Biological and Climatic Resistance of Cement Composites Based on Biocidal Binders

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Abstract. The aim of this work is to study the durability in biological media of cement composites based on biocidal cements. Formulation of biocidal cements was carried out using the following components: Portland cement clinker (JSC "Mordovcement", Russia); calcium sulfate dihydrate (Poretskoe deposit, Nizhny Novgorod region); fly ash of Krasnoyarsk CHP-3 (Russia), biocide preparations: sulfuric acid sodium, sodium fluoride. The finished specimens were incubated in a medium consisting of 10 species of mycelial fungi. Identification of dominant micromycetes infesting the surface of composites made on ordinary Portland cement and several types of biocidal cements obtained using different biocidal preparations was carried out. No more than 2 species of mycelial fungi were found around specimens based on biocidal cements as opposed to ordinary ones—against 7–8. At the same time, it is important that the absence of the most dangerous for human health micromycetes (Aspergillusniger) around the composites made with biocidal cements was revealed. Long-term tests (up to 180 days) revealed high fungal resistance and fungicidity of individual compositions with biocidal additives and insignificant variability of properties both in standard biological environment and in the products of their metabolism. The biocidal properties of the developed composites were tested in full-scale climate conditions of the Black Sea coast and the Leningrad Region. Developed biocidal cements with active mineral additives are recommended for use in buildings with biologically active media in the manufacture of concrete and other cement composites with increased biological resistance.

Keywords: Biocidal cements · Compositions · Study of properties · Change in mass content · Strength · Fungal resistance · Fungicidity · Climatic resistance · Properties of cements

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1 Introduction

Concretes of various types are widely used in the construction industry. This contributes to the development of conducting numerous researches in the world practice. To date, rational compositions of concretes and other cement composites with improved indicators of physical and mechanical properties and increased durability have been developed [1–6]. Portland cement, which is produced by grinding Portland cement clinker with calcium sulfate dihydrate, is the most widely used as a binder in the production of concrete and products based on them. In the construction industry cements of grades 400, 500, 550, 600 are used. In addition to strength, cements have a number of other requirements, such as technological (normal density, setting time), for durability. Information is given on special binders, including mixed, composite, activated. Improving the properties of cement composites is achieved by introducing plasticizers and other additives.

The introduction of fine-grained active fillers into both cements and concrete mixes is particularly effective, leading to improvements in a number of properties of cement-based concretes.

It is shown that the use of industrial wastes in the process of obtaining various construction materials, including binders and concretes, allows increasing their physical and mechanical properties and simultaneously solving such problems as: environmental protection and the creation of waste-free technologies of production of construction materials. The most promising direction, according to a number of experts, is the use of ash as a component of mixed cements.

Recently, more and more researchers and practitioners pay attention to improving the biostability of materials and products and the elimination of the negative fact of biodeterioration of buildings and structures [7, 8].

The economic damage from bio-damage worldwide is estimated at tens of billions of dollars annually, and the list of human diseases caused by microscopic organisms is growing [9, 10].

Both in our country and abroad there is intensive research on improvement of known and development of new types of cements. Many authors have established that the most corrosive for cement and other construction composites are the following types of bacteria: Nitrosomonas, Ciostridium, Micrococcus, Tiobacillus, Desulfovibrio, Acetobakter, и мицелиальных грибов: Aspergillus, Penicillium, Trichoderma, Mycor, Cladosporium, Cepfalosporium, Torula, Saccharomyces, Pichia, Coniophora, Poria, Serpula.

In addition to destruction processes, microorganisms have a negative impact on human life. The population living in contaminated premises is prone to diseases of the respiratory, circulatory, digestive, nervous, musculoskeletal and connective tissue systems, endocrine and genitourinary systems, etc. The main causative agents of diseases associated with biodegradable microbes are micromycetes. The most common pathogens are fungi of the genus Aspergillius, which cause aspergillosis. In addition, certain species of mold fungi are potentially oncogenic.

The paper considers the issues of chemical and biological corrosion of cement concrete, durability of products based on them under the influence of aggressive media. Mechanisms of destruction of cement composites under the influence of mycelial fungi, bacteria and products of their metabolism are analyzed. Various fouling and waste products of bacteria and fungi have a destructive effect on concrete and other materials. The destruction of concrete structures, building elements is further aggravated by the combined effects of microorganisms in the presence of mechanical damage, climatic factors.

