2023; 15 (5): 408–417



Original article



Controlling the structural formation of porized cement composites in the production of thermally efficient enclosure structures of enhanced quality

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ABSTACT: Introduction. Research to improve the quality of foam concrete products through targeted control of technological processes of structure formation of cement-based compositions is relevant. The strength of foam concrete is significantly influenced by the adhesion strength of the hardened cement paste to the aggregate. This article discusses methods of chemical and mechano-chemical activation of foam concrete filler with glyoxal-containing additives, interaction with hydration products of binders and increasing the strength of the contact zone. The quality of foam concrete can be improved by controlling the properties of the contact zone. **Materials and methods.** The study was conducted using standard test procedures specified in national standards. **Results.** With preliminary chemical and mechano-chemical exposure of the sand surface to glyoxal-containing additives and its subsequent introduction into the foam concrete mixture the grade of strength of foam concrete increases to B1 while maintaining the average density grade D500 in comparison with foam concrete of the control composition, while the shrinkage value decreases by 20–38.5% and thermal conductivity coefficient by 37%. **Conclusion.** The use of glyoxal-based additives in foam concrete mixtures by pre-activating the sand surface can improve the quality of cement foam concrete.

KEYWORDS: cement-based foam concrete, glyoxal-containing additives, structure formation, plastic shrinkage, porosity, compressive strength, average density, thermal conductivity coefficient.

ACKNOWLEDGMENTS: The research was carried out by the research teams of the companies and strategic partners, that are members of the Construction and Architecture Industrial Consortium, with the financial support from MGSU within the framework of the 2023 competition for fundamental and applied research (R&D) with a view to the implementation of the MGSU Development Program for 2021-2030 within the framework of the federal academic leadership program Priority 2030.

FOR CITATION: Steshenko A.B., Kudyakov A.I., Inozemtcev A.S., Inozemtcev S.S. Controlling the structural formation of porized cement composites in the production of thermally efficient enclosure structures of enhanced quality. *Nanotechnologies in Construction*. 2023; 15 (5): 408–417. https://doi.org/10.15828/2075-8545-2023-15-5-408-417. – EDN: YNCWWI.

INTRODUCTION

In accordance with the concept for the development of the Russian construction industry until 2030, important tasks are the effective resource provision of housing facilities, including individual construction, with the maximum use of local raw materials, as well as reducing energy costs in the production of building materials and building maintenance. The optimal solution to the tasks set is to increase the thermal properties of enclosing structures, reduce the cost of construction, provide a comfortable and safe environment for living by using high-quality heat-insulating foam concrete for the construction of buildings. Heat-insulating foam concrete in wall structures provides good thermal protection of housing, the required load-bearing capacity and durability [1].

When producing non-autoclaved foam concrete with low average density, effective in terms of thermophysical parameters, problems arise in controlling the processes of ensuring a stable cellular structure, high strength, reducing drying shrinkage and thermal conductivity [2, 3]. It is necessary to scientifically substantiate and develop innovative ways to control structure formation in order to improve its quality for the widespread use of non-auto-



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claved foam concrete in the production of products and in monolithic construction [4].

Improving the physical and mechanical properties of foam concrete is achieved through the improvement or development of new technological methods that make it possible to regulate volumetric structure-forming processes at different scale levels, namely: at the micro level – the cement matrix; at the contact of interacting components, cement stone – aggregate; at the macro level – a durable frame made of fine aggregate (sand) [5].

Al components of foam concrete, regardless of the state of aggregation, participate in mechanical and physicochemical interaction during structure formation. The chemical activity of the surface of the grains increases and the process of interaction with the cement matrix at the phase interface is accelerated with controlled pre-activation of the filler. Chemical or mechanochemical activation of the foam concrete mixture filler using a structural-phenomenological analysis of the relationship between microstructural indicators and physical and mechanical properties allows us to predict the improvement of the main technological and technical parameters of the quality of foam concrete.

It has been established that treating the aggregate with aqueous solutions of acids leads to an increase in the chemical activity of the surface, the interaction of hydrate compounds of hardened cement paste with aggregate grains, and an increase in the microhardness of the contact zone and the strength of foam concrete [6, 7].

Currently, the issue of using modifying additives that affect the rheological properties of the cement matrix and the structure formation of the foam concrete mixture has been studied quite well [8–12]. Using modern modifying additives, they control the workability and setting time of the foam concrete mixture, reduce shrinkage, increase the homogeneity, and the strength of foam concrete up to 25% [9,13], reduce cement consumption by 10-15% [14-16]. The surface of the aggregate should be pre-activated to obtain the greatest effect from the action of modifying additives. The adhesion strength of the cement stone to the aggregate has a significant influence on the strength of foam concrete. [17]. The contact zone is the most important structural element of foam concrete, by controlling which it is possible to ensure the required quality parameters of the designed concrete.

