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Structural and heat-insulating cement-based concrete with complex glyoxal based additive

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ABSTRACT: Introduction. The article presents the results of studies of the effect of complex additive based on glyoxal on the properties of cement-based foam concrete mix and foam concrete of natural hardening. The relevance of the study is determined by the necessity to provide the required process parameters of mixture for transportation and laying the formwork, as well as providing strength and thermal and physical characteristics of wall structures for the development of the northern regions of Russia, including the Arctic zone. It has been proposed to decrease the shrinkage deformations of the concrete mix and increase compressive strength of hardened foam concrete by affecting the cement matrix with complex modifier based on glyoxal. Materials and methods. The effect of modifying additives on the properties of the foam concrete mixture and foam concrete was studied at a W/S mixture ratio of 0.45. Research has been carried out using test methods set out in national standards. The results of the study of the effect of complex modifying additives (CMA) based on an aqueous solution of glyoxal and organic acids on the rheological and strength properties of foam concrete are presented, the regularities of the processes and the mechanism of structure formation of the modified foam concrete are determined. Results. The use of modifying additives leads to increase result in increasing the aggregative stability and reducing the plastic shrinkage of the foam concrete mix by 22–70%. In foam concrete with the complex additive LA 0.5% + GI 0.55% the compressive strength rises from 1.96 to 2.43 MPa at the age of 28 days while maintaining the average density of D600. The thermal conductivity coefficient of foam concrete modified with various additives decreased by 5-30% compared to references. Conclusions. The obtained results of the study create in the construction industry the basis for the import substitution of modifying additives on the domestic mineral resource base and the production of effective structural and heatinsulating concretes for the development of the northern regions of Russia.

KEYWORDS: glyoxal, structure formation, modifying additives, foam concrete, porous structure, plastic shrinkage, compressive strength, average density, thermal conductivity.

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INTRODUCTION

The development of the northern regions of Russia, including the Arctic zone, has a state development priority and is increasingly relevant in the domestic economy [1]. One of the key points of growth is the construction industry. Providing resources for the construction of modern buildings and structures, especially in the northern regions of Russia, special attention must be paid to the quality of building materials and, above all, products based on cement compositions [2]. Taking into account the specifics of these territories, the demanded result for the production of building materials on a cement binder is a composite with specified performance properties. Therefore, research on the development of scientific approaches to control structure formation in the technology

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of manufacturing modern multifunctional concretes with high performance indicators, which include structural and heat-insulating concretes, is one of the promising areas [3].

2022; 14 (5):

353-362

Foam concrete produced at present does not always meet the increased requirements in the design of building envelopes and modern building technologies [4]. As a rule an increase in the bearing capacity of enclosing structures is achieved by increasing the strength and average density of foam concrete, which simultaneously leads to an increase in the thermal conductivity coefficient and a decrease in the thermal protection of the building [3].

The structure of foam concrete is represented by macropores formed by foam (volume 50-92%, size $0.25\div10^{-4}$ cm), and interpore partitions, consisting of hydration products and non-hydrated cement, silica component, capillary ($10^{-6}\div10^{-4}$ cm) gel pores. The strength of the interpore partitions increases with a decrease in the number of capillary pores in it and the thermal conductivity and the average density grade of foam concrete decrease with an increase in the content of macropores [5]. It is necessary to manage the processes of structure formation of foam concrete, taking into account the factors affecting its pore structure:

- degree of deformation of spherical pores and their dimensions (pore shape);
- the character of the inner surface of the pores. The strength of foam concrete increases with an increase of pores with a dense and smooth inner surface;
- closed porous structure. A decrease in water absorption and high frost resistance of foam concrete are provided with an increase in the content of closed pores with a hydrophobized inner surface;
- uniform distribution of pores in the volume of the material, which lead to increase the uniformity of quality parameters (strength and thermal conductivity) of foam concrete;
- thickness and density of interpore partitions. This indicator depends on the water-solid ratio, the composition of the solid components, as well as the rheological characteristics of the mixture. Initially, part of the water is adsorbed at the air-solution interface, and most of it is located in a structured interpore wall. Part of the water is used to hydrate the cement, and the rest is removed during drying, forming a network of capillary pores of various sizes, directions and closures [6].