One of the effective ways to increase the biostability of cement composites is the introduction of fungicidal and bactericidal additives in their composition.

The expediency of increasing the biostability of cement composites through the use of special cements with biocidal properties is substantiated below.

The aim of the study is to investigate the durability of cement composites based on biocidal cements with active mineral additives.

1. To study the durability of composites based on biocidal cements with an active mineral additive in the environment of mycelial fungi and products of metabolism of fungi and bacteria, in laboratory conditions, in sea water, climatic conditions of the sea coast in full-scale tests.
2. To select biocidal additives effective for use in cement composites based on the results of strength and biostability studies.
3. To establish quantitative indicators of resistance of composites based on biocidal cements with an active mineral additive in biological media, water and aqueous acid solutions.
4. To study the climatic resistance of cement composites composed on the basis of biocidal Portland cement with an active mineral additive.

2 Materials and Methods

The following components were used for the manufacture of biocidal cements: Portland cement clinker (JSC "Mordovcement") of mineralogical composition: $3CaOSiO_2$ (59–63%), $2CaOSiO_2$ (16–18%), $3CaOAl_2O_3$ (6–7.5%), $4CaOAl_2O_3Fe_2O_3$ (11–12%), calcium sulfate dihydrate (GOST 4013–82) (Poretskoe deposit), biocidal preparations: sodium sulphate (GOST 4166–76), sodium fluoride (GOST 4463–76), fly ash of the Krasnoyarsk CHP-3.

Biocidal cements were produced by grinding cement clinker, calcium sulfate dihydrate and biocidal additive (Na_2SO_4, NaF, PHMG-C) to achieve a specific surface area of 2900–3000 cm^2/g.

When evaluating the biostability of cement composites for comparison with the developed materials as binders were used ordinary and white Portland cement of domestic and foreign production: Shchurovsky, Volsky, Mordovsky, Danish, Egyptian and many others, the chemical and mineralogical composition of which was also determined in the work [11].

Standard Volsky sand $M_k = 2.45$ (GOST 6139–91) and natural quartz sand for the manufacture of concrete and mortars $M_k = 1.4$ (GOST 8736–93) (Smolninskoye deposit of Republic of Mordovia) were used as aggregates.

The technology for making specimens is described in Ref. [11].

Physical and mechanical properties of cements and composites based on them were determined according to GOST 310.3–76, GOST 30,744–2001, GOST 310.6–85. Physical and mechanical methods of research (RFA, TG, DTG, DSC, calorimetry, etc.), as well as biological methods (method of imprints, method of sampling, method of determining fungal resistance and fungicidity according to GOST 9049–91) were used in the work.

Biological, climatic and chemical resistance of cement composites with an active mineral additive was studied in comparison with cement composites made on ordinary Portland cement. Specimens of composites were tested for fungal resistance and the presence of fungicidal properties in accordance with GOST 9049–91. The following species of micromycetes were used as test organisms: Aspergillusoryzae, Aspergillusniger, Aspergillusterreus, Chaetomiumglobosum, Paecilomycesvariotii, Penicilliumfuniculosum, Penicilliumchrysogenum, Penicilliumcyclopium, Trichodermaviride.

3 Results and Their Analysis

Studies of resistance of biocidal cement composites with an active mineral additive in a standard biological medium and products of metabolism of mycelial fungi and bacteria have been conducted. The results of their tests for fungal resistance according to GOST 9.049–91 are given in Table 1.

Identification of dominant species of micromycetes on the surface of composites based on cements from 10 national and foreign manufacturers with differences in chemical and mineralogical composition after 1 month of testing in a standard medium of mycelial fungi was carried out.