In this work it is proposed to use crystalline glyoxal and a 40% aqueous solution of glyoxal for chemical and mechanochemical activation of the surface of quartzfeldspar sand. In the alkaline environment of hydrating cement, the glyoxal additive interacts with calcium hydroxide, which is formed as a result of the hydrolysis of alite, a cement mineral, with the formation of calcium glycolate (HO-CH₂-COO)₂Ca, increasing the volume of neoplasms and the foam concrete matrix [18–21]. Calcium glycolate is adsorbed on needle-shaped ettringite crystals, reducing the volume of pore space in the hardened cement paste and increasing strength [22, 23]. Based on this, it is assumed that by pre-activating the surface of aggregate grains with glyoxal additives and introducing them into the foam concrete mixture, it is possible to reduce porosity and increase the strength of hardened cement paste in contact areas with aggregate grains, which will improve the physical and mechanical parameters of the quality of foam concrete.

The purpose of the study is to establish the patterns of chemical and mechanochemical activation of filler with glyoxal additives on the structure formation and properties of cement foam concrete.

MARERIALS AND METHODS

The materials presented in table 1 were used carrying out the research.

Technical characteristics of glyoxal additives are presented in [1].

Table 1

Raw ma	terials	for	foam	concrete	mixture
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Material	Compliance with the requirements of the regulatory document
Portland cement CEM I 42.5N from Topkinsky plant	Russian State Standards GOST 31108-2020
Quartz-feldspathic sand from the Kudrovskoye deposit in the Tomsk region with a fineness modulus of 1.8	Russian State Standards GOST 8736-2014
Foaming agent PB-2000	Technical Specifications 2481-185-05744685-01; Russian State Standards GOST 24211-2008
Tap water	Russian State Standards GOST 23732-2011
40% aqueous solution of glyoxal (TH «Novokhim», Tomsk)	Technical Specifications 2633-003-67017122-2011
Glyoxal crystalline (TH «Novokhim», Tomsk)	Technical Specifications 2633-004-67017122-2011



Table 2

The basic composition of foam concrete for $1 \ m^3$

	Consumption of raw materials per 1 m ³					
	cement, kg	quartz-feldspathic sand, kg	tap water, l	foaming agent, l		
Basic composition D500	270	135	202	1.3		

The physical and mechanical properties of foam concrete were determined in accordance with the requirements of national standards:

- testing and quality assessment of foam concrete (Russian State Standards GOST 25485–2019);
- average density (Russian State Standards GOST 12730.1-2020);
- compressive strength and quality assessment of a series of 6 samples (Russian State Standards GOST 10180-2012 and GOST 18105-2018);
- thermal conductivity using the ITS-1 thermal conductivity meter (Russian State Standards GOST 7076-99). The determination of plastic shrinkage was carried

out according to the author's method presented in [8].

The structure of pore volume of foam concretes was investigated using Quanta 200 3D Two-beam Scanning Electron Microscope. The images were obtained within the low vacuum mode at the stable accelerating voltage up to 20kV. In order to study porous structure of foam concrete mercury injection method was applied using Quantachrome 33 Porosimeter.

The basic composition of the foam concrete mixture was developed taking into account the requirements of Russian Standard SN 277-80 "Instructions for the production of cellular concrete products" (table 2).

The foam concrete mixture was prepared according to one-stage technology using a laboratory foam concrete mixer.

A block diagram of studies of improved quality thermal insulating cement based foam concrete modified with glyoxal additives was developed using the results of previous studies (fig. 1).

Scientific substantiation and choice of modifying additive

A positive effect was established when controlling the water demand, the kinetics of early structure formation and hardening of cement paste with a glyoxal additive according to the results of previous studies [1, 19, 23, 24]. That was the basis for choosing the additive to achieve this goal.

Establishment of the method of introduction and the optimal content of glyoxal additives in the foam concrete mixture

As a result of the analysis of various methods of preparation (activation) and the sequence of introducing components in the process of preparing a foam concrete mix-



Fig. 1. Block diagram of foam concrete research

ture, taking into account the interaction of hardened cement paste with aggregate, it is accepted that the methods of separate or joint mechanical and chemical activation of the surface of the components are promising.

