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Composition and properties of cement system with glutaraldehyde

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Abstract. The experience of operating livestock farms shows that periodic reorganization does not prevent biological corrosion of building structures. In this work, the possibility of using glutaraldehyde as a fungicide for volumetric administration in the preparation of biostable concrete was investigated. The study aims to minimize the adverse effects of the components of commercial biocidal products on the physical and mechanical characteristics of products. For research, we used an aqueous solution of glutaraldehyde with an active substance concentration of 0.5 and 1 %, which was introduced into Portland cement CEM I 42.5 N as mixing water. The degree of influence of the active substance on the properties of cement paste and mortar was estimated according to data on heat release during hydration, strength, and phase composition. The fungicidal activity evaluation of glutaraldehyde in cement systems and the degree of their growth with the Aspergillus niger test culture were evaluated. It was established by isothermal calorimetry that, in mixtures with glutaraldehyde, a delay in the achievement of the main hydration peak is observed – by 30 minutes for 0.5 %, by 1 hour for 1 %. After 72 hours of hydration, a lower total amount of released heat of hydration is observed – for 0.5 and 1% solutions by 6.8 and 5.8 %, respectively. According to X-ray phase and differential thermal analyzes, the phase composition of the initial and aldehyde-modified cement stone on day 28 does not differ and consists of the following phases: ettringite, portlandite, calcite and clinker minerals. Varying the phase composition leads to a change in strength: when using a 0.5 % solution of aldehyde, the power of the cement-sand mortar increases by 6.5 %, and a 1 % solution decreases by 6.7 %. The use of a 0.5 % solution of glutaraldehyde provides a decrease in the intensity of fungal development and the formation of a fungistatic effect. Based on the results, a 0.5 % solution of glutaraldehyde can be recommended for the development of cement composites with prolonged bio-resistance.

1. Introduction

The construction of new agricultural construction objects is becoming more and more in demand, which is due to an increase in the consumption of livestock products and crop production. The ability to achieve a high level of environmental safety of food products and technologies for their production depends, among other things, on the conditions of keeping animals, birds, or plants [1–3]. Therefore, regular sanitary and preventive maintenance is essential, as well as costly, part of the general technological process of agricultural enterprises. The experience of the operation of farms shows that periodic cleaning and sanitation of premises does not allow preventing the "biological fatigue" of building materials and structures [4−8]. In particular, biological corrosion, which is the result of many interrelated chemicals, physical, and (micro-) biological processes, affects elements of building structures, which is a problem with social and economic consequences [3, 9–13].

The most used method of protecting building cement materials of already constructed structures against biological corrosion is the use of coatings based on polyurethane, acrylic and epoxy resins, silanes, siloxanes [14, 15]. The disadvantages of using surfaces include a decrease in the breathability of structures,

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poor adhesion, and low durability, which leads to the need for periodic updating of the protective layer. As volumetric methods to increase the natural resistance of cement materials, structure compaction is used by reducing the water-cement ratio, using active mineral additives or polymer fillers [16]. The disadvantage of introducing polymer fillers is the slowdown of cement hydration and their low affinity for concrete components.

It is assumed that the most appropriate solution for the construction of agricultural facilities is the use of building materials with initial resistance to biological corrosion agents. The biological strength of the building material will impede the formation, growth, and development of microorganisms in the inter-recovery period, both on the surface and in volume [2, 17, 18].

Previous studies have justified the need to use biocides as components of building materials that ensure the resistance of finished products to corrosion agents. For example, the volumetric introduction of bactericidal and fungicidal additives, such as nickel powder, calcium tungstate, calcium formate, silver-containing zeolite is well known [16].

The main problem of using this approach is the negative effect on the processes of phase and structure formation, physicomechanical characteristics of the cement system [19−21].

In this regard, when choosing an additive to increase the biological resistance of concrete in the conditions of farms, it is necessary to evaluate both its biocidal activity and the effect on cement paste and mortar, as a factor of durability and safety of the designed composite [3, 20, 22–26].