Species dominance has been established on almost all decorative cement composites: Aspergillusniger, Penicilliumcyclopium и Penicilliumchrysogenum. On the surface of composites on ordinary cements two dominant species of micromycetes of the genus Penicillium (Penicilliumchrysogenum and Penicilliumcyclopium) are distinguished. In addition to micromycetes of the genus Penicillium, two species of the genus Aspergillus were identified - Aspergillusniger, on all composites made on national cements, which is associated with increased content of Mg, K, Na in them, and Aspergillusterreus, identified on the surface of composites on Krasnoyarsk and Mordovian standard composition cements. It should also be noted the presence of Trichodermaviride on the surface of composites on cement of JSC "Volskcement" and JSC "Mordovcement", which is associated with an increased content of P and Mn in their composition, and on the surface of composites on cement of French production Paecilomycesvariotii, which has a lower content of Mg, K, Na, P and Mn.

After 6 months of keeping in standard medium of mycelial fungi strength of cement stone samples of normal density on white cements decreased by 15–20%, and on ordinary cements by 20–30% depending on pore space of composites, chemical and mineralogical composition of cements and on dominating micromycetes in vicinity and on surface of composites (see Table 1).

Table 1 Fouling and coefficient of biostability of composites based on cements of national and foreign production

№ of composition	Name of cement. Producer	Assessment of fungi growth, points		Characteristics according to GOST 9.049-91	Biological resistance coefficient		
		Method 1	Method 3		1 month	3 months	6 months
1	M 600 D0 (France)	0	4	Fungal resistant	1.04	0.93	0.88
2	M 500 D0 (Russia, Krasnoyarsk)	0	4	Fungal resistant	1.05	0.9	0.8
3	M 500 D0N(Russia, Chamzinka village)	0	4	Fungal resistant	1.05	0.9	0.85
4	M 500 D0 (Russia, Volsk)	1	3	Fungal resistant	1.07	0.87	0.78
5	M 500 D0N (Russia, Volsk)	0	4	Fungal resistant	1.06	0.92	0.83
6	M 400 D0 (Russia, Stary Oskol)	0	4	Fungal resistant	1.09	0.85	0.73
7	M 500 D0 (Russia, Ulyanovsk)	0	4	Fungal resistant	1.07	0.85	0.76
8	M 500 D0 (Russia, Chamzinka village)	0	4	Fungal resistant	1.03	0.93	0.82
9	White M 500 D0 (Russia, Kolomna)	0	4	Fungal resistant	1.04	0.96	0.85
10	White M 600 D0 (Egypt)	0	4	Fungal resistant	1.05	0.90	0.8
11	White M 600 D0 (Denmark)	1	4	Fungal resistant	1.04	0.95	0.83

(*continued*)

Table 1 (*continued*)

№ of composition	Name of cement. Producer	Assessment of fungi growth, points		Characteristics according to GOST 9.049-91	Biological resistance coefficient		
		Method 1	Method 3		1 month	3 months	6 months
12	White M 600 D0 (Italy)	1	4	Fungal resistant	1.05	0.93	0.82

The biostability of cement composites modified with sodium sulfate, sodium fluoride and polyhexamethylene guanidine stearate has been established. The following cement compositions modified with biocidal preparations were tested: 1—100 wt. h. clinker, 6 wt. h. CaSO4 2H2O, 4.5 wt. h. Na2SO4; 2—100 wt. h. clinker, 0–8 wt. h. CaSO4 2H2O, 3–4.5 wt. h. NaF; 3—100 wt. h. clinker, 0–6 wt. h. CaSO4 2H2O, 1–2 wt. h. PHMG–C. At this content of components cement composites have fungicidal properties. After 6 months of incubation in a standard medium of mycelial fungi the strength of composites based on cements with fungicidal properties decreased only by 3–8% in comparison with non-added compositions, the strength of which decreased by 25–40% (see Tables 2, 3 and 4).

The dominant species of micromycetes near specimens made with biocidal cements were identified after long-term tests in standard mycelial fungi medium. Around the specimens based on cements modified with sulfuric acid sodium there is a dominance of species—Aspergillusterreus and Penicilliumcyclopium; modified with sodium fluoride—Aspergillusterreus, Penicilliumcyclopium and Aspergillusoryzae; modified with polyhexamethyleneguanidine stearate—Aspergillusoryzae. At the same time, the presence of the most dangerous for human health micromycetes (Aspergillusniger) around the composites made with biocidal cements was not detected.

