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Durability behaviors of foam concrete made of binder composites

V.S. Lesovik[a,b], E.S. Glagolev[c], V.V. Voronov[a], L.Kh. Zagorodnyuk[a], R.S. Fediuk*[d], A.V. Baranov[d], A.Kh. Alaskhanov[e], A.P. Svintsov[f]

[a] V.G. Shukhov Belgorod State Technological University, Belgorod, Russia
[b] Research Institute of Building Physics of the Russian Academy of Architecture and Building Sciences, Moscow, Russia
[c] Administration of the Belgorod region, Belgorod, Russia
[d] Far Eastern Federal University, Vladivostok, Russia
[e] Grozny State Oil Technical University named after Academician M.D. Millionshchikov, Grozny, Russia
[f] Peoples' Friendship University of Russia, Moscow, Russia
** E-mail: roman44@yandex.ru*

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Abstract. The article is devoted to the determination of the patterns of the formation of the microstructure of foam concrete using Portland cement, opoka marl and fly ash. Binder composites obtained by joint grinding of these were prepared in the form of new compounds, on the basis of which concrete with improved mechanical properties and performance characteristics are created. The complex of experimental studies included studies of the thermal intensity of hydration, shrinkage, average density and compressive strength. A number of operational characteristics were also comprehensively investigated: frost resistance, thermal conductivity and vapor permeability. Both microstructural and morphological studies of the developed composites were investigated using the analysis of SEM images, X-ray diffraction patterns and DTA patterns. The experimental results of composite binders and foam concrete based on it are presented. The mechanism of the influence of fly ash on the formation of the microstructure of the foam concrete mixture for building envelopes is determined. Binder composites obtained by co-grinding the components have a compressive strength of up to 60 MPa with Portland cement savings of up to 40 %. Based on the binder composites, foam concrete with a density of 500–700 kg/m^3 and compressive strength above 4 MPa was obtained. In addition, a technological scheme was developed for the production of non-autoclaved foam concrete for the manufacture of blocks, as well as for monolithic construction.

1. Introduction

The natural energy resources are running out and are became more expensive, but at the same time the construction industries spend huge large quantities and very uneconomically [1–2]. About 30 % of fuel resources are spent on creating thermal comfort in the premises. At the same time, almost a third of these resources lost in the process of transportation, as well as through leaks through the enclosing structures of buildings and structures [3–4]. Only through the introduction of energy-efficient building materials that combine multifunctionality and low cost, it can optimize the construction and improve the thermal characteristics of buildings and structures [5–7]. One such material is cellular concrete [8–10].

Naturally, to reduce energy costs, it is necessary to exclude autoclave processing from the list of technological processes necessary for the production of this class of concrete. However, it is rather difficult to obtain a durable material with an optimal pore structure. Complex modification of the mixture through the use of active mineral components contributes to the quality indicators of the cellular material. This, on the one hand, increases the stability and viability of the foam concrete mix, and on the other hand, increases the complex of physicomechanical quality indicators of the composite [11–15].

Using special composite binders that optimize the synthesis process at all stages from foaming and porosity of the mixture to curing and operation of the composite, it is possible to increase the efficiency of

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these building materials [16–18]. The selection of silica-containing components from both technogenic and natural raw materials is the most important task on this path. The use of these additives allows not only to reduce the consumption of the most energy-consuming and expensive component of foam concrete – Portland cement, but also allows you to control the structure formation of the cellular composite [19–21].

Among the main advantages of foam concrete (Fig. 1), is that, Chica and Alzate [22] highlighted the low weight of the material and structures made using foam concrete, which reduces the load on the foundations and, thus, reduces the cost of their construction. The low weight of the products results in low transportation costs. The article [23] states that foam concrete is characterized by high vapor permeability with a simultaneous coefficient of thermal conductivity. This indicates a very important fact – the material "breathes", while not violating the humidity conditions, both indoors and inside the structure itself. Other researchers [24] cite facts characterizing the high durability of non-autoclave foam concrete.

