Biodegradation Resistance of Wood-filled Caustic Magnesite Composites

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The article deals with composite materials based on caustic magnesite binder and wood fillers used in the fabrication of various types of objects in mechanical engineering, construction engineering, and oil and gas industries. Under operational conditions, caustic magnesite composites can be exposed to aggressive actions of microorganisms. This study looked into resistance of wood-filled composites upon exposure to byproducts of filamentous fungi (micromycetes). This research substantiated the choice of model medium for testing – byproducts of metabolism of micromycetes. Designed experiments were carried out. The samples were held in model solutions with different concentrations of aggressive medium agents. Lines of equal values of materials' resistance were plotted. It was found from experiments that composites without fillers had a lower biocorrosive resistance compared to those filled with pine sawdust.

DOI: 10.15376/biores.20.3.7738-7749

Keywords: Composite materials; Caustic magnesite; Wood fillers; Caustic magnesite composites; Filamentous fungi; Metabolic byproducts; Matrix of planning; Mass absorption graphs; Bioresistance coefficients

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INTRODUCTION

Solving problems related to the biological destruction of materials and structures, leading to the protection of buildings and structures from biological degradation is an extremely important issue, since almost all building materials are subject to biological damage. Biodegradation is understood as the destruction of materials and disruption of the products performance caused by exposure to macro- or micro-organisms. The research findings show that more than 40% of such damage is associated with the activity of microorganisms: bacteria and filamentous fungi (Startsev 2007; Evseev *et al.* 2009). Bacteria develop when there is an abundant moisture in materials, for example, when the latter are immersed in a liquid. Furthermore, in the presence of moisture, bacteria also give way to fungi, which develop at moisture content above 75%. Statistics show that filamentous fungi have the greatest damaging effect among microorganisms (Svetlov *et al.*

2021; Jiang *et al.* 2022).

The first stage of biological degradation occurs as follows: microorganisms begin to colonize the surface of objects, penetrate deeper and form colonies, and metabolic byproducts accumulate; the structural material deteriorates due to simultaneous exposure to microorganisms, humidity, temperature, and chemical aggressive media (Voitovich *et al.* 2004; Stroganov and Sagadeev 2014). Soluble substances diffuse into the microorganism cell because of different concentrations inside and outside of those substances, while the cell membrane absorbs the necessary elements, blocking the harmful ones. Carbon and nitrogen compounds that are found on the surface of substrates from the external environment or are synthesized by other microorganisms such as bacteria can also serve as food sources for fungi. That is, organic acids produced by microorganisms play a dual role: on the one hand, they form an aggressive external medium, and on the other, being incomplete products of hydrocarbon oxidation, they serve as a carbon source for the further development of microorganisms.

Having highly efficient and diverse enzyme systems, filamentous fungi are able to use both organic and inorganic-based building materials as a food sources (Evseev *et al.* 2009). It is known that biological damage to industrial and building materials can occur not only because of the growth of microscopic fungi on their surface, but also under the influence of certain exometabolites released by them into the surrounding environment (Stroganov *et al.* 2011). Metabolites released by destructor fungi, leading to the decay of materials, also impair their physico-chemical characteristics.

Even relatively fungus-resistant materials are vulnerable when exposed to metabolites of micromycetes formed during the growth of fungi. The material may suffer from biodegradation, and their physico-mechanical characteristics may be impaired (Shafigullin *et al.* 2017). Although these materials have a certain fungal resistance, nevertheless, they can suffer from damage when exposed to aggressive exometabolites (Shafigullin *et al.* 2017; Erofeev and Elchishcheva 2020).

Among the aggressive metabolites secreted by microscopic fungi during their growth on building materials and involved in the destruction of materials, organic acids and hydrogen peroxide play an essential role (Andreyuk and Bilay 1980; Erofeev *et al.* 2016; Erofeev 2016). For example, organic acids released by filamentous fungi during the biodegradation of polymer materials are directly involved in the acid hydrolysis of chemical bonds. The acids also facilitate the acidification of the medium, providing a pH-optimum zone for the action of other aggressive metabolites-exoenzymes. Hydrogen peroxide is also capable of oxidizing various chemical components of building materials, which leads to a change in the chemical composition and structure of the latter (Stroganov *et al.* 1985; Stroganov *et al.* 2009; Bulgakov *et al.* 2016; Travush *et al.* 2017; Erofeev *et al.* 2019).