One of the effective ways to control the porous structure and strength of the interpore partitions of foam concrete is the introduction of modifying additives into the mixture during mixing, which reduce the water content while maintaining the necessary mobility of the concrete mixture and increase the strength of the cement matrix in the interpore partitions [5, 7–10]. Developing the composition of foam concrete, it must be taken into account that one part of the water is spent on foam formation, and the other part is used for cement hydration. The use of such additives, both mineral and chemical (synthetic) nature, is one of the tools that provides the ability to control the processes of structure formation at the recipe level. At the same time, a significant role is given to the formation of the structure of hardened cement paste at the micro level [11], because it predetermines the potential for the development of complex modifying additives that implement versatile mechanisms for controlling structure formation [12, 13]. Mainly imported additives are used in the domestic construction industry. Therefore, the development and use of import-substituting modifying additives is relevant. This becomes possible with the development of chemical industry technologies.

In the Tomsk region (Russia) has been developed a technology for the synthesis of oxalic acid dialdehyde – glyoxal, which has a number of unique properties, which makes it very popular in various industries (pharmaceuticals, oil refining, textile, leather, paint and varnish, adhesive, metallurgical industries, etc.). Based on the existing experience of using glyoxal in the construction industry [14–17] it is relevant to review studies on the use of glyoxal-containing additives in building materials manufacturing technologies in order to develop promising areas for further study and creation of new materials with using Russian-made glyoxal.

The creation of an effective complex concrete mix modifier based on glyoxal is possible by combining glyoxal and organic compounds suitable for it according to the "retarder-accelerator" principle [18, 19]. The available scientific groundwork shows the possibility of forming a denser and more durable structure of hardened cement paste in the composition of concrete due to the combined control of structure formation, which consists in the simultaneous control of both the number of crystallization centers and their growth [11, 13, 14]. This is realized by a combination in the composition of the modifying additive of components that perform the function of an accelerator and retarder of the hardening of the binder, respectively [19]. That is, the implementation of complementary crystallization processes is ensured, which optimize the process of structure formation and composite strength gain [20].

The authors propose to use organic bifunctional acids, their salts and oligomers in the form of a combined additive based on glyoxal and a selective effect on the processes of foam concrete structure formation [21].

The aim of this work is to study the effect of a complex modifier based on glyoxal on the processes of structure formation, the features of changes in the technological and mechanical properties of foam concrete. Achieving this goal will make it possible to establish the optimal formulation solutions for obtaining the additive and the concentration of its use to obtain structural-heat-insulating concretes with improved performance properties.



METHODS AND MARERIALS

The raw materials used in the experimental studies were Portland cement without additives CEM I 42.5H of Topki Cement Factory (Russian State Standard GOST 30515-2013), fine aggregate, foaming agents, complex modifying additives and tap water (Russian State Standard GOST 23732-2011).

Characteristics of Portland cement are given in Table 1.

Quartz-feldspar sand of the Kudrovskoe deposit of the Tomsk region (Russia) with a fineness modulus of 1.8 (Russian State Standards GOST 8736-2014 and GOST 26633-2012) was used as a fine aggregate for the manufacture of porous structural and heat-insulating concretes.

The following materials were used as foaming agents: foaming agent PBS and BLAIZER PB (Russian State Standards GOST 24211-2008). Technical characteristics of foam agents are presented in Table 2.

We used a 40% aqueous solution of glyoxal (oxalaldehyde, ethandial) manufactured by Novokhim Trade House, Tomsk (Russia). It obtained by oxidative dehydrogenation of ethylene glycol. It is presented in the form of glyoxal hydrate in aqueous solution. Chemical formula: $C_2H_2O_2$. Crystalline glyoxal is made from a 40% aqueous solution of glyoxal. It belongs to the third hazard class (moderately dangerous) (Russian State Standards GOST 12.1.007-76). Technical characteristics of glyoxalcontaining additives are presented in Table 3.

