Properties of foam concrete based on highly concentrated aluminosilicate binder suspension

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Abstract. Cementless binders and building materials are promising. The article presents results of production of foam concrete compositions using high-concentration aluminosilicate binder suspension based on granodiorite. Their basic physical and mechanical characteristics and microstructural features are described. The method of multifactor experiment was used, which allows to vary the parameters: water-solid ratio, amount of binder component and stabilizing additive. Thus, during steps of mathematical planning, optimal parameters were recommended, the observance of which is necessary to obtain cementless cellular composites with the best properties.

1. Introduction
In the current economic conditions, the already tested concrete materials, including heat-insulating and structural-heat-insulating non-autoclaved foam concrete, are popular.

The traditional representatives of these products are cement-based composites. Significant carbon dioxide emissions, high material and energy intensity of the cement industry, the complex manufacturing process, and the high cost of cement require the development and search for alternative types of binders for the production of non-autoclave foam concrete products using low- and non-clinker binders.

Scientists of Belgorod State Technological University n.a. V.G. Shukhov (Russia) achieved success in the development of composite cementless highly concentrated and nanostructured cementing systems, as well as their application for the production of building materials [1–14].

The team described issues of obtaining and studying a highly concentrated aluminosilicate binder suspension (HCABS) which is a cementless system based on intrusive rocks of acidic composition – granodiorites [12, 15].

This study presents the results of the development of foam concrete compositions using HCABS. It describes their structural and physico-mechanical characteristics which determine their role in the general classification of building materials.

2. Methods and materials
When designing effective construction materials, it is necessary to choose appropriate raw materials. Designing compositions with a number of standardized and specified building and technological properties determines a link in the production of both materials and structures based on them.

One of the methods to increase the research efficiency is the use of mathematical methods for planning experiments, which can reduce costs and duration of research while improving quality of the results.
The method of mathematical planning of experiments makes it possible to develop a model of the dependencies. The nature of the dependence can be unknown. But it can solve practical problems. It is enough to find its approximate expression.

The multivariate experiment method consists of the following steps:

- preliminary study of the object;
- building the mathematical model;
- interpretation of the mathematical model.

The experiment planning involves identification of the mathematical relationship between the material properties, their consumption and properties of constituent components and technological factors.

Depending on the established conditions of the problem during the experiments, all factors (input parameters – \( X_1, X_2, \ldots, X_n \)) vary at three levels: main (0), lower (–1) and upper (+1). In this case, the lower and upper levels differ from the main one by a certain value – the interval of variation (\( \Delta X_i \)).

The experiment is carried out according to the planning matrix and is determined by the number of factors and conditions.

To process the results, the method of mathematical statistics is used. It helps obtain algebraic equations that reflect the relationship between the varied factors and the results obtained during the research (output parameters).

Thus, the mathematical design of the experiment was used to develop rational foam concrete compositions. Humidity of the main binder component – HCABS (22 %), was taken into account. A stabilizing additive – polyvinyl alcohol (PVA) was used as a 5 % aqueous solution.

The optimization of properties was evaluated based on the analysis of physicomechanical characteristics of foam concrete: compressive strength and average density of the composite. The quantity of binder and additives, as well as the water-solid ratio (W/S) were used as variable parameters (Table I).

Table 1. Conditions for planning hcabs-based foam concrete composition development experiment

| Factors      | Variation levels | Variation interval |
|--------------|------------------|--------------------|
| natural     | codified         | –1    | 0   | +1     |
| W/S          | \( X_1 \)        | 0.35  | 0.40 | 0.45   | 0.05 |
| HCABS, kg    | \( X_2 \)        | 312   | 390  | 468    | 78   |
| PVA solution, kg | \( X_3 \)    | 0.75  | 1.25 | 1.75   | 0.5  |

* The composition is calculated based on the volume per 1 m\(^3\) of the mixture.

The study of the basic physical and mechanical characteristics of foam concrete samples was carried out according to the regulatory and technical documents of the Russian Federation: GOST 25485–89 “Cellular concretes. Specifications”, GOST 10180-2012 “Concrete. Methods for determining strength of control samples”, GOST 12730.1–78 “Concretes. Methods for determining density”, GOST 30256–94 “Method for determining thermal conductivity with a cylindrical probe”, GOST 25898–2012 “Building materials and products. Methods for determining vapor permeability”, GOST 24816–2014 “Building materials. Method for determining equilibrium sorption humidity.”