This work is aimed at studying the possibilities of minimizing the adverse effects of the components of commercial sanitation products on the phase and structure formation of cement. Based on this goal, glutaraldehyde (HA) was selected as a biocide for volumetric administration as a part of a significant amount of commercial biocides as the primary substance active against micromycetes. Glutaraldehyde interacts with the amino group of the cell wall to form an amine bridging bond. It thereby stops cell division, which slows down or stops the growth and development of microorganisms. In an acidic environment, glutaraldehyde can penetrate the cell; in an alkaline environment, it quickly reacts with the outer layers of cells [27]. A study of its effect on cement systems has not been previously conducted, that was the purpose of this study.

2. Materials and Methods

In this work, glutaraldehyde as a biocidal additive; Portland cement CEM I 42.5 N according to Russian State Standard GOST 31108-2016 "Cement for general construction. Special conditions" produced by Belgorodsky Cement CJSC (Belgorod, Belgorod Oblast, RF); reference multifunctional sand according to Russian State Standard GOST 6139−2003 "Sand for cement testing Technical conditions" were used.

Glutaraldehyde $CH_2(CH_2CHO)_2$ is a water-soluble oily liquid containing two aldehyde groups, used as a disinfectant. The biocidal activity of glutaraldehyde is due to the alkylation of sulfhydryl, hydroxyl, carboxyl, and amino groups of the proteins of microorganisms, which violates their vital functions and leads to their death. Glutaraldehyde is actively used for disinfecting livestock and poultry facilities, subject to the recommended concentrations. In this case, the optimal dosage of the active substance in the preparation of its aqueous solution for prophylactic disinfection of premises is a concentration in the range 0.1–0.3 %. With this concentration, degradation of pathogenic microflora is ensured in the absence of toxic effects on animals in farms.

To assess the effect of glutaraldehyde on the properties of the cement system, we used an aqueous solution with an active substance concentration of 0.5 and 1 %, which was introduced into the cement as mixing water. In the volume of the composite, this concentration does not exceed the recommended dosages for the preventive disinfection of rooms.

The following physicochemical and physicomechanical characteristics of cement paste and mortar were studied: heat release during hydration, mineral composition, compressive strength, and also mushroom resistance.

For the manufacture of cement paste and mortar samples, the ratio of glutaraldehyde/cement solution was 0.4. In the manufacture of control samples, the cement was closed with water at a water-cement ratio of 0.4. Hardening of cement was carried out under standard conditions at a temperature of 20±2 °C and relative humidity of 90±5 % until the test age was reached.

For the study, the kinetics of the set of strength, cement-sand mortar samples prepared by EN 196-1 were used. The cement-sand ratio was 3:1. The water-cement ratio of cement-sand mortar and the ratio "glutaraldehyde solution / cement" ratio was 0.55. The increase of water-cement ratio is justified by the need to obtain materials with a higher predetermined porosity in order to create conditions for more complete unhindered development of the fungus on their surface and / or in volume. The compressive strength of cement-sand mortars was determined per the requirements of Russian State Standard GOST 30744–2001 "Cement. Test Methods Using Polyfractional Sand". To determine the strength, beam samples with size 40×40×160 mm were prepared. For the compressive strength, the arithmetic mean value of the test results of six halves of the beam samples for each composition was taken.

To determine the heat release during portland cement hydration, we used a ToniCAL model 7338 differential heat flow calorimeter (Toni Technik Baust off prüf Systeme GmbH, Germany). Differential thermal analysis with thermogravimetry of cement stone samples at the age of 28 days was performed on an STA 449 F5 thermal analysis device (NETZSCH-Geräte Bau GmbH, Germany); X-ray phase analysis – on a D8 Advance X-ray diffractometer (BRUKER Corporation, USA) (Cu Kα, λ = 1.54 Å).

The fungus resistance of cement stone was assessed by the growth of samples (the ability to grow and propagate on them) by mold fungi. The test was conducted according to Russian State Standard GOST 9.049–91 "Unified system of corrosion and ageing protection. Polymer materials and their components. Methods of laboratory tests for mould resistance"(Method 3).

The evaluation of the fouling of the cement material by microscopic fungi was carried out using an AXIOSCOPEA1 biological microscope. The degree of development of fungi was evaluated on a 6-point scale, according to GOST 9.048–89. Material has fungicidal properties if no fungi are found on its surface or the intensity of their development is estimated with less than 1 point.