On the basis of the conducted complex research the following compositions of cements for the manufacture of materials, products and structures, resistant in biologically active media are recommended: 1—100 wt. h. clinker, 4.5 wt. h. Na_2SO_4, 6 wt. h. $CaSO_4 * 2H_2O$; 2—100 wt. h. clinker, 3 wt. h. NaF, 0–8 wt. h. $CaSO_4 * 2H_2O$; 3—100 wt. h. clinker, 4.5 wt. h. NaF, 0–6 wt. h. $CaSO_4 * 2H_2O$; 4—100 wt. h. cklinker, 1–2 wt. h. PHMG–C, 0–6 wt. h. $CaSO_4 * 2H_2O$.

In a study of the fouling properties of cement composites made of Portland cement, biocidal Portland cement, Portland cement with an active mineral additive in laboratory conditions in a standard biological environment found that the composites based on Portland cement without biocidal additive have fungal resistance 0 and 1 point in the test by method 1, but fouled in the test by method 3 (3 and 4 points), that is, they are non-fungicidal.

Based on the studies of the biostability of cement composites, the following biocidal compositions of binders were identified, which showed a fungistatic effect

Table 2 Effect of $CaSO_4 * 2H_2O$ and Na_2SO_4 content on fouling and bioresistance coefficient of cement stone

№ of composition	Content of $CaSO_4$ * $2H_2O$, wt.h	Content of Na_2SO_4, wt.h	Assessment of fungi growth, points		Characteristics according to GOST 9.049-91	Biological resistance coefficient		
			Method 1	Method 3		1 month	3 months	6 months
1	0	0	0	4	Fungal resistant	0.99	0.8	0.60
2	2	0	0	4	Fungal resistant	1.06	0.87	0.76
3	4	0	0	4	Fungal resistant	1.02	0.88	0.75
4	6	0	0	4	Fungal resistant	1.07	0.86	0.75
5	8	0	0	4	Fungal resistant	1.07	0.79	0.63
6	0	1.5	2	4	Fungal resistant	1.07	0.94	0.79
7	2	1.5	2	4	Fungal resistant	1.06	1.00	0.86
8	4	1.5	0	4	Fungal resistant	1.06	1.02	0.85
9	6	1.5	2	4	Fungal resistant	1.09	0.98	0.82
10	8	1.5	2	4	Fungal resistant	1.06	0.92	0.77
11	0	3	0	2	Fungal resistant	1.02	0.86	0.70
12	2	3	2	3	Fungal resistant	1.05	0.99	0.92
13	4	3	2	4	Fungal resistant	1.05	1.05	0.91
14	6	3	2	4	Fungal resistant	1.01	0.97	0.93
15	8	3	2	4	Fungal resistant	1.08	0.94	0.82
16	0	4.5	0	3	Fungal resistant	1.05	0.99	0.90
17	2	4.5	0	3	Fungal resistant	1.00	0.98	0.95
18	4	4.5	0	3	Fungal resistant	1.02	1.05	0.98
19	6	4.5	0	0 (0)	Fungicidal	1.07	1.02	0.98
20	8	4.5	0	3	Fungal resistant	1.05	1.00	0.96
21	0	6	1	3	Fungal resistant	1.05	1.03	0.97
22	2	6	1	4	Fungal resistant	0.99	0.95	0.89
23	4	6	1	3	Fungal resistant	0.95	0.92	0.88
24	6	6	0	4	Fungal resistant	0.97	0.94	0.88
25	8	6	2	4	Fungal resistant	0.97	0.94	0.91

(wt. h.): (1) Portland cement clinker—100, CaSO4·2H2O—6.0–11.2, Na_2SO_4—7.0, fly ash—10.0—20.0 wt. h.; (2) Portland cement clinker—100, CaSO4·2H2O—6.0–8.6, Na2SO4—3.5, fly ash 13—10.0–20.0 wt. h.; (3) Portland cement clinker—100, CaSO4·2H2O—6.0–11.2, NaF—2.0–4.0, fly ash—10.0–20.0 wt. h. It follows from the research results that cement composites without a biocidal additive become overgrown with microorganisms, therefore, even high-density materials will be exposed to the aggressive effects of metabolic products.