Up to this point, researchers mainly have assessed bio-damaging processes and developed bio-resistant composite materials based on cement (Rodionova *et al.* 1990; Hastrub *et al.* 2012; Javaid *et al.* 2019), glass-alkali (Erofeev *et al.* 2016; Vilkova 2017), and polymer (Bassil *et al.* 2015; Muthukumar and Veerappapillai 2015; Erofeev *et al.* 2016) binders. Among building materials, a certain niche is given to the use of such materials as caustic magnesite composites, which are used in mechanical engineering, civil construction, and oil and gas industry (Vigdorovich *et al.* 1991; Voitovich *et al.* 2004). Caustic calcinated magnesite (Sorel cement), hydrated with magnesium chloride (bischofite), is more often used as a binder in their fabrication.

Caustic magnesite composites are more widely used in construction engineering and drilling technologies in the manufacture of industrial floors, dry building pre-mixtures, glass-magnesium sheets, grouting mortars for cementing, and for the repair and plugging of oil and gas wells. Products based on caustic magnesite composites are characterized by low shrinkage and resistance to wear.

An important advantage of caustic magnesite binders compared to cement ones lies in the absence of undesirable reactions between caustic magnesite stone and wood fillers. As is known, finely ground wood cannot be used as a filler for mixtures based on Portland cement without special treatment (mineralization). This is because calcium hydroxide formed during the hardening of this binder causes lignin hydrolysis, which leads to the destruction of the microstructure of wood particles (Zaprudnov 2017). Caustic magnesite is inert to wood. Therefore, the use of wood fillers in composite materials has favorable prospects. The raw wood resources are abundant, because forests are continuously renewed, and life on the planet is possible only if there is a forest that provides oxygen and protects people ecologically. Wood waste, the volume of which is at least 30% of industrial wood, can be effectively used in the fabrication of composites (Vigdorovich *et al.* 1991). Therefore, the increased output of composite materials is less expensive if we rely on wood-filled caustic magnesite composites.

When operated as floors, plasters, and materials for filling wells, caustic magnesite composites are susceptible to bio-damage. There has been a lack of studies available on the bio-damage of caustic magnesite composite materials.

Revealing the impact of select fungal metabolites on physical and technical characteristics of caustic magnesite composites is very important in order to predict the intensity of biodegradation among various materials. This will make it possible to assess the possibility of caustic magnesite composites to yield to biological damage and identify the technical characteristics that influence the damage to a greater extent under these conditions. Therefore, the analysis of the mechanism of biodestruction among caustic magnesite composites and the subsequent development of measures to improve bioresistance is a highly relevant problem.

The purpose of this research effort was to investigate in the physico-chemical characteristics of caustic magnesite composites under exposure to filamentous fungal metabolites. Hence, the research objectives included the following:

1. To assess the scope of already-completed research on the biological resistance of caustic magnesite composites.

2. To substantiate the methods of analysis of the metabolic byproducts secreted by microorganisms.

3. To review the variants of filamentous fungi and identify the types of organic acids and hydrogen peroxide released.

4. To establish the biological resistance of composites in a model medium composed of filamentous fungi.

5. Using the methods of mathematical planning of experiments to reveal the most aggressive model medium for running accelerated tests.

6. To prepare a visual interpretation of the experimental results.

The most universal type of damaging factors in relation to constructional materials (both organic and mineral origin) is microbiological destruction. Among the main pathogens of microbiological damage, fungi of the deuteromycete class prevail, including representatives of the genera *Trichoderma*, *Geotrichum*, *Fusaris*, *Scopulariopsis*, *Alternaria*, *Cladosporium*, *Aspergillus*, and *Penicillium*. Micromycetes of these classes readily adapt to environmental conditions due to their active enzymatic system and labile metabolism (Allsopp *et al.* 2004).