After 28 days of hardening, mortars placed under ultraviolet light in a laminar cabinet for 6 hours with a calculation of 3 hours on one side in order to remove possible organic contaminants from other microflora on the surface and in the volume of the sample.

For the glutaraldehyde fungicidal activity evaluation of in cement systems (pastes and mortars), the *Aspergillus niger* test culture was used. This fungus is one of the most aggressive types of molds. The development of this fungus on the surface of construction sites due to its high survival rate causes the maximum degree of degradation effect [19].

As a nutrient medium for the test culture, Chapek's prepared medium was used, having the composition: 1000 ml of distilled water (pH = 6.7): monosubstituted potassium phosphate – 0.7 g; potassium phosphate disubstituted 3-water – 0.3 g; magnesium sulfate 7-water – 0.5 g; sodium nitrate – 2.0 g; potassium chloride – 0.5 g; iron (II) sulfate 7-water – 0.01 g; sucrose – 9 g; distilled water – 1000 ml, agar – 20 g. The finished mixture was poured into 15 ml tubes and placed vertically in an MLS-2420U Sanyo autoclave for heat treatment for 15 minutes at 122 °C. At the end of the process, the tubes were placed in a laminar cabinet, and the nutrient medium was uniformly poured on the bottom of the Petri dishes, followed by sterilization of the latter.

The infection of the nutrient medium was carried out by spraying a solution containing the spores of the control fungus with a spray gun. After sowing, samples of mortars were placed in the center of the Petri dishes. Then, closed Petri dishes were placed in a RI 115 thermostat with natural ventilation red LINE by Binder for 14 days at t = 30 °C, W = 90 %.

3. Results and Discussion

3.1. Effect of the biocide at early age

A study of the heat release of a cement paste with glutaraldehyde (Fig. 1) showed that its introduction does not lead to significant changes in the hydration process in the first 72 hours. The similar nature of the heat release of all the compositions under consideration, which corresponds to a typical Portland cement test, is noted. The peak in the first five minutes of the study refers to the process of exothermic wetting and reactions of the early stage of hydration. The second (central) peak mainly corresponds to the reactions of the middle stage of hydration with the formation of phases C-S-H and CH. Then, the heat release rate gradually decreases, while slow reactions of the late stage of hydration also occur with the formation of C-S-H and CH. An unclear third peak (about 13 hours for the materials understudy) is associated with the formation of AFm phase from C_3A and ettringite [28–30].

In mixtures with glutaraldehyde, there is a slight delay in reaching the main hydration peak – by 30 minutes for 0.5 %, by 1 hour for 1 %. Its intensity (Fig. 1a) also decreases slightly. As a result, after 72 hours of hydration, a lower total amount of released heat of hydration is observed in comparison with the control composition.