At the macro level, the study of structural parameters of foam concrete was carried out using a Hirox KH-7700 video microscope, at the micro level – using a high resolution scanning electron microscope Mira 3 FesSem in a high vacuum discharge mode at different magnifications.

Non-autoclave foam concrete was studied. This material was produced by mixing all components: the cementless binder, the GreenFroth protein blowing agent, water and a stabilizing additive which was used as a solution of polyvinyl alcohol. The speed of the mixing plant gradually increased to a maximum.

In the research, a highly concentrated aluminosilicate binder suspension based on the screening of crushing of holocrystalline acidic intrusive rocks – granodiorite (deposit in Pavlovsk, Russia) was used as the main binder component. The main properties of the suspension are shown in table II. The
technology for producing HCABS consists in one-stage mechanical activation of a coarse fraction of granodiorite screenings (> 1.25 mm) in the presence of a modifying component, which is sodium silicate solute. It should be noted that the choice of the optimal size fraction of the raw material is determined by the content of the minimum amount of layered aluminosilicates in it, which negatively affect the mechanochemical synthesis of the suspension [15].

Table 2. The properties of HCABS and stone based on HCABS

| Indicator (HCABS suspension) | Value | Indicator (stone based on HCABS) | Value |
|-----------------------------|-------|---------------------------------|-------|
| Grinding residue Nº 0063 [%] | less 1 | Compression strength [MPa] | 5.05 |
| Viscosity [Pa·s]            | 17–20 | Deflection strength [MPa]      | 2.10 |
| Environment pH              | 8.0–9.0 | Density [kg/m³]               | 2100 |
| Humidity [%]                | 20–22 | Specific efficiency of natural radionuclides [Bk/kg] | 60.8 |

3. Results and discussions

Based on the experimental planning conditions, a planning matrix was created (Table III). According to the matrix, foam concrete samples were molded. Basic physical and mechanical characteristics were determined (compressive strength and average density).

Table 3. Experiment planning matrix

| No | X₁ | X₂ | X₃ | W/S (X₁) | HCABS (X₂) | PVA (X₃) | Strength, MPa (Y¹) | Density, kg/m³ (Y²) |
|----|----|----|----|---------|-----------|---------|-------------------|---------------------|
| 1  | +1 | +1 | +1 | 0.45    | 468       | 1.75    | 1.35              | 541                 |
| 2  | +1 | +1 | –1 | 0.45    | 468       | 0.75    | 1.23              | 508                 |
| 3  | +1 | –1 | +1 | 0.45    | 312       | 1.75    | 0.85              | 342                 |
| 4  | +1 | –1 | –1 | 0.45    | 312       | 0.75    | 0.81              | 320                 |
| 5  | –1 | +1 | +1 | 0.35    | 468       | 1.75    | 1.48              | 527                 |
| 6  | –1 | +1 | –1 | 0.35    | 468       | 0.75    | 1.32              | 503                 |
| 7  | –1 | –1 | +1 | 0.35    | 312       | 1.75    | 0.92              | 346                 |
| 8  | –1 | –1 | –1 | 0.35    | 312       | 0.75    | 0.86              | 322                 |
| 9  | +1 | 0  | 0   | 0.45    | 390       | 1.25    | 1.07              | 419                 |
| 10 | –1 | 0  | 0   | 0.35    | 390       | 1.25    | 1.29              | 430                 |
| 11 | 0  | +1 | 0   | 0.40    | 468       | 1.25    | 1.33              | 513                 |
| 12 | 0  | –1 | 0   | 0.40    | 312       | 1.25    | 0.83              | 340                 |
| 13 | 0  | 0  | +1  | 0.40    | 390       | 1.75    | 1.03              | 412                 |
| 14 | 0  | 0  | –1  | 0.40    | 390       | 0.75    | 0.96              | 398                 |
| 15 | 0  | 0  | 0   | 0.40    | 390       | 1.25    | 1.12              | 406                 |

Based on the experimental data, regression equations were obtained for constructing nomograms of the dependence of compressive strength and density.