The reason for the biodestruction of materials of organic origin is the ability of micromycetes to use organic carbon compounds as nutrients for the metabolism and catabolism. The resulting waste products of microorganisms can actively damage the materials of inorganic nature, causing chemical destruction.

The physiological activity of micromycetes is considerably influenced by a number of factors, the most significant of which are the temperature and pH of the environment, the degree and cyclicity of illumination, and moisture content.

Using the optimal temperature as a value which provides the greatest vital activity, micromycetes are classified as psychrophilic, growing at temperatures in the range from -3 to +10 °C, and mesophilic, for which temperatures from +10 to +50 °C and above are most favorable. In humid moderately-cold climates, fungi of the genera *Aspergillus* and *Penicillium* are most common, but fungi of the *Aspergillus* genus are active at higher temperatures than micromycetes of the genus *Penicillium*.

Undoubtedly, the pH of the environment is of great importance for the vital processes of fungi that cause the biodestruction of constructional materials. Thus, for most representatives of micromycetes, the pH value at the level of 4 to 5 is considered the most favorable, which corresponds to a slightly acidic reaction of the medium.

Research into the damage exerted to constructional materials under the influence of biological factors is complicated by the difficulty in ensuring reproducibility of the composition and activity of the biodestructor. This is because in real operating conditions the biological damage of said materials is impacted simultaneously by fungi contact cells, yeast cultures, bacteria, their spores and byproducts of their metabolism. The main damaging factors in biodestruction are, firstly, the mechanical action of fungal hyphae, and, secondly, the action of their metabolites, where this last factor is the most aggressive. Such metabolites with expressed aggressiveness are organic acids, which act both directly as aggressive agents against constructional materials and as a source of food and further development of fungi, as well as agents for creating an acid environment favorable for their activity. Among the byproducts of micromycetes activity, the following organic acids are important: succinic, oxalic, fumaric, malic, gluconic, lactic, and citric. Moreover, the aggressiveness of various acids of this series varies. It is known (Zaikina and Deranova 1975) that citric, tartaric, and fumaric acids are particularly potent even at sufficiently low concentrations (from 0.09% to 0.4% by weight.).

Previous publications dealing with changes of the total acidity of the culture fluid of a pure culture of the micromycete *Penicillium chrysogenum* (Gurvich *et al.* 2003) demonstrate that in the first eight days of *R. chrysogenum* cultivation, the total acid content increases to 26.17 mg/L, and then it plateaus. Ion exchange and paper chromatography methods were used to identify the *P. chrysogenum* metabolic byproducts. The exposed native *solution* was passed through a KU-2 cation exchange and Duolite C 20 anion exchange column after a 192-h exposure. The purified eluate was then subjected to paper chromatography (normal phase) using different ratios of n-butanol, acetic acid, and water as solvents. It was found that tartaric and citric acids had the largest area of chromatographic zones, and a similar analysis of the culture liquid of *R. chrysogenum* with carbohydrate (sucrose) additives confirmed that the composition of metabolites and their relative concentration practically did not change. Given the above, it is logical to use solutions of the most active metabolite agents as model media, such as organic acids, in particular citric acid, which has an acid dissociation constant value of $K_{a1} = 8.5 \times 10^{-4}$, K_{a2}

= 1.8×10^{-5}, $K_{a3} = 4.7 \times 10^{-7}$, which is comparable to acid dissociation constants of other metabolite acids: for tartaric acid, K_{a1} = 9.1 × 10-4, for malic acid, $K_{a1} = 3.5 \times 10^{-4}$, for succinic acid, $K_{a1} = 6.5 \times 10^{-5}$, and for fumaric acid, $K_{a1} = 2.9 \times 10^{-5}$.