Two technological methods for the preparation and introduction of glyoxal-containing additives into the foam concrete mixture were scientifically substantiated to conduct experimental studies to establish the patterns of structure formation during the activation of the components:

- preliminary application of a 40% aqueous solution of glyoxal to the surface of the filler by spraying. Quartz sand was treated with a 40% aqueous solution of glyoxal by spraying with a spray gun and then introduced into the concrete mixer;
- dry quartz-feldspathic sand was ground in a mill together with crystalline glyoxal. Activation of the sand surface is carried out due to simultaneous compressive and shear deformation (abrasion), as well as the chemical interaction of the glyoxal additive with the surface of the filler.

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RESULTS AND DISCUSSION

A foam concrete mixture with a mobility of 11-14 cm (the diameter of the flow on the Suttard device) and a W/T ratio equal to 0.5 was used during the research [8, 9, 25].

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The results of the study of the structure of foam concrete, dried to a constant weight, with activated and nonactivated filler are shown in fig. 2.

A perforation and intensive destruction of the pore walls, as well as their association, are observed in the basic sample. It contributes to the development of the sedimentation process, a decrease in the homogeneity of the microstructure. It was found that foam concrete with chemical and mechanochemical activation has a more uniform structure, the pores evenly fill the entire volume of the material the, contact area of the cement matrix increases. Preliminary activation of sand contributes to the formation of a stable structure of foam concrete with closed fine pores.

The results of studying the structure of foam concrete with an activated sand surface are given in table 3.

According to the research results, it was established that the mechanochemical activation of sand by grinding with crystalline glyoxal leads to decrease the average pore diameter of foam concrete from 45.8 to 36.9 microns, the standard deviation of the average diameter from 23.6 to 12.1, and to increase the content of conditionally closed pores of foam concrete from 25.6% to 35.8% compared to samples without additives. It was also established that an increase in the polydisperse pore size distribution corresponds to a decrease in the quality of foam concrete.

The results of studies to determine the values of compressive strength and average density of foam concrete



Fig. 2. The structure of cement based foam concrete. On the left – zoom x250, on the right – zoom x2000: a) basic (reference) b) grinding sand with crystalline glyoxal 0.01%

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Fig. 2. The structure of cement based foam concrete. On the left -zoom x250, on the right -zoom x2000: c) sand treatment with 40% glyoxal solution d) grinding sand with crystalline glyoxal 0.05%

Table 3Porosity of foam concrete samples

Foam concrete with activated sand	Average density, kg/m ³	Average pores diameter, µm	δ of pores diameter	General porosity, %	The volume of open capillary pores, %	Porosity of conventionally closed pores, %	Microporosity index
Basic (reference) D500	470	45.8	23.6	79.57	21.5	25.57	0.04
Sand treatment with 40% glyoxal solution	480	40.1	22.5	77.89	15.6	25.17	0.03
Grinding sand with crystalline glyoxal 0.01%	480	36.9	12.1	6.99	10.1	35.80	0.03
Grinding sand with crystalline glyoxal 0.05%	487	39.2	18.9	77.06	13.7	28.80	0.03





Fig. 3. Influence of the method of sand activation and the type of glyoxal additive on the strength characteristics of foam concrete

samples with sand activated with glyoxal are shown in fig. 3.

It has been established that mechanochemical activation of sand with crystalline glyoxal leads to an increase in the compressive strength of foam concrete at 28 days of age by 66% and with chemical activation of sand by spraying a 40% aqueous solution of glyoxal – by 42%. An increase in the strength of thermal insulating foam concrete is observed as a result of the absorption of glyxalcontaining additives on the surface of the finest particles of cement and hydrate formations [7, 21, 25]. Therefore, the hardened cement paste of the contact zone and the near-pore space of foam concrete has higher strength characteristics than the hardened cement paste in the volume of foam concrete.

Thus, the mechanochemical activation of sand with crystalline glyoxal in an amount of 0.01% by weight of cement leads to increase the compressive strength of D500 foam concrete o 1.5 MPa. The positive effect of activation of sand with an additive is explained by the fact that

activated quartz-feldspathic sand with a higher specific surface area over time more intensively realizes its reaction activity than unground sand [5, 17].

Data on the uniformity of quality parameters for average density and compressive strength of foam concrete are presented in tables 4 and 5.

In foam concrete with mechanochemically activated sand with an optimal content of glyoxal additive (0.01%) the coefficient of variation of the average density of foam concrete decreased from 3.4 to 0.54%, and the coefficient of variation of compressive strength from 10.5 to 7.1%. Grinding sand with glyoxal and treating the sand with a 40% glyoxal solution lead to increase the foam concrete strength grade to B1 while maintaining the density grade D500. In accordance with experimental data, foam concrete with an average density of 470 to 487 kg/m³ was obtained, which corresponds to the D500 grade.