Two series of complex modifying additives were used in the work. The basis of which is a 40% solution of gly-

Table 3Technical properties of glyoxal

Parameter	Glyoxal
Mass fraction of glyoxal	40±1
Density (20°C), g/cm ³	1.20-1.35
Molecular weight (in amu)	58.04
Melting point, °C	15
Boiling point, °C	51
рН	2-3.5

oxal in an amount of 0.01% by weight of the binder in terms of dry matter, hydroxy acids act as the second component: lactic acid and its oligomer – polylactic. It has been established that the preliminary preparation of the glyoxal solution makes it possible to convert the glyoxal molecule into the aldehyde form, which increases the efficiency of the modifier by 10% [22–27].

Lactic acid (LA) is an aqueous solution of 80% concentration in the form of a transparent, yellowish liquid, with the smell of whey, "Purac" (Amsterdam, The Netherlands).

Glycolic acid (GA) is a clear yellow liquid. Aqueous solution 67%, "Purac" (Amsterdam, The Netherlands).

Oligomers were obtained from lactic and glycolic acids obtained by polycondensation in the laboratory of polymers and composite materials of National Research Tomsk State University [28, 29].

Table 1Cement characteristics

Bulk density, kg/m³Residue on the sieve 0.08, %Normal density, %			Setting ti	me, h-min	Tensile stre at the age	U , ,
	start of setting	end of setting	bending	compression		
1010	2.5	26.4	2-20	3-20	6.3	50.4

Table 2

Technical characteristics of foaming agents

Parameter	PBS	BLAIZER PB
Appearance	clear to brown liquid	yellowish liquid
Foam density at, g/cm ³	1.020 - 1.120	1.080 ± 0.004
Foam concentrate consumption per 1 m ³ of foam concrete, 1	0.25 - 0.9	0.1 - 1.5
pH	7.0 - 11.0	7.0 - 10.5
Foam ratio of 4% foam concentrate solution, not less than	7	7
Foam stability, s, not less	250	250
Warranty period of storage	1 year	1 year

2022; 14 (5): 353–362



CONSTRUCTION MATERIAL SCIENCE

The determination of the physical and mechanical properties of the obtained cement based compositions were carried out using standard methods for testing building materials in accordance with national standards. It includes the determination of compressive strength (Russian State Standard GOST 10180-2012), water absorption (Russian State Standards GOST 12730.3-2020), thermal conductivity coefficient (Russian State Standards GOST 12730.3-2020), thermal conductivity coefficient (Russian State Standards GOST 12730.3-2020), thermal conductivity coefficient (Russian State Standards GOST 7076-99), average density (Russian State Standard GOST 12730.1-78), and frost resistance (Russian State Standard GOST 25485-2019). Original methods were used to determine the plastic shrinkage of foam concrete [5, 30].

The design of the composition of the foam concrete mixture was carried out taking into account the requirements of Russian Standard SN 277-80 "Instructions for the production of cellular concrete products".

The basic compositions of foam concrete (Table 4) were designed taking into account the results of optimizing the technological regimes for the preparation of foam concrete in a laboratory mixer [5, 9, 13].

The study was conducted with the following additives:

- 40% solution of glyoxal (Gl) in the amount of 0.55% by weight of cement;
- lactic acid (LA) with various dosages (from 0.25 to 1% by weight of cement);
- polylactic acid (PLA) with dosages of 0.5, 1, 1.5% by weight of cement;
- complex modifying additive Gl 0.01+LA, concentration 0.01% by weight of cement with a ratio of glyoxal and lactic acid 1:50.

Due to establish the regularities of the influence of additives on the properties of foam concrete mixture and foam concrete, additives were introduced into the basic composition of foam concrete without changing the number of components, followed by adjustment of the recommended composition according to the values of the actual average density of the foam concrete mixture. The preparation of the foam concrete mixture was carried out according to a one-stage technology using a laboratory foam concrete mixer.