In addition to micromycetes' acidic metabolites, enzymes catalyzing the formation of highly aggressive substances (*e.g.*, oxidases such as aldehyde oxidase, xanthine dehydrogenase, xanthine oxidase, and glucose oxidase) are also significant damaging factors produced by the micromycetes. Catalase and peroxidase are the primary oxidoreductases of filamentous fungi. Micromycetes of the genera *Penicillium, Aspergillus*, *Verticillium*, *Fusarium*, *Alternaria*, *Cladosporium*, and *Helminthosporium* (Krivushina *et al.* 2020) exhibit significant peroxidase activity. The end product of such catalytic reactions is hydrogen peroxide; therefore, it is reasonable to introduce controlled amounts of hydrogen peroxide into model media.

EXPERIMENTAL

The following components were used for the fabrication of caustic magnesite composites: Caustic magnesite from the Satka deposit (South Urals, Russia), grade PMK-75 was used as a binder, an aqueous solution of bischofite ($MgCl_2$) with a density of 1,25 g/cm^3 was used as a hardener, and crushed pine wood was used as a filler. The filler size used was assumed to be in the range of 0.16 to 0.315 mm (size was determined using an Olympus BX51M optical microscope).

The composite fill level was chosen based on the mobility value (mobility is the ability of the construction mixture to spread and fill the mold in which it is placed). For ensuring proper placement of magnesium mixtures in floor coverings this value, according to the (GOST 310.4-81 2003), should be within 11 to 17 cm. Taking into account these requirements, samples with fillers were made with a ratio of bischofite/magnesite equal to 1:1.5. Non-filled composites were used as control samples to the above. The ratio of caustic magnesite and bischofite in this case was assumed to be 1:1.

When preparing composites based on caustic magnesite, the dry components (caustic magnesite and filler) were first thoroughly mixed. After that, a bischofite solution (density 1.25 g/cm^3) was added to the working mixer, and the resultant mixture was mixed until a homogeneous mass. Samples of 1 × 1 × 3 cm in size were formed from the resulting mixture, which were held for 7 days under normal temperature and humidity conditions, after which they were tested for compressive strength and biological resistance. Strength tests were run in accordance with the (GOST 10180-2012 2018). The composites' bioresistance was identified against criteria for changes in the mass content and strength of samples held in a model solution of metabolic byproducts of filamentous fungi in accordance with the (GOST 9.048-89 1989). The composition of the biological environment and the exposure time of the samples were being changed.

RESULTS AND DISCUSSION

When conducting research in a model biological environment, as pure chemicals – possible agents of biocorrosion – various combinations of citric acid and hydrogen peroxide in an aqueous solution were used. To test the developed composite materials in model solutions, media were prepared that imitate the composition of metabolites using reagents qualified as analytically pure. The ratios of the concentrations of the components are shown in Table 1. The approximate intervals of production of these substances by said fungi, according to the references (Erofeev *et al.* 2016), were: for citric acid – 0.02 to 0.2 molar (or 0.1% to 10%); and for hydrogen peroxide – 0.01 to 0.1 molar (or 0.03% to 3%). Taking into account the above intervals, the researchers fabricated the media and analyzed them to reveal such combinations of citric acid and hydrogen peroxide that led to the greatest biodegradative damage of composites prepared with a caustic magnesite binder with various fillers. The problem was being solved using a designed matrix, based on a Kono plan, comprising 9 experiments. In accordance with Kono's plan for multifactor experiments in geometric interpretation, the range of variation of factors is represented by a multidimensional cube, which will be further referred to simply as a cube. For two factors, this cube degenerates into a square. Kono plan experiments are conducted at the vertices of the cube, the midpoints of the edges and the center of the cube. The location of the points of Kono's strategic plan are on the points of the square and the cube. The variable factors were the concentration of citric acid (*X*1) and hydrogen peroxide (*X*2) in the aqueous solution. The experimental plan is presented in Table 1.