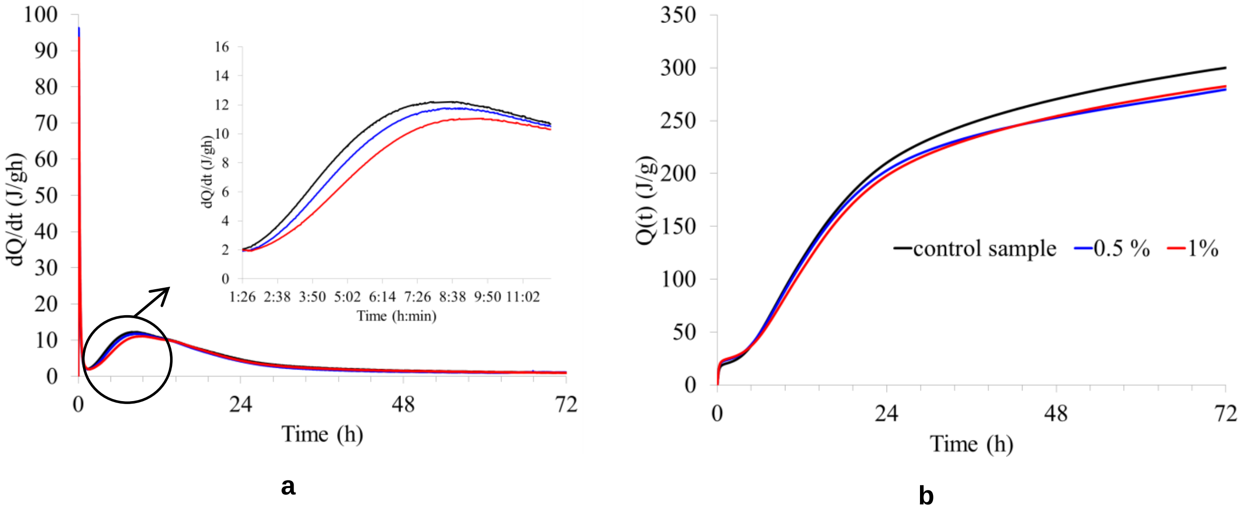


Figure 1. Rate of heat evolution (a) and total heat of hydration (b) for different glutardialdehyde content.

According to the data obtained, it can be said that glutaraldehyde in the early stages slightly slows down the hydration of Portland cement. Most likely, this process may occur due to adsorption. In this process, one or both of its functional groups are capable of reacting with hydroxyl groups on the surface of Portland cement particles.

3.2. Effect of the biocide at 28 days

The results of differential thermal analysis with thermogravimetry show the presence of an equal endo effect at 100–120 °C for all three samples, which is attributed to dehydration of C-S-H and ettringite (Fig. 2). Mass losses at temperatures of 430-470 °C correspond to the decomposition of portlandite. The end effect attributable to portlandite is most intense for the composition with 0.5 % glutaraldehyde. The subsequent end effect at higher temperatures corresponds to the decarbonization of calcite present in the anhydrous cement.

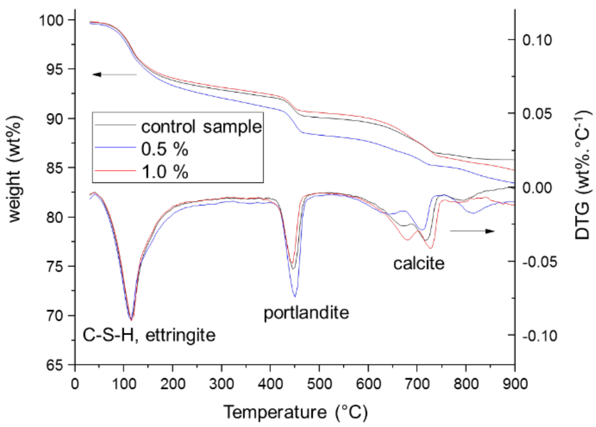


Figure 2. TGA/DTG of cement paste with different glutardialdehyde content after 28 days hydration.

As reported in [31, 32], A CO_2 weight loss peak is observed at around 720 °C because of the presence of relatively coarse calcite in the anhydrous cement and a second weight loss peak may be present at 600 to 650 °C because of the presence of mono- or hemicarbonates and due to the carbonation of portlandite and possibly C-S-H. The peak above 800°C may be due to the decomposition of C-S-H to wollastonite [31].

The absence of a significant effect of glutaraldehyde on the phase formation processes of Portland cement is also confirmed by the results of x-ray phase analysis of hardened cement at the age of 28 days (Fig. 3).