Regression equation for compressive strength:
\[ Y_1 = 1.103 - 0.056 \cdot X_1 + 0.244 \cdot X_2 + 0.045 \cdot X_3 + 0.094 \cdot X_1^2 - 0.006 \cdot X_2^2 - 0.091 \cdot X_3^2 - 0.013 \cdot X_1 \cdot X_2 + 0.023 \cdot X_2 \cdot X_3 - 0.008 \cdot X_1 \cdot X_3. \]

Regression equation for average density:
\[ Y_2 = 411.039 + 0.2 \cdot X_1 + 92.2 \cdot X_2 + 11.7 \cdot X_3 + 11.109 \cdot X_1^2 + 13.109 \cdot X_2^2 - 8.391 \cdot X_3^2 + 3.125 \cdot X_1 \cdot X_2 + 1.375 \cdot X_2 \cdot X_3 + 0.875 \cdot X_1 \cdot X_3. \]

Based on the regression equations, nomograms of the dependences of density and compressive strength were constructed (Figure 1).

Compliance with the given conditions allows us to produce a material with density of 320–540 kg/m³. When analyzing the data, one should take into account the influence of all factors both individually and in combination. First of all, an increase in the average density of foam concrete is due to the amount of binder introduced and directly related to this factor. Samples with W/S = 0.45 with an
increased binder content are characterized by the highest density values. A decrease in the amount of binder is accompanied by a decrease in density with the subsequent intersection of the planes W/S = 0.45 with W/S = 0.35 (Figure 1, a).

Figure 1. General view of the dependence of HCABS-based foam concrete characteristics on variation factors: a – average density; b – ultimate compressive strength.

The characteristics of the compositions with different water-solid ratios have no significant differences and are equal. With a high W/S value, the system has an excess of water which leads to a deterioration in quality of the material. As a result, an irrational structure has a negative effect on the basic physical and mechanical characteristics of the composite. When forming samples with W/S = 0.45, shrinkage was revealed. It is reflected in density values. The less water in the system (up to a possible acceptable value taking into account the moisture content of the binder itself and the amount of PVA solution), the higher the quality of concrete.

The constructed dependencies are confirmed by the presented strength nomograms (Figure 1, b). An increase in compressive strength is determined by the dependence on the water-solid ratio in the following sequence: W/S = 0.35 → W/S = 0.45 → W/S = 0.4. It is known that a large amount of water decreases the strength. In this case, the characteristics of samples with W/S = 0.45 are higher than those with W/S = 0.4. A decrease or loss of stability of the mixture occurs in the initial period of formation of coagulation structures. As a result, this leads to the subsidence and unjustified compaction of the system, and increased strength. In this case, the sample is characterized by the irrational structure of its porosity with pronounced open and torn pores; therefore, it is inferior in characteristics with W/S = 0.35.

Taking into account the regression equation, the maximum strength values were found. For D500 foam concrete, the highest value (R_{ad} = 1.52 MPa) was observed in samples with the following parameters: water-hard ratio W/S = 0.35, 468 kg of binder and 1.45 kg of PVA. For D400 foam concrete, the maximum strength (R_{ad} = 1.24 MPa) is achieved at W/S = 0.35, the amount of BABS 390 kg and PVA 1.15 kg. The minimum content of the additive (0.75 kg) does not have a significant structural effect and does not decrease and prevent destructive processes.

Thus, during the mathematical planning, the following parameters should be observed to obtain foam concrete based on HCABS with the best strength characteristics: minimum W/S (0.35), average PVA additive content, the amount of binder is determined by the required density of finished products.

Based on the analysis, the optimal compositions were selected and samples were produced. They were tested in accordance with GOST 25485–89 “Cellular concrete. Technical conditions.” Foam concrete products with W/S = 0.35 and density values corresponding to D400 and D500 were developed (Table IV).

According to GOST 25485–89, the following classes of compressive strength were established: D400 – B0.75 and B0.5; D500 – B1 and B0.75. The materials satisfy and exceed the requirements of
the regulatory documents. This is true for foam concrete D400. For the heat-insulating concrete, frost resistance is not standardized. Thermal conductivity indicators, in comparison with those stated in the GOST requirements decreased up to 20–30 %.

Table 4. Compositions and main characteristics of hcabs-based foam concrete

| Components | Characteristics |
|------------|-----------------|
| HCABS, kg  | Density Grade   |
| Frother, l | Tensile strength under compression, MPa |
| PVA solution, l | Strength class |
| Density, kg/m³ | Thermal conductivity, (W/m °C) |
| Density Grade | Vapor permeability, mg/(m · h · Pa) |
| Strength class | Sorption humidity, % |

Experimental data

| 390 | 6.3 | 23 | 419 | D400 | 1.24 | B1 | 0.08 | 0.23 | 5.6 | 9.3 |
| 468 | 7.9 | 28.7 | 524 | D500 | 1.52 | B1 | 0.085 | 0.21 | 5.9 | 10.1 |

Indicators according to GOST 25485–89

| D400 | B 0.75 | No more than 0.1 | No less than 0.23 |
| D500 | B 0.75 | No more than 0.12 | No less than 0.20 |

* calculation of the composition per 1 m³ of foam concrete

HCABS-based foam concrete is a neocomposite which requires a more detailed study.