The results of tests to establish the species composition of microorganisms contaminating the surface of specimens of cement composites aged in the open area and under a canopy on the Black Sea coast and in the climate of the Leningrad region, as well as after aging in sea and ground water are presented. The Black Sea coast is characterized by a

Table 3 Effect of $CaSO_4 * 2H_2O$ and NaF content on fouling and bio-resistance coefficient of cement stone

№ of composition	Content of $CaSO_4$ * $2H_2O$, wt.h	Content of NaF, wt.h	Assessment of fungi growth, points		Characteristics according to GOST 9.049-91	Biological resistance coefficient		
			Method 1	Method 3		1 month	3 months	6 months
1	0	0	0	4	Fungal resistant	0.99	0.8	0.61
2	2	0	0	4	Fungal resistant	1.06	0.87	0.76
3	4	0	0	4	Fungal resistant	1.02	0.88	0.75
4	6	0	0	4	Fungal resistant	1.07	0.86	0.75
5	8	0	0	4	Fungal resistant	1.07	0.79	0.63
6	0	1.5	0	3	Fungal resistant	1.05	0.88	0.81
7	2	1.5	0	3	Fungal resistant	1.07	0.90	0.81
8	4	1.5	0	3	Fungal resistant	1.08	0.92	0.85
9	6	1.5	0	3	Fungal resistant	1.08	0.95	0.86
10	8	1.5	0	3	Fungal resistant	1.05	0.93	0.85
11	0	3	0	0 (40 mm)	Fungicidal	0.99	0.97	0.92
12	2	3	0	0 (40 mm)	Fungicidal	1.00	0.98	0.93
13	4	3	0	0 (40 mm)	Fungicidal	0.98	0.96	0.94
14	6	3	0	0 (40 mm)	Fungicidal	0.99	0.97	0.95
15	8	3	0	0 (40 мм)	Fungicidal	0.98	0.96	0.94
16	0	4.5	0	0 (40 mm)	Fungicidal	0.97	0.94	0.88
17	2	4.5	0	0 (40 mm)	Fungicidal	0.95	0.92	0.86
18	4	4.5	0	0 (40 mm)	Fungicidal	0.94	0.93	0.89
19	6	4.5	0	0 (40 mm)	Fungicidal	0.83	0	0
20	8	4.5	0	0 (40 mm)	Fungicidal	0.72	0	0

warm climate, in which case the samples are exposed to variable humidity, salt spray, wind, and, in the open area, also to ultraviolet irradiation. Leningrad Region belongs to the zone with moderate climate, which is characterized by high humidity. It is known that such conditions intensify the processes of biodegradation.

The species composition of microorganisms isolated from the surface of specimens aged in warm and temperate climatic zones are shown in Tables 5 and 6.

Climatic zone Exposure conditions of specimens Species composition of microorganisms on specimens 100 wt.h. clinker, 8.6 wt.h. CaSO4·2H2O, 10.

The test results showed that the species and quantitative composition of mycobiota, contaminating cement composites is ambiguous, and depends on the formulation of materials and the effect on them of environmental factors.

Table 4 Effect of $CaSO_4 * 2H_2O$ and PHMG–C content on fouling and the coefficient of wear resistance of cement stone

№ of composition	Content of $CaSO_4 *$ $2H_2O$, wt.h	Content of PHMG–C, wt.h	Assessment of fungi growth, points		Characteristics according to GOST 9.049-91	Biological resistance coefficient		
			Method 1	Method 3		1 month	3 months	6 months
1	0	0	0	4	Fungal resistant	0.99	0.80	0.61
2	2	0	0	4	Fungal resistant	1.06	0.87	0.76
3	4	0	0	4	Fungal resistant	1.02	0.88	0.75
4	6	0	0	4	Fungal resistant	1.07	0.86	0.75
5	0	0.5	0	3	Fungal resistant	1.03	0.89	0.78
6	2	0.5	0	2	Fungal resistant	1.04	0.91	0.82
7	4	0.5	0	2	Fungal resistant	1.04	0.95	0.87
8	6	0.5	0	2	Fungal resistant	1.05	0.94	0.88
9	0	1	0	1	Fungicidal	1.02	0.97	0.92
10	2	1	0	0 (0)	Fungicidal	1.02	1.00	0.95
11	4	1	0	0 (0)	Fungicidal	1.01	1.00	0.95
12	6	1	0	1	Fungicidal	1.01	1.01	0.95
13	0	2	0	0 (3 mm)	Fungicidal	1.01	1.00	0.97
14	2	2	0	0 (5 mm)	Fungicidal	1.01	1.01	0.98
15	4	2	0	0 (5 mm)	Fungicidal	1.02	1.02	0.98
16	6	2	0	0 (4 mm)	Fungicidal	1.02	1.01	0.97