Table 1. Experimental Plan

№	Planning Matrix		Work Matrix		Components Content in an Aqueous Solution	
	X_1	X_2	Citric acid (mole)	Hydrogen peroxide (mole)	Citric acid + + H_2O (g)	6% Hydrogen peroxide + + H_2O(g)
1	0	0	0.20	0.10	1.92+88.8	3+57
2	+1	+1	2.00	1.00	19.2+80.8	3+3
3	-1	+1	0.02	1.00	0.19+99.8	3+3
4	-1	-1	0.02	0.01	0.19+99.8	3+597
5	+1	-1	2.00	0.01	19.2+80.8	3+597
6	+1	0	2.00	0.10	19.2+80.8	3+57
7	0	+1	0.20	1.00	1.92+88.8	3+3
8	-1	0	0.02	0.10	0.19+99.8	3+57
9	0	-1	0.20	0.01	1.92+88.8	3+597

Tests of caustic magnesite wood composites filled with pine sawdust with a dispersion of 0.16 to 0.315 mm were carried out. The control samples for the filled composites were those of caustic magnesite stone cured with magnesium chloride, as well as composites with filler.

Table 2. Results of Experiments for Non-filled Caustic Magnesite Composites

№ of Medium Composition	Percent Change in Mass after Exposure for a Period of Time (%)			Change in Resistance Coefficient after Exposure for a Period (rel. units)		
	30 days	60 days	90 days	30 days	60 days	90 days
1	5.37	7.02	8.25	0.52	0.41	0.24
2	–	–	–	0.01	0.01	0.01
3	4.93	6.49	7.50	0.86	0.61	0.35
4	4.56	5.95	8.24	0.25	0.15	0.15
5	1.83	4.59	6.41	0.86	0.61	0.30
6	–	–	–	0.01	0.01	0.01
7	4.42	6.27	8.47	0.50	0.42	0.20
8	5.10	5.56	6.35	0.37	0.14	0.08
9	9.80	5.50	6.17	0.37	0.28	0.14

Table 3. Results of Experiments for Caustic Magnesite Composites Filled with Pine Sawdust in Model Environments

№of Medium Composition	Percent Change in Mass after Exposure for a Period (%)			Change in Resistance Coefficient after Exposure for a Period of Time (rel. units)		
	30 days	60 days	90 days	30 days	60 days	90 days
1	8.65	14.19	16.33	0.84	0.75	0.46
2	–	–	–	0.01	0.01	0.01
3	9.11	12.04	11.53	0.66	0.35	0.18
4	7.66	10.27	12.70	0.19	0.09	0.06
5	5.89	8.81	13.30	0.66	0.35	0.21
6	–	–	–	0.01	0.01	0.01
7	13.66	12.51	13.63	0.59	0.49	0.29
8	8.85	10.47	12.76	0.31	0.20	0.11
9	9.25	11.66	11.83	0.43	0.34	0.16

During the tests, it was found that in the model medium of filamentous fungi, compositions № 2 and № 6 of the model medium had the greatest aggressive effect on all the studied composites (see Tables 2 and 3). In this case, samples of both non-filled and filled composites deteriorated their performance over compared to baseline a short exposure time.

Based on the results, regression equations were obtained for the mass content change (ΔG) and the bioresistance coefficient (Br.c) of composites after exposure in a medium for 90 days.

For non-filled compositions:

$$\Delta G = 6.787 - 2.613 \cdot X_1 - 0.808 \cdot X_2 - 1.418 \cdot X_1 \cdot X_2 - 2.880 \cdot X_1^2 + 1.265 \cdot X_2^2 \quad (1)$$

$$\text{Br.c.} = 0.138 - 0.097 \cdot X_1 - 0.007 \cdot X_2 + 0.083 \cdot X_1 \cdot X_2 - 0.047 \cdot X_1^2 + 0.083 \cdot X_2^2 \quad (2)$$

For compositions filled with pine sawdust:

$$\Delta G = 0.330 - 0.023 \cdot X_1 + 0.007 \cdot X_2 - 0.082 \cdot X_1 \cdot X_2 - 0.210 \cdot X_1^2 + 0.640 \cdot X_2^2 \quad (3)$$

$$\text{Br.c.} = 13.466 - 3.843 \cdot X_1 - 2.007 \cdot X_2 - 3.190 \cdot X_1 \cdot X_2 - 5.653 \cdot X_1^2 + 0.697 \cdot X_2^2 \quad (4)$$

In formulas (1) through (4), variables X_1 and X_2 represent the coded values of the factors citric acid and hydrogen peroxide according to Table 1.