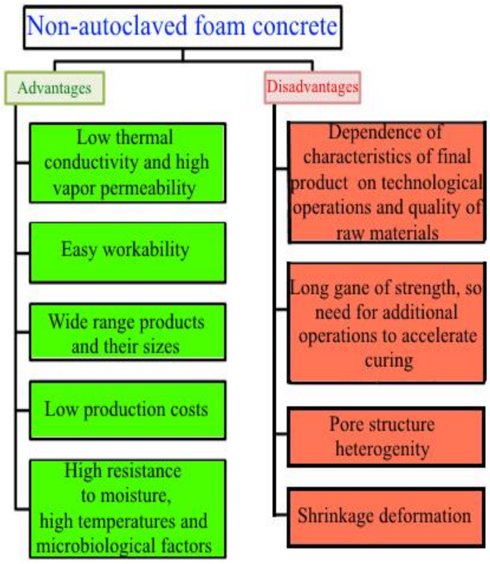

Figure 1. Advantages and disadvantages of non-autoclaved foam concrete.

A wide range of products and their sizes is made possible thanks to the simple workability of the material and the possibility of using both blocks and monolithic foam concrete. There are a number of technology foam concrete blocks, for example, as a result of pouring into molds to match the size of the product, or cutting into blocks of larger arrays. The authors of article [25] highlight the simplicity and affordability of technology, as well as the low cost of production, as advantages of non-autoclaved foam concrete. Given all this, there is a huge number of manufacturers of both raw materials (foaming agents and binders - traditionally use Portland cement), and the blocks themselves made of foam concrete. Despite this, foam concrete has a number of drawbacks, the main one of which is shrinkage resulting from a long-term increase in strength [26–27]. Immediately after the preparation of the foam, processes of spontaneous destruction immediately arise, which cease only when the composite is significantly hardened. Accordingly, to reduce these deformations, it is necessary to ensure stable characteristics of the foam [28–29]. It is known that for cement composites it is necessary to control that the hydration of clinker minerals takes place in full; only in this case is the required strength of the hardened composite

Given all of the above, it can be concluded that the process of forming the macrostructure of aerated concrete is difficult to control and regulate [32–33]. This conclusion is explained by the need to simultaneously control a large number of technological parameters: the quality of the raw material and the accuracy of its dosage, the water-solid ratio of the system and its rheological characteristics, temperature and pH of the medium, which change during the manufacturing and curing of foam concrete [34–36].

Using prescription and technological methods, it is possible to reduce the negative effect of these shortcomings. The use of modifying additives, the selection and development of foaming additives, quality control of materials and the flow of technological processes can improve the efficiency of the manufactured cellular concrete.

Summarizing all of the above, we note the following. Despite the fact that a lot of research has been done on foam concrete, there are many “white spots” that need to be addressed as soon as possible. Thus, the object of the study are non-autoclave foam concrete. And the subject of research in this case will be the durability characteristics of these promising materials.

The goal of the study is to optimize the durability of non-autoclaved foam concrete using a composite binder. In the course of achieving this goal, a number of tasks were solved:

a) research of optimal compositions of foam concrete on binder composites via both organic and mineral admixtures;

b) study of the possibility of controlling the processes of structure formation at the synthesis of foam concrete made based on binder composites;

c) study of the properties of modified foam cement systems and the development of materials science and technological methods for their regulation;

d) research and development of composites for both structural and heat-insulating un-autoclaved foam materials.

2. Materials and Methods

2.1. Materials

For synthesize of binder composite and foam concrete for monolithic construction, opoka marl (OM) and Portland cement CEM I 42.5N (Belgorod cement, Russian Federation) were used. Foam concrete for the production of blocks was prepared on the binder composite using the same Portland cement as well as fly ash (FA) from Novotroitskaya TPP (Russian Federation). The chemical content of the CEM I 42.5N used are shown in Table 1.

Table 1. Chemical content of CEM I 42.5N.

Chemical composition (%)						
calcium oxide	silica	aluminium oxide	iron oxide	magnesium oxide	sulfur oxide	sodium oxide
65.9	21.7	5.0	4.2	1.25	0.40	0.78

The opoka marl belongs to the group of medium and highly leached marls, which contain CaO – 28.0–33.0 %. This is a dense rock of gray color with a green tint, often fractured with thin deposits of iron hydroxides along the crack planes, a random texture, pelitomorphous globular, relict organogenic structure with varying silica and calcium carbonate contents. The main rock-forming mineral of the opoka-like marl is represented by organogenic calcite, the average content of which is 35 ... 38 %, opal – up to 15 %, the rest is mixed-layer clay formations and zeolites, clay minerals are replaced by opal. A feature of these minerals is that some of them are amorphous or have a defective crystal lattice, which determines their sorption and pozzolanic activity. The natural moisture content of the rock is 21–26 %; porosity – about 47 %; the ductility number is about 12.3 %; a fraction content of less than 0.005 mm is 58–65 %; average total radioactivity A_{eff} = 56.0 bc/kg). The natural moisture content of the rock is 21–26 %, porosity is about 47 %, and the ductility number is 13.5.