When studying the structure of foam concrete at the macro level, the surface layer of the sample was analyzed. We investigated samples of foam concrete of a rational composition, i.e. modified with PVA; for visual comparison, we studied unmodified foam concrete.

The general structural characteristic of cellular materials consists of the description of pores and the spatial arrangement of pores, pore size distribution, maximum and average sizes, their shape, and thickness of inter-pore partitions.

Foam concrete of rational compositions is characterized by a uniform distribution of pores whose diameter is 200 μm – 2 mm (Figure 2, a). The polydisperse nature of the pore size distribution determines a denser inter-pore septum which positively affects the characteristics of the finished material as a whole.

Figure 2. The macrostructure of the HCABS-based foam concrete surface: a – modified, b – unmodified

An insignificant number of large pores is observed. Its large size is due to their fusion, as indicated by the elongated shape of these cellular formations. Pores have a size of 500-800 microns, a regular shape in the form of a sphere with closed porosity. By the number of expressed peaks, their distribution should be considered bimodal.
An unmodified foam concrete sample is characterized by a different structure (Figure 2, b). The failure of this material is already evident at the molding stage, when visual signs of the initial coagulation process appear. With further hardening, significant shrinkage of the mass occurs due to the macrostructure of the sample surface.

In the unmodified foam concrete sample, a pronounced large-pore structure is observed, pore sizes are about 2 mm. Pores have an irregular elongated shape. The large distance between the pores (1 mm) indicates that the system was initially unstable, and the cellular structure was partially destroyed. This indicates the impossibility of producing foam concrete materials based on HCABS without modification.

Figure 3. Photographs of the microstructure of non-autoclave foam concrete based on a cementless HCABS
Microstructural studies were carried out only with modified foam concrete compositions with a D400 density grade. The internal structure of the sample surface produced by the mechanical action was studied.

The general view of the microstructure shows the nature of the pore system expressed by cells in a large size range (Figure 3).

Porosity is determined by the presence of pores of a spherical shape with clear boundaries between them, no merging is observed which confirms the positive effect of the modifying component on the cellular structure of materials. The inter-pore septum has a different thickness of 20–100 microns. The total mass of the system is composed of a polydisperse matrix of angular particles.

With a larger-scale increase in various elements of the foam concrete structure (inter-pore septum, pore space surface), a large number of binder particles of various sizes appear on the surface.

The accumulation of nanoscale particles of regular shape attached to the surface of larger particles may indicate the epitaxial growth. This process determines the crystallization of silicic acid on quartz particles, and silicon-alumina acid – on plagioclase particles [15]. This statement is based on a previous study of the HCABS matrix system. It was found that during the hardening process of the binder suspension, the phase concentration changes. The amount of the amorphous component, consisting of colloidal solutions of silicic and alumosilicic acids, monotonously decreases, and the concentration of crystalline rock-forming components (quartz and feldspar) increase.

4. Conclusion

Thus, with application of method of mathematical planning non-autoclaved foam concrete based on a cementless highly concentrated aluminosilicate binder suspension was produced. It has the following characteristics: density – 419–524 kg/m$^3$; ultimate compressive strength – 1.24–1.52 MPa; thermal conductivity – 0.08–0.085 W/(m·ºC); vapor permeability – 0.21–0.23 mg/(m · h · Pa); sorption humidity – 5.6–5.9 and 9.3–10.1 % (with relative air humidity of 75 and 97 %, respectively). Moreover, the products comply with the requirements of regulatory and technical documents of the Russian Federation.

The structural features of foam concrete based on HCABS were studied. The positive effect of the modifying component in the form of polyvinyl alcohol on the structure formation of materials and preservation of the porous structure was described. The accumulation of nano-particles attached to the surface of larger particles indicates the occurrence of epitaxial crystallization in the binder suspension system.

Acknowledgment

The work was carried out as part of the Presidential Scholarship SP-2116.2018.1.

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