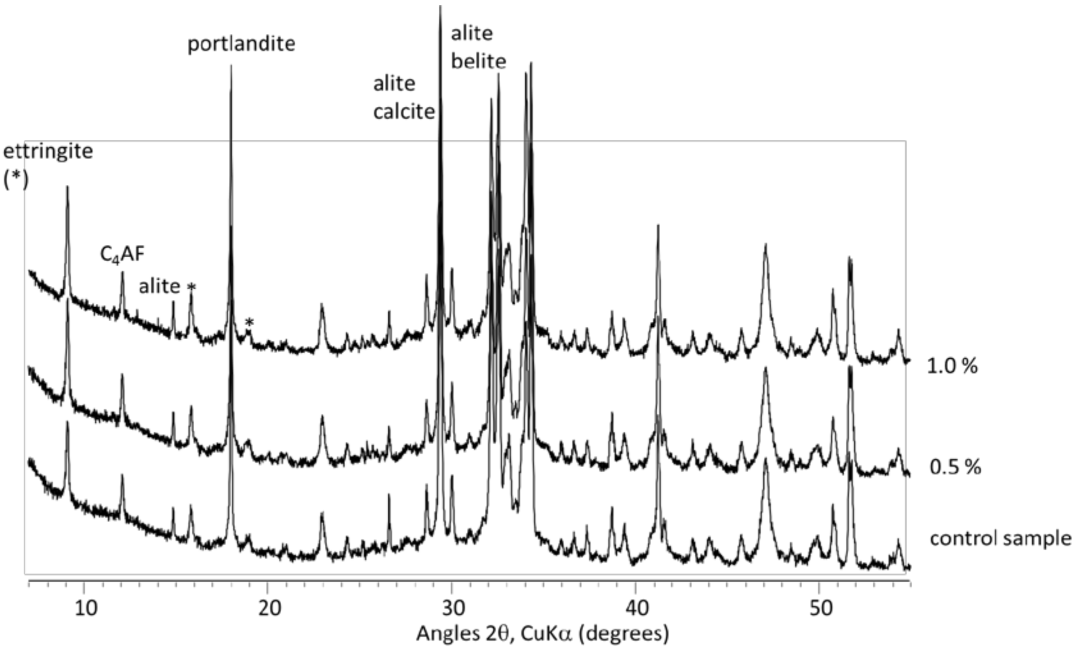


Figure 3. X-ray diffraction patterns of hardened cement paste with different glutardialdehyde content after 28 days hydration (Standard cement chemistry notation is used. As per this simplified notation: C = CaO, A = Al_2O_3, F = Fe_2O_3 and S = SiO_2).

In all the studied samples, both control and glutaraldehyde, the following were noted: the remains of unreacted clinker minerals (alite, belite, calcium aluminate C_3A and calcium aluminoferrite C_4AF phases) and crystalline hydration products (ettringite, portlandite).

When studying the kinetics of the set of compressive strength of a cement-sand mortar with glutaraldehyde, its significant influence is also not observed (Fig. 4).