The Muraplast FK 19 superplasticizer (SP) (MC-Bauchemie, Germany) was used for improve the rheological characteristics of the mixes. The alpha olefin sulfonate sodium ASCO 93 (Korea) was used as a foaming agent.

2.2. Methods

A MicroSizer 201 laser particle analyzer (Scientific instruments, Russian Federation), a Reostat 4.1 rotational viscometer (Germany) and a high resolution TESCAN MIRA 3 LMU scanning microscope (Czech Republic) were carried out for the study of raw materials, binder composites and foam concretes. The X-ray diffraction patterns of the samples were tested by an ARL XTRA device (United States) by the method of powder X-ray diffraction. A STA 449 F1 Jupiter derivatograph (NETZSCH, Germany) was used for obtaining the differential-thermal (DTA) patterns of the samples.

A ToniCAL 7338 differential heat flow calorimeter (Toni Technik, Germany) tests the binder hydration in the early stages. The method of a cylindrical probe was used for the study of the thermal conductivity of the cellular concrete by an ITP – MG4 Probe thermal conductivity meter (Stroypribor, Russian Federation). Frost resistance was researched on specimens of 100×100×100 mm size by a Polair CV-105S freezer (Russian Federation) at a temperature of -18°C; each freezing cycle was 150 minutes, the thawing cycle at a temperature of 20°C – 120 minutes. The UNI EN ISO 12572 method using specimens of 200×100×70 mm in size was used to study vapor permeability.

3. Results and Discussion

3.1. Mix design

The structure and properties of binder composites are determined by the choice of starting materials – cement and the type of mineral additive, as well as their ratio, dispersion, activity and interaction. To determine the rational amount of mineral additives in the composition of binder composites, various doses were added, varying the amount of cement in the range 50...90 %, opoka marl – 2.5...12.5 % and fly ash – 10...50 %. The opoka marl was pre-dried, then crushed by a laboratory jaw crusher and then crushed in a vibration mill to a specific surface area up to 500 m2/kg.

In the course of further studies, the nature of the influence of the composition of the binder composite on their physicomechanical characteristics was revealed (Table 2). At the same time, the strength characteristics of the BC specimens with a binary mineral additive (40 % fly ash + 10 % opoka marl) increase up to 70 % compared to cement without additives.

Table 2. Content and properties of binder composites for foam concrete manufacturing.

ID	Composition, %			Specific surface area m^2/kg	with superplasticizer Muraplast FK 19 (0.1 %)			
	Portland cement	Opoka marl	Fly ash		Normal density of cement paste, %	Setting time, min start/end	Compressive strength, MPa 7 d	Compressive strength, MPa 28 d
1	100	–	–	324	27	150/250	19.9	43.5
2	90	10	–	551	23	15/168	45.3	79.3
3	60	–	40	549	24.5	23/168	41.7	62.2
4	50	10	40	552	23	19/169	40.1	72.3

Opoka marl and fly ash in the BC content direct to an raise in the volume concentration of hydrated new growths as a result the interaction of calcium hydroxide with active additives of the binder composite. The quantitative ratio of hydration products (Fig. 2) can be seen by the intensity of diffraction reflections: calcium hydroxide (d = 4.93; 3.11; 2.63; 1.93; 1.79; 1.69 Å), alite (d = 2.76; 2.19 Å), belite (d = 2.78; 2.74; 2.19 Å), ettringite (d = 9.7; 5.9; 4.92 Å) and calcium hydrosilicates (d = 9.8; 4.9; 3.07; 2.85; 2.80; 2.40; 2.80; 2.00; 1.83 Å) X-ray diffraction patterns showed that in the samples of the binder composite with OM, FA and binary mineral additive (10 % OM + 30 % FA), the reflection intensity of calcium hydroxide decreases by 1.7; 3.3 and 1.6 times, respectively.