For preparing a foam concrete mixture with additives firstly cement, sand and part of water are mixed. Then, an aqueous solution of the modifying additive was introduced into the mixer in the required amount. The mixing process continues until a homogeneous plastic mass is obtained. Then, a foaming agent diluted in water was introduced into the resulting mixture, and all components were mixed for another 4 minutes.

The finished foam concrete mix was placed in metal molds $15 \times 15 \times 15$ cm. A series of samples was made from 6 pieces of each composition for testing. Molded foam concrete specimens were kept under normal conditions at a temperature of $20\pm 2^{\circ}$ C for a day and then stored for 27 days in a normal curing chamber at a temperature of $20\pm 2^{\circ}$ C and a relative humidity of at least 90% before testing. Testing and quality assessment of foam concrete was carried out according to Russian State Standards GOST 25485-2019. The diameter of the mixture flow was determined on the Suttard device for each mixture, as also the actual density of the foam concrete mixture was.

RESULTS AND DISCUSSION

The studies were carried out on a foam concrete mixture with a flowability of 12-15 cm (diameter on the Suttard's Viscosity Analyzer). The effect of modifying additives on the properties of the foam concrete mixture and foam concrete was studied at a W/S mixture ratio of 0.45. The research results are presented in Figures 1-3 and in Table 5.

In a foam concrete mixture with modifying additives, a certain amount of water is released (water-reducing effect) and the foaming ability of the mixture increases [6]. As a result, the water content in the structure of interpore partitions decreases, which leads to a decrease in the number of capillary pores and an increase in the strength of foam concrete [9].

A certain amount of water is released (water-reducing effect) in a foam concrete mixture with modifying additives and also the foaming ability of the mixture increases. As a result, the water content in the structure of interpore partitions decreases, which leads to a decrease in the number of capillary pores and an increase in the strength of foam concrete [6].

The most expressed plasticizing effect is observed when the PLA additive is introduced in an amount of

Table 4

The basic composition of foam concrete, for 1 m³

Decis composition	Component consumption			
Basic composition	cement, kg	sand, kg	water, l	foaming agent, l
D600	330	165	222.75	1.2
D700	380	180	252	1.1
D800	400	200	270	1

Nanotechnologies in construction Нанотехнологии в строительстве 2022; 14 (5): 353–362



CONSTRUCTION MATERIAL SCIENCE

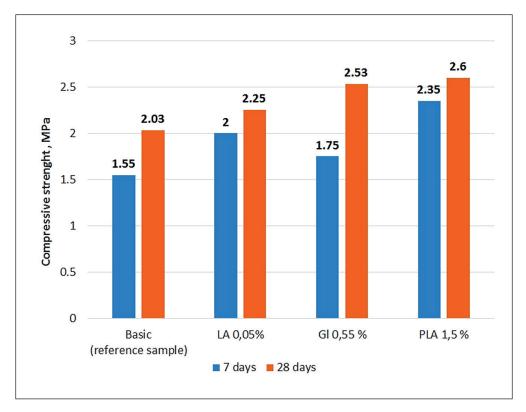


Fig. 1. Influence of modifying additives on the compressive strength of foam concrete by average density $\mathbf{D700}$

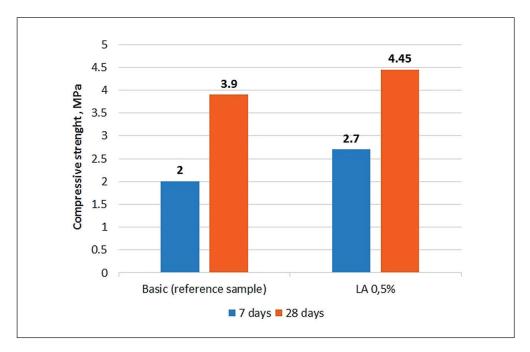


Fig. 2.Influence of lactic acid on the compressive strength of foam concrete by average density ${\rm D800}$