Using the above equations, graphical dependences of mass content change and resistance coefficient of non-filled and filled composites were plotted (Figs. 1, 2, 3, and 4).

The obtained equations (1) - (4) make it possible to determine their maximum and minimum values for the coded values of factors - X_1 (hydrogen peroxide content) and X_2 (citric acid content). Graphs such as Figs. 1 to 4 do not make it possible to fully find the maximum and minimum values of the target functions of the two variables (1) to (4). The results of maximization and minimization were determined on the basis of GlobalSearch global optimization of MATLAB system, and the results are summarized in Table 4.

The results show that composites filled with pine sawdust exhibited higher resistance to degradation when exposed to fungal metabolic products compared to non-filled composites. The graphs indicate that combined exposure to the media leads to even lower values of bioresistance.

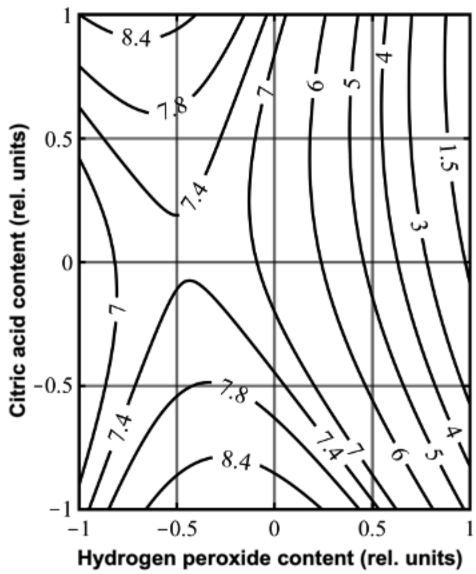

Fig. 1. The relationship between changes in the mass content of non-filledcompositions and the concentration of hydrogen peroxide and citric acid in model biological media

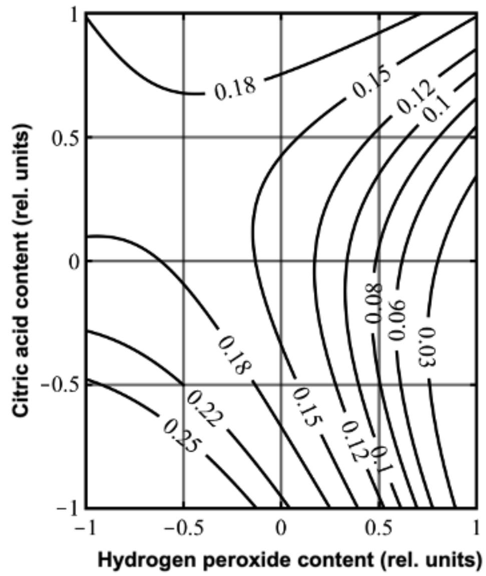

Fig. 2. The relationship between changes in resistance coefficient of non-filled composites and the concentration of hydrogen peroxide and citric acid in model biological media

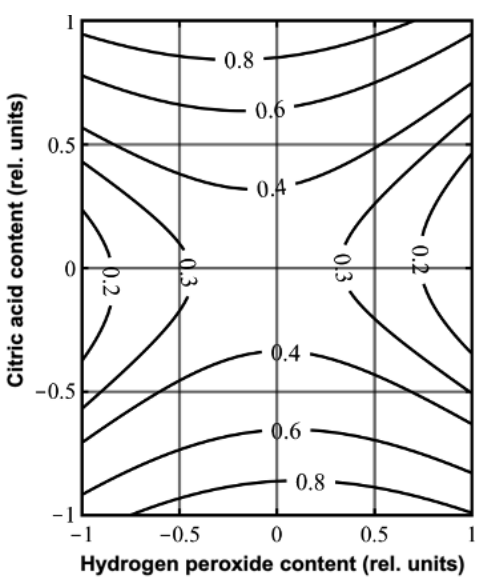

Fig. 3. The relationship between changes in the mass content of filled compositions (pine sawdust 0.315 to 0.63mm) and the concentration of hydrogen peroxide and citric acid in model biological media