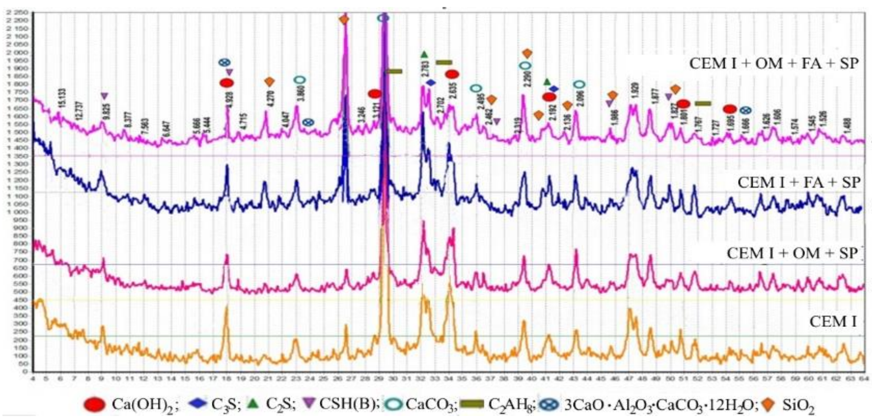

Figure 2. X-ray diffraction pattern of 28-days hardened composite binder with mineral admixtures: 1 – CEM I; 2 – CEM I + OM + SP; 3 – CEM I + FA + SP; 4 – CEM I + OM + FA + SP.

An increased amount of low basic calcium hydrosilicates is also noted. As you know, this has a positive effect on the strength of the cured composite. At the same time, a decrease in the amount of ettringite in all hydrated composites (with opoka marl, fly ash and binary mineral additive) is achieved in comparison with the control composition, this fact is explained by the low basicity of calcium hydroaluminates. Due to the use of superplasticizer in cement and composite compositions with fly ash, it slows down the hydration process in the early stages. In the subsequent stages, for the composite with the opoka marl, as well as the binary FA + OM, the hydration process is accelerated, which leads to an increase in strength compared to non-additive cement and is confirmed by the results of physical and mechanical tests (Table 2). Thus, the structural features of composite binders using the opoka marl and fly ash have been established, which are included in the optimization of the synthesis of new growths due to the multicomponent composition of composite binders. The presence of marl flask in the cementitious composite, which, along with calcite and clay-mixed clay formations, zeolite and opal, as well as fly ash, accelerates the setting process of the foam concrete mixture by the optimal time parameter. The peculiarity of hydration and the influence of mineral components on the properties of the binder composite is confirmed by the dynamics of heat release, expressed by the dependence $dQ/dt = f(t)$ in the initial curing period (up to 1 day), as well as the total amount of heat released, described by the function $Q = f(t)$ for 3 days using a differential calorimeter (Fig. 3).

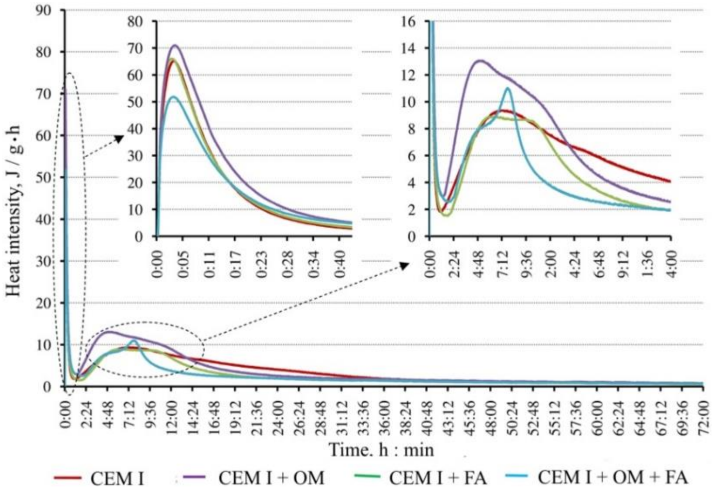

Figure 3. Kinetics of heat release at hydration of binder composites.