2022; 14 (5): 353–362



CONSTRUCTION MATERIAL SCIENCE

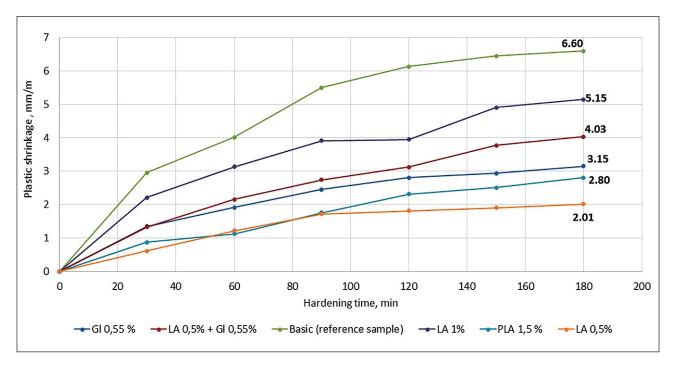


Fig. 3. Influence of modifying additives based on glyoxal on plastic shrinkage of foam concrete mix

Table 5
Influence of modifying additives on the compressive strength of foam concrete by average density D600

Type of fibers	Mixture flowability, cm	Average density of foam concrete, kg/m ³	Compressive strength of foam concrete at the age of 7 days, MPa	Compressive strength of foam concrete at the age of 28 days, MPa
Basic (reference)	12.5	610	1.35	1.96
PLA 1.5%	15	594	1.6	1.7
PLA 0.5%	13.5	577	1.35	1.75
PLA 1%	13	609	1.7	2.2
Gl 0.55%	12.75	600	1.45	2.1
LA 0.25%	13	591	1.35	2.15
LA 0.5%	13.75	608	1.65	2.23
LA 1%	13.8	605	1.5	2.3
LA 0.5% + G1 0.55%	14	580	1.75	2.43

1.5% by weight of the cement. The spread diameter of the mixture increases from 12.5 cm to 15 cm in comparison with the reference.

The use of modifying additives makes it possible to increase strength in the early stages of hardening (Table 5). For example, the introduction of PLA additives in the amount of 1% and 1.5% by weight of cement into foam concrete mixture of the average density grade D600 leads to increase the compressive strength at the age of 7 days by 26 and 18%, respectively, compared with the basic composition. The introduction of LA additives in

the amount of 0.5% and 1% by weight of cement leads to increase the compressive strength at the age of 7 days by 22 and 11%, respectively, compared with the reference. The best result in increasing the compressive strength at 7 days and 28 days of age is achieved with the use of a complex additive LA 0.5% + Gl 0.55% – 30% and 24%, respectively.

A positive effect of early structure formation is also observed with the introduction of modifying additives into a foam concrete mixture with a grade of average density D700 (Fig. 1) and D800 (Fig. 2). The best results are



observed with the introduction of PLA additive in the amount of 1.5% by weight of cement. The compressive strength increases by 52% at 7 days of age, by 28% at 28 days of age compared with the basic composition while maintaining the D700 average density grade.

The use of LA additives in the amount of 0.5% by weight of cement leads to increase the strength of foam concrete at 7 days of age by 35%, at 28 days of age by 14% in comparison with the reference.

The parameters of performance properties for the compositions with the best results in terms of compressive strength were carried out as also, as the effect of modifying additives on the plastic shrinkage of the foam concrete mixture.

Plastic shrinkage is a consequence of the rapid loss of water from the concrete surface, leading to the formation of negative capillary pressure in micropores [5]. It causes compression of the mixture, which leads to the formation of cracks. Plastic shrinkage tests of foam concrete mixtures with and without additives were carried out in order to establish the regularity of the influence of additives on the shrinkage of the foam concrete mixture in the first hours of hardening.

The results of the study of plastic shrinkage of foam concrete mixtures are shown in Fig. 3. The assumption proposed by the authors was confirmed experimentally. The data obtained confirm the results of the study on the effect of modifying additives on the process of early structure formation of foam concrete. Accelerating this process, glyoxal makes it possible to stabilize the porous structure of the foam concrete mixture, thereby reducing shrinkage deformations of the foam concrete mixture. Plastic shrinkage decreases (aggregative stability increases) by 22-70% compared to the control composition in foam concrete mixtures with modifying additives. It has been established that the smallest plastic shrinkage (2.01 mm/m) is observed in the foam concrete mixture with LA 0.5% by weight of cement (Fig. 3). The obtained values of plastic shrinkage are 70% less basic composition without additives.