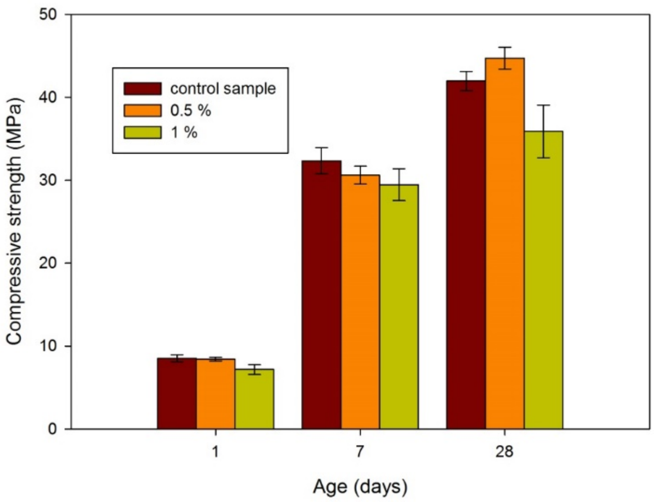


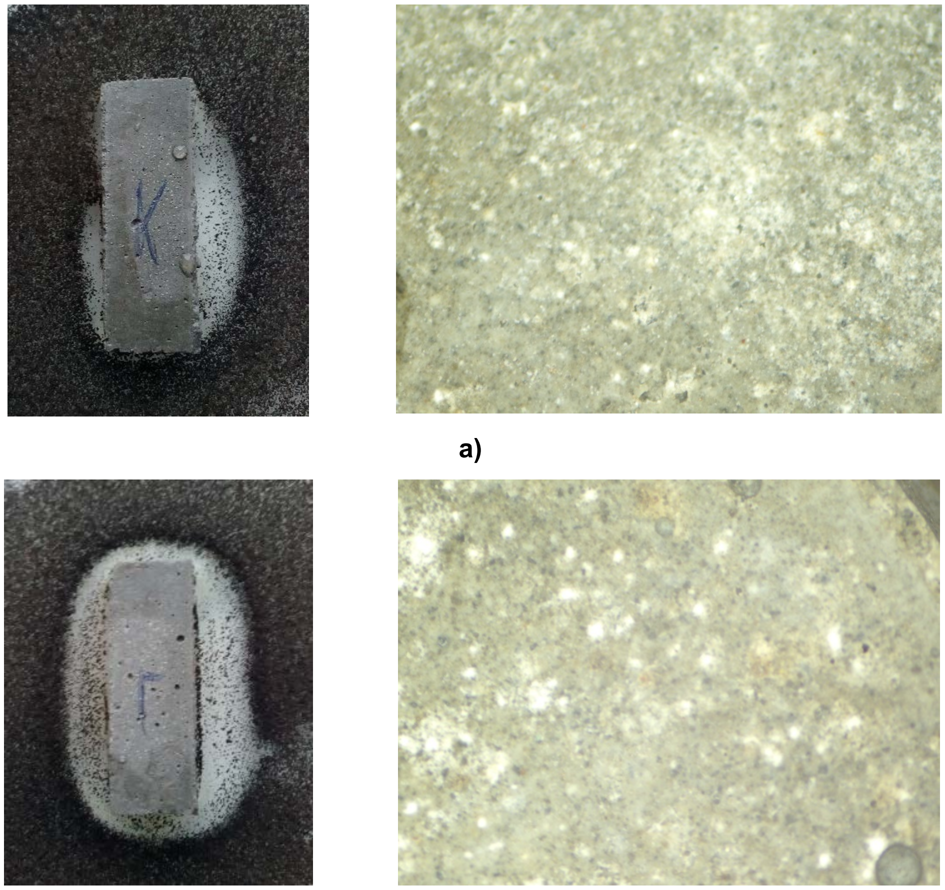
Figure 4. Compressive strength of hardened cement-sand mortar with different glutardialdehyde content.

The use of an aldehyde solution at a concentration of 0.5 % leads to a slight increase in the strength of the system on the 28^{th} day of hydration hardening compared with the control sample, and in a concentration of 1 % to a decrease.

3.3. Effect of the biocide at antifungal effect

After 14 days from the start of the experiment, the active growth of Aspergillusniger fungus on the surface of the nutrient medium in all Petri dishes can be noted. Moreover, spores, conidia, and mycelium are absent on the surface of the studied samples of Portland cement stone (Fig. 5).

I II



a) b)

Figure 5. Development of the Aspergillus niger test culture on a growth medium in the presence of a Portland cement stone sample: a – control, b – with glutaraldehyde (0.5 %): I – general view; II – shooting with a biological microscope (× 60 times).

The cement system has a porous structure, which contributes to the growth of the microorganisms on the surface and in the volume of the material, and a high alkalinity in the early stages of hardening. Terms can reach several months before the onset of a high degree of carbonation during operation. However, despite these features of cement, the composition of cement impedes the growth of living organisms [26]. In this regard, a small zone of fungicidal activity is noted even around the control sample (Fig. 5 a). However, it can be noted that the use of glutaraldehyde increases the fungicidal zone around cement stone samples (Fig. 5, b). The data are confirmed by surveying the surface of the samples under a biological microscope (Fig. 5, II).

On the surface of the control sample of cement-sand mortar based on Portland cement (Fig. 6, a), the significant development of *Aspergillus niger* fungus is noted. On a sample with glutaraldehyde (Fig. 6, b), only individual elements of the mycelium are present. This is probably due to a decrease in the total alkalinity of the samples due to a decrease in the mass percentage of the active component – cement – when adding a filler. The result is a decrease in the resistance of the material to the action of filamentous fungi [33].

When assessing the fouling of samples in points (Table 1), we can quantitatively note an increase in the fungal resistance of a sample with glutaraldehyde. In the case of the original cement stone and with the addition of glutaraldehyde, a fungistatic effect to *Aspergillus niger* fungus is observed: the inhibition zone increases significantly. In the case of a cement-sand mortar without additives, there is no fungicidal effect. The aldehyde-modified mortar retains fungicidal properties with a slight decrease in the zone of inhibition.