This fact can be explained as follows. When opoka marl is added to the cement system, hydration processes intensify during the induction period, which is accompanied by an increase in the completeness of hydration of the clinker minerals. This is due to the manifestation of the pozzolanic reaction and active binding of calcium hydroxide, as well as a higher concentration of accumulated new growths - the calcium hydrosilicates of the second generation. The manufacturing quality of foam concrete is more dependent on three elements: the stability of the foam, obtaining a cellular suspension using cementitious composites, as well as curing the resulting porous mass. High-quality microstructure of foam concrete is achieved by the optimal ratio of time to reach maximum system porosity and setting time. The foam-cement mixture on pure cement, after mixing, foams to a volume of 800 cm^3, after 60 minutes it decreases to 700 cm^3 and remains at this level for 12 hours of storage (Table 3). The foam-cement-ash mass has half the volume of foaming – 400 cm^3, which after 60 minutes of storage drops to 300 cm^3, and by 12 h – up to 280 cm^3. The expansion volume of the cement-marl composition was 820 cm^3, after 60 minutes it dropped to 810 cm^3, and after 12 hours it stopped at around 800 cm^3. Thus, the cement-marl composition showed the best characteristics.

Table 3. Behaviors of binder composites for cellular concrete manufacturing.

Mix ID	Water/cement ratio	Foamer, % wt.	Volume of foam, cm^3			Multiplicity of system, K_s
			immediately after mixing	after		
				60 min	12 h	
CEM I+SP	0.45	0.2	800	700	700	3.5
CEM I+FA+SP			400	300	280	1.4
CEM I+OM+SP			820	810	800	4

Foam-cement mixtures based on the binder composite (cement-marl) are also characterized by optimal rheological characteristics. This makes it possible to optimize such important parameters as the intensity of formation of a porous microstructure, the onset of setting, and the curing of the system. Table 4 lists the selected formulations for monolithic foam concrete. This selection was carried out taking into account the characteristics of the binder composites, such as water requirements, setting and curing rates, rheological characteristics and activity. The test results of the developed compositions of foam concrete with opoka marl showed that the best is the composition on the developed binder composites for joint grinding of cement and opoka marl in a ratio of 90 %: 10 %. The compressive strength of specimens of foam concrete with a density of 703 kg/m^3 was 4.32 MPa, which is 1.8 times greater than that of the reference specimen on pure Portland cement, and significantly higher than that of industrial foam concrete blocks. In general, all synthesized foam concrete with a range of densities of 300–700 kg/m^3 obtained on the basis of BC have a compressive strength higher than control specimens prepared on Portland cement, while providing significant economy of Portland cement.

Table 4. Behaviors of foam concrete on binder composites incorporating opoka marl.

Characteristics	Type of binder used,% by weight of cement								
	Reference specimen (CEM I)			Joint milled 90 % CEM I+10 % OM (489 m^2/kg)			Separately milled 90 % CEM I+10 % OM (484 m^2/kg)		
Composition, kg/m^3									
Portland cement	260	435	610	234	385	540	234	385	540
Opoka marl	–	–	–	26	50	70	26	50	70
Water	200	260	320	200	300	370	200	300	370
Foamer, % by CEM I wt.	1.25	1	1.25	1.25	1	1.25	1.25	1	1.25
Characteristics									
Average density, kg/m^3	309	505	701	308	498	703	305	503	695
Compressive strength, MPa	0.39	1.48	2.42	0.58	2.83	4.32	0.55	2.37	3.27
Thermal conductivity, W/m·ºC	0.08	0.12	0.18	0.065	0.10	0.16	0.07	0.11	0.18
Frost resistance, cycles	20	20	25	25	30	35	20	25	35
Vapor permeability, mg/mhPa	0.29	0.24	0.21	0.27	0.22	0.18	0.28	0.23	0.18
Shrinkage, mm/m	1.33	1.29	1.22	1.04	0.87	0.96	1.12	1.02	0.95
Note	Compared to specimens on the BC, the reference specimens have larger porosity, looser and more heterogeneous microstructure, and greater water separation			water separation is not observed			water separation is not almost observed		

Using the binder composites obtained by co-grinding Portland cement and 10 % opoka marl to a specific surface area of 489 m^2/kg, a wide range of foam concrete was developed for monolithic construction with a wide range of densities of 300–700 kg/m^3. The increase in strength (almost 1.8 times for foam concrete with a density of 700 kg is explained by the positive effect of using a binder composites at all stages of the preparation of the composite, from foaming and porosity to hardening and operation. Given the high demand for foam concrete in low-rise individual construction, dry mixes have been developed for monolithic construction It was found that the compressive strength of foam concrete on binder composites obtained by joint grinding of Portland cement (60 %) and fly ash (40 %) up to a specific surface of 500–550 m^2/kg, increases 1.37 times compared to the reference specimens (Table 5) with high Portland cement economy. The values of thermal conductivity, vapor permeability and shrinkage during drying are in accordance with the standards. In this regard the developed foam concrete compositions can be recommended for the manufacture of blocks with strict observance of technological regulations at the cement-ash binder.

