additives on the porosity of non-autoclave cement foam concrete on the basis of man-made production wastes.
2. Experimental Method

Figure 1. Mercury porometry of non-autoclave foam concrete (a) without additives, (b) with diopsid, (c) with wollastonite.

2.1. Materials

Portland cement PC 500 D0 (Iskitim Cement Plant), acid fly ash (TPP-5, Novosibirsk), water, foaming agent Foamcem were used in the research.

Wollastonite CaO•SiO₂ and diopsid (Ca,MgO)•2SiO₂ are calcium silicates, that is, they are natural analogues of clinker minerals that can provide stronger adhesion of newgrowths of hardening cement paste to the surface of such silicate additives.

The wollastonite of Altay deposit was used with the following chemical composition, by weight, %: SiO₂ – 46.1; Al₂O₃ – 2.93; Fe₂O₃ – 4.44; CaO – 45.12; MgO – 0.9. The true density of wollastonite is 2455 kg/m³, the specific surface area is 90 m²/kg. Wollastonite additive is represented by elongated particles with the maximum length of 4-6 mm, and the minimum particle size is 150-200 µm.

The diopsid of Slyudyansk deposit was investigated with a true density of 2778 kg/m³ and a specific surface area of 100 m²/kg. The chemical composition of the diopsid is represented by the following elements, weight, %: CaO - 25.03, MgO - 20.01; SiO₂ – 51.33, Al₂O₃ – 1.88; Fe₂O₃ – 0.84;
MgO – 20.01; K₂O – 0.17. The used diopsid is represented by irregular-shaped particles with a particle size of 400-500 × 50-400 µm.

2.2. Procedure
Foam concrete samples were prepared for 4.5 minutes in a foam generator. The cement ash mixture was prepared separately: portland cement was mixed with fly ash and mineral additive for 2 minutes then water was added to the mixture. The mixture was prepared in a turbulent mixer for 3-4 minutes. Then technical foam was added to the gotten mortar mix with further stirring of the components for 2 minutes. Foam concrete tests were performed in 28 days of aging under normal conditions.

2.3. Experimental Test
The optimal amount of additives was found in the amount of 1% as a result of preliminary experiments.

The foam mix concentration is 2.5%. The optimal composition of foam concrete with D500 density was designed, where the ratio of aggregate to binder was 0.6, and W-S R (water-solids ratio) = 0.49.

Compressive strength (GOST 10180), average density (GOST 12730.1), dry thermal conductivity (GOST 30256), coefficients of thermal conductivity (GOST 30256), mark of frost resistance and moisture sorption (GOST 25485) were defined in accordance with normative documents.

The mineral composition of foam concrete samples was determined by D8 Advance diffractometer with CuKα-copper radiation unit detected with one-dimensional Lynx-Eye detector (Institute of Solid State Chemistry and Mechanochemistry of the Siberian Branch of the Russian Academy of Sciences).

The thermal behaviour of the samples was examined by STA 449 F1 Jupiter NETZSCH GmbH simultaneous thermal analysis instrument (Germany) in an argon current of high (99.995%) purity of 50 ml/min at a temperature range of 40-800°C at a constant heating rate of 10°C/min (ISSC SB RAS).

The determination of pore size and distribution in foam concrete samples was carried out by the method of mercury porosimetry by porosimeter Quantachrome PoreMaster 33 (examinations were carried out by the equipment of Shared Knowledge Center at Tomsk Polytechnic University). The maximum pressure for the porosimeter PoreMaster 33 is 33.000 psi. The range of determined pore sizes is 0.0064...950 µm.

The porosity of foam concrete was also determined in accordance with GOST 12730.4-78 “Concretes. Methods of determination of porosity parameters”.

The macroporosity of foam concrete was examined on samples sections using HITACHI TM-1000 scanning electron microscope (Institute of Solid State Chemistry and Mechanochemistry of the Siberian Branch of the Russian Academy of Sciences). The imaging was conducted in a low vacuum mode with the resolution of 30 nm.