4 Conclusions

1. The resistance of cement composites under the influence of water and chemical aggressive media: acid solutions, machine oil, gasoline were experimentally investigated. Using Excel table processor the functions describing the change in mass content and resistance coefficient of biocidal cements with active mineral additive were obtained. After 110 days of exposure the highest resistance has the following compositions, (wt.h.): (1) clinker—100, CaSO4·2H2O—8.6, Na2SO4—3.5, fly ash—20; (2) clinker—100, CaSO4·2H2O—8.6, NaF—4.0, fly ash—10.
2. The degree of fouling by mycelial fungi was revealed and the resistance of the investigated cement composites to microbiological aggressive media was established. Composites compositions modified with biocidal additives showed fungistatic effect. The kinetic dependences of changes in mass content and strength of cement composites specimens on the duration of soaking in the media were analyzed. Quantitative dependences of changes in strength and mass content of cement compositions in a standard biological medium depending on the formulation factors were revealed. Compositions of biocidal cements were revealed (wt. h.): (1) clinker—100, CaSO4·2H2O—6.0, Na2SO4—3.5; (2) clinker—100, CaSO4·2H2O—6.0,

Table 5 Test results of specimens exposed in the warm zone

Climatic zone	Specimen exposure conditions	Species composition of microorganisms on the specimens	
		100 wt.h. clinker, 8.6 wt.h. $CaSO_4 \cdot 2H_2O$, 10 wt.h. fly ash, 7.0 wt.h. sodium sulfate	100 wt.h. clinker, 11.2 wt.h. $CaSO_4 \cdot 2H_2O$, 20 wt.h. fly ash, 4.0 wt.h. sodium sulfate
Warm (Novorossiysk)	In an airy environment under a canopy	*Alternaria brassicae, Alternaria tenuissima*	*Aspergillus oryzae, Cladosporium herbarum*
Warm (Novorossiysk)	In an open air environment	*Botryotrichum piluliferum, Fusarium miniliforme*	***Not found***
	On the open area after aging in seawater	***Not found***	*Cladosporium elatum, Penicillium claviforme, Penicillium cyclopium*

Na2SO4—7.0; (3) clinker—100, CaSO4·2H2O 8.6–11,2, NaF—2,0; (4) clinker—100, CaSO4·2H2O—8.6–11.2, NaF–2.0.

3. The efficiency of using biocidal cement composites with an active mineral additive in full-scale conditions of exposure to media characteristic of bacteria and mycelial fungi was confirmed. It was established that composites on the developed compositions within the wide limits of the formulation were more resistant in comparison with materials on ordinary cement in the climatic conditions of the Black Sea coast and Leningrad region.

Table 6 Test results of specimens exposed in the temperate zone

Climatic zone	Specimen exposure conditions	Species composition of microorganisms on the specimens	
		100 wt.h. clinker, 8.6 wt.h. $CaSO_4 \cdot 2H_2O$, 10 wt.h. fly ash, 7.0 wt.h. sodium sulfate	100 wt.h. clinker, 11.2 wt.h. $CaSO_4 \cdot 2H_2O$, 20 wt.h. fly ash, 4.0 wt.h. sodium sulfate
Temperate (St. Petersburg)	In an airy environment under a canopy	–	*Alternaria brassicae, aspergillus ustus, Fusarium moniliforme*
Temperate (St. Petersburg)	In an open air environment	–	***Not found***
	On the open area after aging in groundwater	–	*Aspergillus fumigatus,Aspergillus niger, Botryotrichum piluliferum, Cladosporium elatum, Cladosporium macrocarpum, Fusarium moniliforme, Penicillium claviforme, Penicillium lanosum, Penicillium nigricans, Trichoderma viride*

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