The results presented in Tables 4 and 5 are superior to analogues [2, 6–8] in compressive strength, thermal conductivity and frost resistance.

Table 5. Behaviors of foam concrete for the production of wall blocks.

Characteristics	Type of binder used,% by weight of cement								
	Reference specimen (CEM I)			Joint milled 60 % CEM I+40 % FA (497 m^2/kg)			Joint milled 60 % CEM I+30 FA +10 % OM (484 m^2/kg)		
Composition, kg / m^3									
Portland cement	250	430	600	166	385	540	234	385	540
Fly ash	–	–	–	107	175	250	80	130	190
Opoka marl					50	70	26	50	70
Water	200	260	320	220	300	370	220	300	370
Foamer, % by CEM I wt.	1.2	1	1.2	1.2	1	1.2	1.2	1	1.2
Characteristics									
Average density, kg/m^3	295	503	706	303	493	697	293	494	698
Compressive strength, MPa	0.39	1.48	2.42	0.47	2.26	3.18	0.50	2.62	4.26
Thermal conductivity, W/m·ºC	0.07	0.12	0.17	0.06	0.10	0.15	0.08	0.12	0.16
Frost resistance, cycles	20	20	25	25	30	35	20	25	35
Vapor permeability, mg/mhPa	0.27	0.23	0.22	0.20	0.24	0.20	0.28	0.22	0.18
Shrinkage, mm/m	1.32	1.30	1.21	1.05	0.86	0.97	1.11	1.03	0.94

Improving the durability characteristics of foam concrete on developed binder composites is explained by the microstructure of the materials obtained (Fig. 4). The SEM-images show that the optimization of the microstructure at the macro level is clearly traceable in comparison with the foam obtained on 100 % Portland cement.