3. Results
The results of mercury porometry for non-autoclave foam concrete with mineral additives are shown in Figure 1. The analysis of the obtained data shows that when wollastonite is introduced, the volume of pores with a diameter greater than 0.1 µm is reduced by 2.34 times. The introduction of diopsid reduces pore volume with a diameter greater than 1 µm by 8.2%, however the pore volume increases with a diameter less than 0.01 µm by 2.1 times. The authors [18-19] have shown that increasing the volume of fine pores with a diameter less than 1 µm in the product increases their frost resistance and resistance in aggressive media, thus adjusting the porosity by decreasing the pore size can increase the durability of building materials.

Physical and mechanical characteristics of the examined foam concrete are given in Table 1.
Table 1. Physical and mechanical characteristics of non-autoclave foam concrete.

| Parameter                                           | Type of an additive |
|-----------------------------------------------------|---------------------|
| Density (kg/m³)                                      | Control  | Diopside | Wollastonite |
|                                                     | 547      | 274      | 375         |
| Coefficients of thermal conductivity (W/(m•°C))      | 0.122    | 0.069    | 0.070       |
| Mark of frost resistance (%                       )    | 20       | 25       | 25          |
| Water absorption in weight,%                        | 28       | 45       | 40          |
| Moisture sorption with 97% relative air humidity of | 13       | 15       | 15          |

The density of the foam concrete decreases significantly when diopside is introduced compared with the situation when wollastonite is introduced, but the thermal conductivity coefficient is almost the same, this fact may indicate the difference in porosity.

The porosity of the foam concrete determined by water absorption is shown in Table 2. Lower microporosity was also obtained for non-autoclave foam concrete samples with wollastonite addition (0.55%) compared to diopside (0.44%), that may also indicate lower thermal conductivity of foam concrete 0.070 W/(m×°C) at higher density (375 kg/m³).

Table 2. Porosity of foam concrete of examined compositions.

| Parameter                                           | Type of an additive |
|-----------------------------------------------------|---------------------|
|                                                     | Control  | Diopside | Wollastonite |
| Total pore volume (%)                                | 67.0     | 87.0     | 81.0         |
| Volume of open capillary pores (%)                  | 14.9     | 13.6     | 15.3         |
| Volume of open non-capillary pores (%)              | 16.22    | 20.53    | 11.90        |
| The volume of conditionally closed pores of foam concrete (%) | 35.9 | 52.9 | 53.8 |
| Microporosity (%)                                   | 0.42     | 0.44     | 0.55         |

The largest volume of conditionally closed pores corresponds to foam concrete with wollastonite and diopside additives of 53.8% and 52.9% respectively. Due to this, the mark of frost resistance may be definitely increased up to F25.

Studying the macrostructure of samples by electron microscopy, closed pores with a glossy surface can also be noted when introducing the examined additives as opposed to foam concrete samples without additives (Figure 2, 3).

The volume of air involved in the system is increased while mixing the mixture and technical foam by adding the examined additives. At the same time the aggregate stability of foam concrete mixture [20] is increased, more closed pores are formed. It can be caused by the examined mineral additives that have a high chemical congeniality with portland cement hydration products, activate the crystallization processes of cement hydration products, i.e., act as a crystallizing component.

The quantitative ratio of hydration products can be indirectly measured by the intensity of calcium hydroxide diffraction reflection (49.20 * 10⁻²nm), higher intensity is the characteristic of wollastonite-added samples compared to control samples. This fact indicates a greater degree of cement hydration. When diopside is introduced, Ca(OH)₂ is used to form an additional amount of low-base calcium hydrosilicates, the highest intensity (30.40*10⁻²nm) on the X-ray pattern corresponds to diopside samples.

It may indicate the completeness of cement hydration. The results of differential thermal analysis also show a deeper hydration of cement for foam concrete samples with wollastonite and diopside that
corresponds to the highest weight losses in the area of the first endoeffect and total weight losses during heating.

Figure 2. The pore surface of foam concrete without additives.

Figure 3. The porosity of foam concrete with mineral additives.

4. Conclusion
The introduction of dispersed wollastonite and diopsid promotes the formation of conditionally closed pores, the volume increases by 47-49% that positively affects the properties of non-autoclave foam concrete. The microporosity increases when wollastonite is introduced by 31%, it can have an effect on the reduction of thermal conductivity of the material. According to mercury microscopy, when diopsid is introduced, pore volume with diameter less than 0.01 μm increases by 2.1 times, and when wollastonite is introduced, pore volume with diameter more than 0.1 μm decreases by 2.34 times.

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