The introduction of modifying additives into the composition of the foam concrete mixture provides the required rheological parameters (mixture flowability) and increases the aggregative stability of the mixture. It leads to increase the strength of the hardened mortar of interpore partitions.

As a result of the analysis of data on statistical processing of measurements of the average density and compressive strength of foam concrete, it can be concluded that the coefficient of variation in compressive strength of foam concrete does not exceed 10% and 3.5% for average density. It indicates the stability of technological processes. The normative value of this parameter is 15% for compressive strength and 5% for the average density of foam concrete. An increase in the compressive strength grade of foam concrete from B1 to B1.5–B2 is provided as a result of increasing the stability of technological processes, while maintaining the average density grade D600, D700 and from B2.5 to B3.5 while maintaining the average density grade D800.

The specified quality indicators are provided with rational structures of foam concrete: compressive strength at a given average density, shrinkage, thermal conductivity and frost resistance. High rates of precisely these properties make it possible to attribute foam concrete to effective building materials.

Based on the foregoing, the work carried out studies of the influence of modifying additives on the coefficient of thermal conductivity and frost resistance of foam concrete. Data on the thermal conductivity of foam concrete samples with additives are given in table. 6.

Analyzing the results, we can conclude that the thermal conductivity coefficient of foam concrete modified with various additives decreased by 5-30% compared to references and corresponds to the standard values of Russian State Standard GOST 25485-2019 for a given average density (Table 6).

Pores with sizes from 0.3 to 0.5 mm are predominantly formed in the cement matrix with modifying additives. The probability of heat transfer due to convection is significantly reduced in such kind of pores. All this together proves the energy efficiency of foam concrete with the studied modifying additives.

As a result of the study, it was found that the frost resistance of modified foam concrete is not inferior to basic samples and is at least 25 cycles at an average density of $600-800 \text{ kg/m}^3$ (Table 7). The introduction of the complex additive LA 0.5% + GI 0.55% into the composition of the foam concrete mixture made it possible to increase the frost resistance grade from F15 to F25. The best result in terms of frost resistance was achieved with

Table 6

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

Type of foam concrete	Thermal conductivity coefficient, W/m • C in dry condition
Basic (reference) D600	0.120
LA 0.5% + Gl 0.55%	0.090
LA 1%	0.085
Basic (reference) D700	0.174
Gl 0.55 %	0.170
PLA 1.5 %	0.168
Basic (reference) D800	0.194
LA 0.5%	0.188



Table 7

Frost resistance grade for foam concrete with modifying additives

Type of foam concrete	Frost resistance grade
Basic (reference) D600	F 15
LA 0.5% + Gl 0.55%	F 25
LA 1%	F 25
Контрольный D700	F 15
G1 0.55 %	F 25
PLA 1.5 %	F 25
Контрольный D800	F 25
LA 0.5%	F 50

the introduction of a PLA additive of 1.5% by weight of cement. The frost resistance grade of foam concrete with an average density of D800 was F50.

The introduction of additives into the composition of foam concrete made it possible to obtain porous structur-

al-heat-insulating concrete with improved performance properties.

CONCLUSION

Using the obtained scientific results, actual compositions have been developed to create a resource-saving technology for the manufacture of cement building compositions with controlled volumetric changes using glyoxal-containing additives for the installation of wall structures in monolithic housing construction.

According to the research results, the use of the optimal amount of modifying additives indicated in this work in the production of cement foam concrete mixtures has a positive effect on the quality of foam concrete and products from it.

The obtained results of the study create the basis for the import substitution of modifying additives on the domestic mineral resource base in the construction industry and the production of effective structural and heatinsulating concretes for the development of the northern regions of Russia.

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2022; 14 (5):

353-362

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