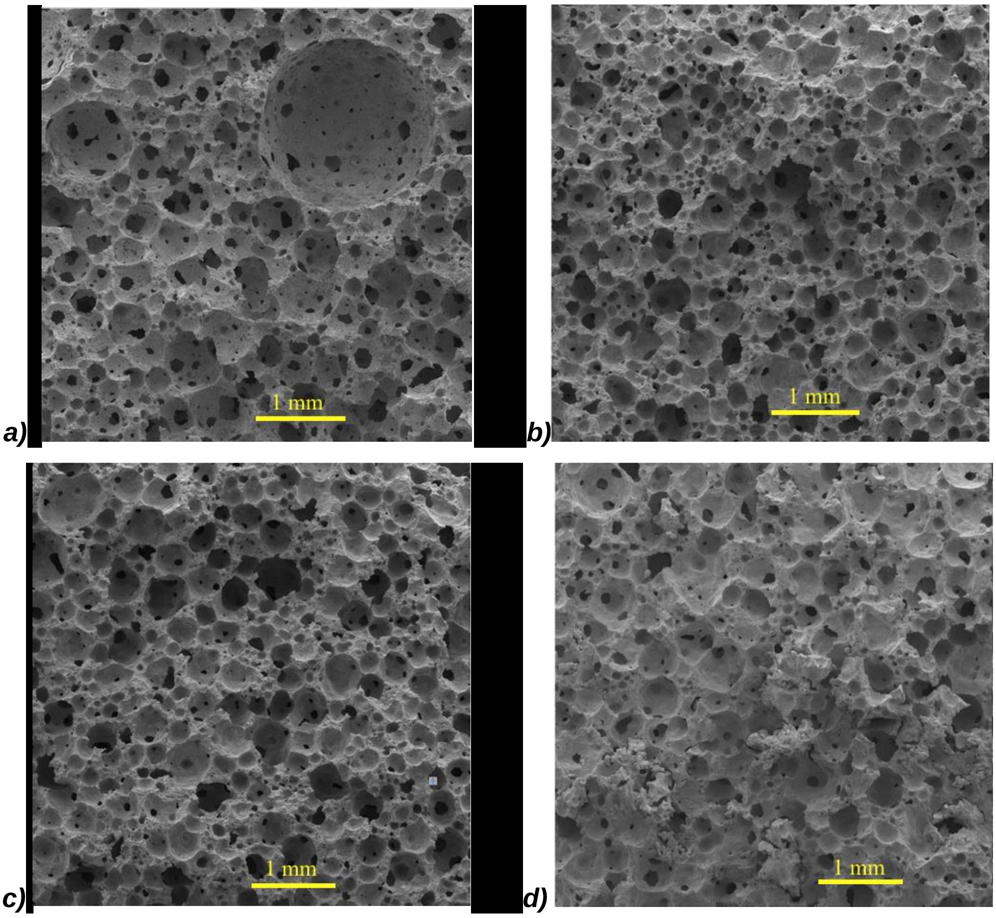

Figure 4. Macrostructure of foam concrete on composites:

***a)* Portland cement; *b)* Portland cement 60 % + fly ash 40 %; *c)* Portland cement 60 % + fly ash 30 % + opoka marl 10 %; *d)* Portland cement 60 % + fly ash 25 % + opoka marl 15 %.**

Here it is necessary to note the important role of optimizing the microstructure of inter-porous partitions, which is formed using the BC, in increasing the physical and mechanical characteristics of foam concrete. The microstructure of the cement paste of the inter-porous foam concrete partitions on the developed BC is more perfect. This is due to the fact that during hydration, the amount of binder water in the curing system is less than in the control compositions due to the presence of opoka marl and fly ash in the BC. In this regard, the liquid phase is more supersaturated with dissolution products, and the conditions for hydration of clinker minerals are improved. It ensures enhance in the number of new growths having a high specific surface area. Enhance in the dispersion of new growths directs to an increase in the number of contacts between new growths and leads to the formation of a denser packing of the microstructure during the formation of thin-layer partitions. This contributes the curing process of the composite. The distraction of part of the water by the components of the opoka marl regulates and improves the plastic and relaxation characteristics, reducing the defectiveness of the inter-porous partitions and reducing their fragility.

Thus, regularities were revealed and substantiated that made it possible to obtain highly efficient foam concrete on the BC using opoka marl for use in monolithic construction with a foam concrete compressive strength of 4.32 MPa at a density of 700 kg/m^3; and for organizing the production of blocks at the plant on a cement-ash binder and using a cement-ash binder with the addition of 10 % opoka marl with a compressive strength of 4.26 MPa with high economy Portland cement.

Modern enterprises are mainly small businesses. Therefore, a promising technology for the production of building materials should include the least possible technical re-equipment of factory facilities. The authors developed technological regulations for the production of heat-insulating and both structural and heat-insulating non-autoclaved foam concrete for monolithic construction based on dry construction mixtures prepared on composite binders using opoka marl. The technology for the manufacture of dry construction mixes from a cement-fly ash composite binder with the addition of marl for blocks from non-autoclaved foam concrete was also improved. Binder composites make it possible to obtain high-quality wall materials directly at the construction site. Moreover, the addition of opoka marl in an amount of 10 % allows you to control the expansion process of the foam concrete mass, and the setting process correlates with the time of maximum porosity of system.

At the final stage of the study, a technological route was developed for the production of non-autoclaved foam concrete, which can be used both for monolithic construction and for the manufacture of blocks (Fig. 5). The technology was based on the possibility and accessibility of material raw materials for small and medium enterprises and the possibility of organizing production at existing production bases of cement and concrete plants.