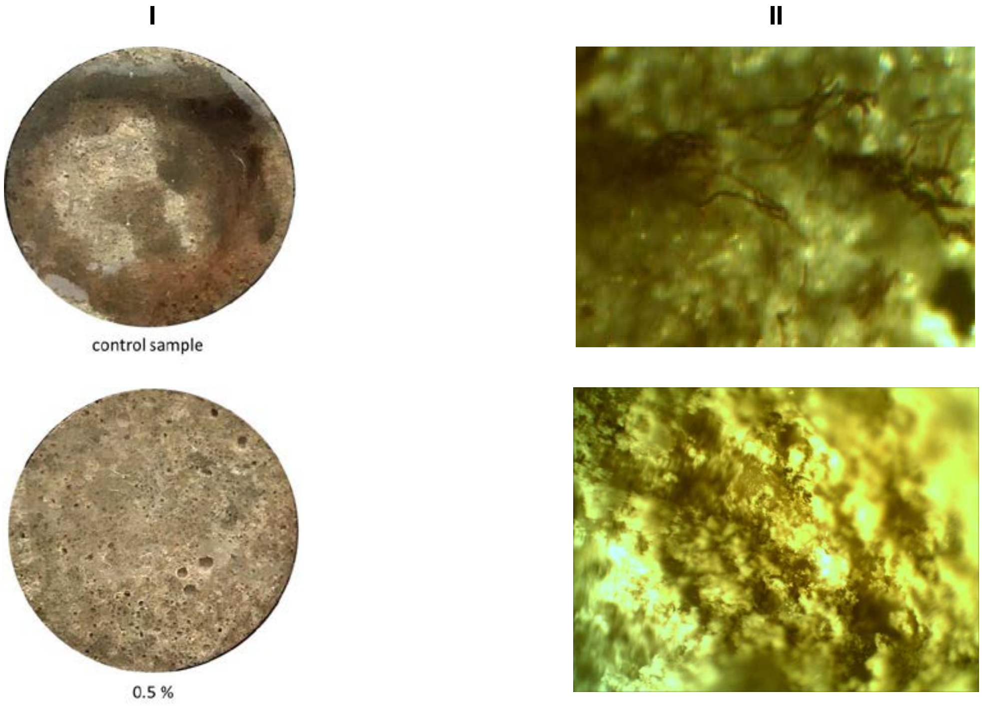


Figure 6. Antifungal effect of hardened cement-sand mortar (diameter 5 cm) with different glutardialdehyde content I – general view; II – shooting with a biological microscope (× 60 times).

Table1. Biostability of cement stone and cement-sand mortar samples to Aspergillus niger.

Sample	Evaluation of fungi growth, points	Inhibition zone, mm	Material evaluation
Cement stone			
Control sample	0	4	Fungicidal
0.5 %	0	10	Fungicidal
Cement-sand mortar			
Control sample	3	–	Fungus-proof (no fungicidal effect)
0.5 %	0	7	Fungicidal

4. Conclusions

A comprehensive analysis of the glutaraldehyde effect as a fungicidal additive on the composition and properties of the cement system was carried out.

1. The effect of fungicidal additives on the heat release of cement paste during hydration in the first 72 hours is established. In mixtures with glutaraldehyde, a delay in the achievement of main hydration peak is observed – by 30 minutes for 0.5 %, by 1 hour for 1 %. After 72 hours of hydration, a lower total amount of released heat of hydration is observed – for 0.5 and 1 % solutions by 6.8 and 5.8 %, respectively.

2. Composition change leads to varying in strength: when using a 0.5 % aldehyde solution, the strength of the cement-sand mortar increases by 6.6% and 1% decreases by 14.5 %.

3. An increase in the resistance of samples for *Aspergillus niger* fungus is shown. The use of a solution of glutaraldehyde (0.5 %) in the case of cement stone leads to an increase in the fungicidal zone. The introduction of the biocide into the composition of the cement-sand mortar provides a fungistatic effect: the development of the fungus on the surface of modified samples is not observed.

Thus, the main effect of glutaraldehyde is to slow down hydration of the hydrating system by possible adsorption on the surface of cement particles and hydrated formations without a subsequent critical negative effect on the compressive strength of cement at early age. At the same time, a significant change in the mineral composition of cement stone is not observed, and the fungal resistance increases. On this basis, a recommendation is proposed for the use of a 0.5 % solution of glutaraldehyde for the development of cement composite materials with bio-resistance. If it is necessary to use a 1 % solution (in environments with increased bioaggression), it is recommended to take into account a negative effect on the rate of increase in compressive strength and its final value.

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