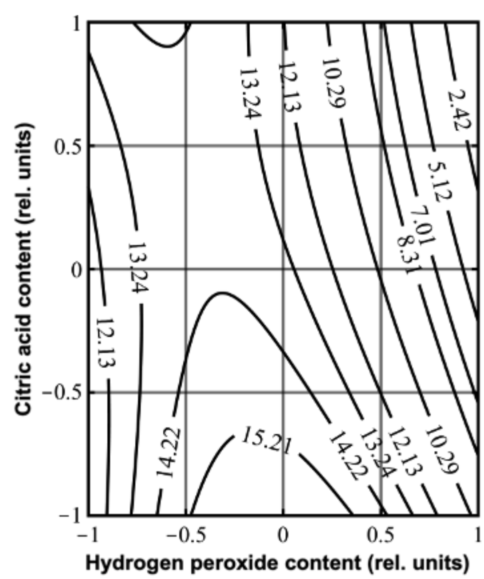

Fig. 4. The relationship between changes in resistance coefficient of filled composites (pine sawdust 0.315 to 0.63mm) and the concentration of hydrogen peroxide and citric acid in model biological media.

The results show that composites filled with pine sawdust exhibited higher resistance to degradation when exposed to fungal metabolic products compared to non-filled composites. The graphs indicate that combined exposure to the media leads to even lower values of bioresistance.

Table 4. Results of Maximizing and Minimizing Objective Functions (1) to (4)

Target Function Number	Maximum of the Objective Function	Minimum of the Objective Function
(1)	8.983961 (X_1 = -0.207465; X_2 = -1.000000)	0.314736 (X_1 = 1.000000; X_2 = 0.879842)
(2)	0.361000 (X_1 = -1.000000; X_2 = -1.000000)	-0.023398 (X_1 = 1.000000; X_2 = -0.457831)
(3)	0.990125 (X_1 = -0.250000; X_2 = 1.000000)	0.094803 (X_1 = 1.000000; X_2 = 0.058594)
(4)	16.188858 (X_1 = -1.000000; X_2 = -1.000000)	-0.530000 (X_1 = 1.000000; X_2 = 1.000000)

The results show that composites filled with pine sawdust exhibited higher resistance to degradation when exposed to fungal metabolic products compared to non-filled composites. The graphs indicate that combined exposure to the media leads to even lower values of bioresistance.

CONCLUSIONS

Strength and bio-resistance evaluations of caustic magnesite composites filled with pine sawdust (0.16 to 0.315 mm particle size) were performed in this study. Bio-resistance was determined by testing the samples in a model medium of micromycetes metabolites, consisting of an aqueous solution of citric acid and hydrogen peroxide.

1. Using mathematical planning of experiments, the concentrations of citric acid and hydrogen peroxide that most severely affected the strength properties of both filled and non-filled caustic magnesite composites were identified.
2. Testing in model media is an effective method for evaluating the durability of materials against biodegradation. Since fungal metabolites are the primary agents of biodestruction, it is reasonable to use model media containing organic acids, such as citric acid, for bio-resistance testing.
3. From the results of analyses of the resistance of different composites exposed to the model media one can conclude the following: model media № 2 and № 6 exerted the most aggressive effect on all tested composites; a similar biodegradation mechanism occurred in both non-filled and filled composites; combined impact of media led to a greater decrease in mechanical strength properties.
4. Comparison of the composites showed that wood-filled composites were characterized by higher bio-resistance than non-filled ones.

ACKNOWLEDGMENTS

The research was funded by the National Research Moscow State University of Civil Engineering (grant for fundamental scientific research, project No 05-661/130).

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Article submitted: January 29, 2025; Peer review completed: March 29, 2025; Revised version received: April 16, 2025; Accepted: July 2, 2025; Published: July 29, 2025.
DOI: 10.15376/biores.20.3.7738-7749