The studies of the plastic shrinkage of the foam concrete mixture, as well as the thermal conductivity of foam concrete, were carried out on the effect of chemical

Type of foam concrete	Average value, kg/m ³	Concrete grade by average density	Standard deviation	The coefficient of variation, %
Basic (reference) D500	470	D500	9.9	3.40
Sand treatment with 40% glyoxal solution	480	D500	3.3	0.67
Grinding sand with crystalline glyoxal 0.01%	480	D500	2.6	0.54
Grinding sand with crystalline glyoxal 0.05%	487	D500	4.6	1.30

Table 4

Statistical	data o	n tho	avorado	donsity	of foam	concrete
Statistical	uata u	m me	average	uensity	of toalli	concrete



Table 5

Statistical data on the compressive strength of foam concrete

Type of foam concrete	Average value, MPa	Compressive strength grade	Standard deviation	The coefficient of variation, %
Basic (reference) D500	0.9	B0.5	0.055	10.5
Sand treatment with 40% glyoxal solution	1.28	B1	0.120	9.2
Grinding sand with crystalline glyoxal 0.01%	1.5	B1	0.048	7.1
Grinding sand with crystalline glyoxal 0.05%	1.0	B1	0.056	7.6

and mechanochemical activation of sand with glyoxal additive.

At an early age, when the mixture is still plastic, evaporation of free water from the surface of concrete and plastic shrinkage are the main factors of crack formation. Plastic shrinkage measurements were carried out over 3 hours.

The plastic shrinkage of the foam concrete mixture is reduced by 61% compared to the control sample (fig. 4) with the combined mechanochemical activation of sand with crystalline glyoxal in an amount of 0.01% by weight of cement. The reason for the higher shrinkage of the samples is the perforation of the walls and increased heterogeneity of the pores. A significant reduction in shrinkage deformations during natural hardening of foam concrete makes it possible to predict a reduced level of stress formation during structure formation in the volume of foam concrete and, as a result, increased operational reliability of products made from this material [2, 16, 25].

The thermal insulation properties of foam concrete are affected by the total volume, size and uniformity of pore distribution in the foam concrete. According to the results of studies of the structure and average density, foam concrete with sand activated by glyoxal additives has a more uniform structure. According to the test results given in table 6, the values of the thermal conductivity coefficient change in accordance with the grade of average density of







Table 6

The values of the coefficient of thermal conductivity of foam concrete samples

Type of fear concrete	Thermal conductivity coefficient, W/m • °C			
Type of loan concrete	at a humidity of 25%	in dry condition		
Basic (reference) D500	0.20	0.100		
Sand treatment with 40% glyoxal solution	0.17	0.081		
Grinding sand with crystalline glyoxal 0.05%	0.17	0.070		
Grinding sand with crystalline glyoxal 0.01%	0.16	0.063		

foam concrete, which is explained for the foam concrete matrix by the homogeneity of the porous structure.

As follows from the table 6 that the lowest thermal conductivity coefficient (0.063 W/m \cdot °C) has foam concrete on sand activated by joint grinding of sand with crystalline glyoxal in an amount of 0.01% by weight of cement.

Preliminary activation of the sand surface with glyoxal additives makes it possible to produce cement foam concrete with improved quality parameters.

CONCLUSION

1. Electron microscopic studies have established that the average pore diameter in foam concrete with the preliminary activation of the surface of sand with crystalline glyoxal decreases from 45.8 to $36.9 \,\mu\text{m}$, the standard deviation of the average diameter decreases from 23.6 to 12.1, and the content of conditionally closed pores increases from 25.6% to 35.8% compared to samples without additives.

2. The shrinkage of the foam concrete with a preliminary physical and chemical effect of additives on the surface of the filler and its subsequent introduction into the foam concrete mixture decreases by 30-61%.

3. The coefficient of variation of the average density of foam concrete decreases from 3.4 to 0.6% and the compressive strength of foam concrete from 10.5 to 7.1% in foam concrete with glyoxal additives, as a result the grade of strength of foam concrete increased to B1 while maintaining the grade of average density D500.

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Aleksei B. Steshenko – scientific guidance; carrying out experimental work; collection and systematization of experimental data; graphical and tabular presentation of research results; writing the original text of the article; drawing up final conclusions. Aleksander I. Kudyakov – concept development and research methodology development; analysis of research results; writing the original text of the article; drawing up final conclusions.

Aleksandr S. Inozemtcev – literature review, processing and analysis of experimental data using machine learning methods. **Sergei S. Inozemtcev** – literature review, processing and analysis of experimental data using machine learning methods.

The authors declare no conflicts of interests.

The article was submitted 10.09.2023; approved after reviewing 04.10.2023; accepted for publication 06.10.2023.