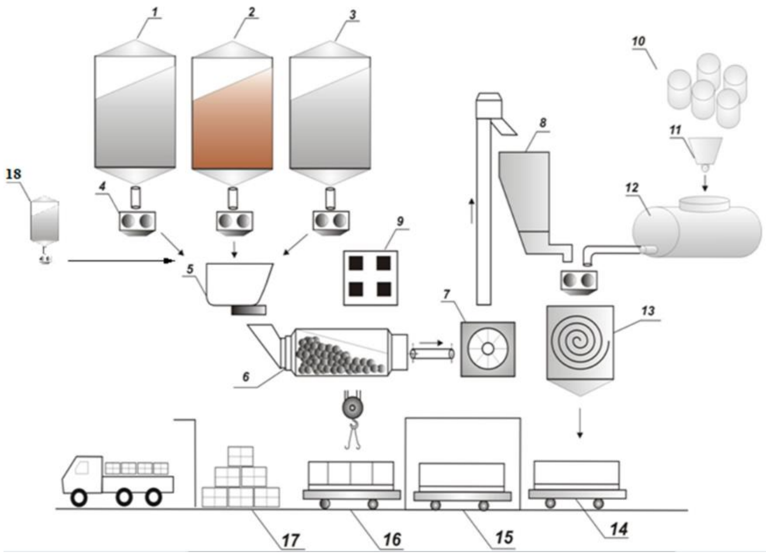

Figure 5. The technological route for non-autoclave foam concrete production:
1 – container for Portland cement; 2 – container for OM; 3 – container for fly ash;
4 – weight batchers; 5 – mill hopper; 6 – vibration mill; 7 – air pump; 8 – container for BC;
9 – control panel; 10 – foamer; 11 – volumetric dispenser; 12 – foam generator;
13 – foam concrete mixer; 14 – molding station; 15 – strength gain station;
16 – stripping station; 17 – packaging and storage; 18 – container for superplasticizer.

Based on the foregoing, the foam concrete compositions on the binder composites were developed and a relationship was established between the microstructure, composition of new growths and the operational characteristics of the composite. The inter-porous foam concrete partitions on the developed BC are nano- and microporous, have a high-density new growth package, consisting mainly of CSH of various basicities. This explains the decrease in the number of microcracks, the increase in the strength of foam concrete in comparison with a composite on a traditional binder with high economy of Portland cement.

4. Conclusions

During the interpretation of the obtained experimental results, the developed binder composites and their influence on the durability characteristics of non-autoclaved foam concrete were studied. As a result, the following valuable findings are established.

1. The theoretical basis of the design and synthesis of foam concrete based on the binder composites are proposed, both for monolithic construction and for the production of blocks. The features of the formation of the microstructure of the binder composites to improve the efficiency of foam concrete, which consist in optimizing the processes of the system "foaming – setting – hardening" due to the polycomponent binder composites, are revealed.

2. The opoka marl in raw mixtures for the production of foam concrete containing, along with calcite and clay minerals, zeolite and opal, accelerates the setting process of the foam concrete mixture, and then, upon curing, amorphous components react with calcium hydroxide, which arises upon hydration of C_3S and C_2S, forming second-generation calcium hydrosilicates. In this case, a dense inter-porous septum forms, which strengthens the final product.

3. The mechanism of the effect of fly ash on the microstructure formation processes of the foam concrete mixture for wall materials is established. The BC obtained by milling components have a compressive strength of up to 60–MPa with Portland cement economy of up to 40 %. Based on the binder composites, the un-autoclaved foam concrete with a compressive strength of up to 4.26 MPa at density of 500–700 kg/m^3 was obtained. Fly ash contributes to a valuable change in the microstructure formation, which practically directs to

the absence of $Ca(OH)_2$ among the new growths, as compared to reference specimens, due to the interaction of active SiO_2 contained in part of the fly ash with $Ca(OH)_2$, which releases alite during hydration process.

4. The economic efficiency of the production and use of foam concrete based on Portland cement, opoka marl and fly ash, which occurs due to the use of new types of raw materials, has been proved. The expansion of the raw material base for the production of foam concrete at the same time helps to reduce material costs compared to traditionally used raw materials. The technology for the production of binder composites is developed for large-scale production of foam concrete based on binder composites.

5. Prospects for further development of the issue

It makes practical sense to consider transdisciplinary approaches to solving urgent problems of building materials science, to develop foam concrete production technologies for a wide range of building composites, including for the development of the northern regions. The technique described in the work can be used in the development of binders to expand the range of foam concrete production, including to improve a comfortable human environment in the architectural and construction design of composites with various specified operational characteristics.

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Contacts:

Valeriy Lesovik, naukavs@mail.ru

Evgeniy Glagolev, mail@belgorodstroy.ru

Vasily Voronov, fedyu.rs@dvfu.ru

Lilia Zagorodnyuk, lhz47@mail.ru

Roman Fediuk, roman44@yandex.ru

Andrey Baranov, de_montgomery@mail.ru

Arbi Alaskhanov, alaskhanov.arbi@mail.ru

Alexander Svintsov, svintsovap@rambler.ru