Effect of Wood Fillers on Strength and Biodegradation of Caustic Magnesite

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During their usage, caustic magnesite composites are susceptible to aggressive microbial action. This paper investigated the resistance of wood-filled caustic magnesite composites in a standard filamentous fungi medium. Caustic magnesite composites based on caustic magnesite, filled with wood sawdust from lime, ash, pine, and aspen trees were studied. The compositions were cured using magnesium chloride. The findings showed that composites filled with fine-fraction wood powders exhibited improved strength and resistance properties of caustic magnesite. If this requirement is met, then optimal conditions are created for the formation of an improved matrix in composites with filler and film phase. Tests in the standard medium showed that wood-filled caustic magnesite composites were fungistatic, but not fungicidal. This means that in case of external contamination, wood-filled caustic magnesite composites are susceptible to biodegradation. Tests demonstrated that exposure to the standard fungal medium resulted in an increased mass content and decreased strength of the samples.

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INTRODUCTION

Virtually all construction materials are susceptible to biocorrosion. Biocorrosion is understood as the destruction of materials and the impairment of their performance due to the action of macro- or microorganisms – birds, insects, higher plants, bacteria, and filamentous fungi. Biocorrosion of building materials is related to the activity of microorganisms (Pramanik *et al.* 2024; Fedosov *et al.* 2021; Bone *et al.* 2022). Bacteria thrive in materials with high moisture content, such as when submerged in liquids. Furthermore, under moisture, bacteria give way to fungi, which also develop at humidity levels above 75% (Haile and Nakhla 2010; Berndt 2011; Magniont *et al.* 2011). Statistics show that among microorganisms, filamentous fungi have the most damaging impact (Svetlov *et al*. 2021; Jiang *et al.* 2022).

The first stage of biological degradation is characterised by the following: microorganisms begin to colonize the surface of objects, penetrate deeper and form colonies, metabolites accumulate; bio-damage is stimulated by the simultaneous exposure to microorganisms, humidity, temperature, chemical aggressive media (Svetlov *et al.* 2021). Soluble substances diffuse into the microorganism cell, which is possible due to different concentrations inside and outside of those substances. The cell membrane absorbs the necessary elements, blocking harmful ones.

Having highly efficient and diverse enzyme systems, filamentous fungi are able to use both organic and inorganic-based construction materials as a food source (Jiang *et al.* 2022). It is known that bio-damage to industrial and construction 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 (De Windt and Devillers 2010; Stroganov *et al.* 2011). Metabolites released by destructor fungi, leading to the destruction of materials, impair their physico-chemical characteristics.

Even relatively fungus-resistant materials, when exposed to metabolites of micromycetes formed during the growth of fungi on neighboring materials that are vulnerable to fungal growth material, may suffer from biodegradation, which impairs their physico-mechanical characteristics (Shafigullin *et al.* 2017a,b). Although these materials have a certain fungal resistance, nevertheless, they suffer from damage when exposed to aggressive exometabolites (Erofeev and Elchishcheva 2020; Erofeev *et al.* 2024).

Up to this point, researchers have assessed bio-damaging processes and developed bioresistant composite materials based on cement (Erofeev *et al.* 2016a,b; Travush *et al.* 2017; Al-Dulaimi *et al.* 2024), glass-alkali (Erofeev 2016; Bulgakov *et al.* 2016), and polymer (Erofeev *et al.* 2019) binders. Among building materials, a certain niche is given to the use of such materials as magnesia composites, which are used in mechanical engineering, civil construction, and the oil and gas industry (Vigdorovich *et al.* 1991). Caustic calcinated magnesite (Sorel cement), hydrated with magnesium chloride (bischofite), is more often used as a binder in their fabrication.

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

An important advantage of magnesia binders compared to cement ones lies in the absence of undesirable reactions between magnesia stone and wood fillers. Therefore, the use of wood fillers in composite materials is highly promising. 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 one relies on wood-filled magnesia composites.

When operated as floors, plasters, and materials for filling wells, magnesia composites are susceptible to bio-damage. However, for magnesia composite materials, there are few studies on their bio-damage.

Assessing the biofouling of materials by microorganisms, particularly the impact of filamentous fungi on altering the physical and technical properties of caustic magnesite composites, is crucial for predicting the intensity of biocorrosion in various materials. This will allow evaluation of the susceptibility of caustic magnesite composites to biodegradation and to define the technical characteristics most susceptible to degradation under these conditions. Therefore, analysing the mechanism of biodegradation in caustic magnesite composites and subsequently developing strategies to enhance bio-resistance is a highly relevant issue.

This research paper looks into changes in several physico-chemical properties of caustic magnesite composites under the influence of a standard biological medium consisting of a set of filamentous fungi. The research objectives were as follows: (1) to assess the scope of already-completed research on the biological resistance of magnesia composites; (2) to determine the influence of the type of wood filler and aggregates on the bioresistance and fungicidal properties of caustic magnesite composites in a standard fungal/mycromicetes medium; (3) to conduct comparative tests of the biostability of caustic magnesite composites using inorganic and organic fillers based on various wood species; (4) to elicit data on the compressive strength of caustic magnesite composites after exposure to a standard filamentous fungi medium, and assess the bio-resistance of the composites with regard to the type of wood filler; (5) to develop a method for assessing the quality of construction materials in the form of numerical metric expressed in arbitrary units; and (6) to prepare a visual interpretation of the experimental results.

EXPERIMENTAL

The following components were used for the fabrication of magnesia 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 wood sawdust from pine, linden, ash, and aspen trees were used as a filler. The fraction of fillers was assumed to be less than 0.16 and 0.16 to 0.315 mm.

The composite fill level was chosen based on the mobility value. For ensuring proper placement of caustic magnesite mixtures in floor coverings this value, according to the GOST 310.4-81, should be within 11 to 17 cm. To compare, samples with fillers were made with a ratio of bischofite/magnesite equal to 1/1.5. Non-filled compositions were used as control samples to the above. The ratio of caustic magnesite and bischofite in this case was assumed to be 1/1. Samples were prepared according to Table 1.

Table 1. Test Compositions

Number	Type of Filler	Magnesite	Filler	$MgCl_2$	$MgSO_4$
1	Without any filler	100	–	100	−
2	Pine sawdust, fraction < 0.16 mm	100	100	100	100
3	Aspen sawdust, fraction < 0.16 mm	100	100	100	100
4	Linden sawdust, fraction < 0.16 mm	100	100	100	100
5	Ash sawdust, fraction < 0.16 mm	100	100	100	100
6	Pine sawdust, fraction 0.16 to 0.315 mm	100	100	100	100
7	Aspen sawdust, fraction 0.16 to 0.315 mm	100	100	100	100
8	Linden sawdust, fraction 0.16 to 0.315 mm	100	100	100	100
9	Ash sawdust, fraction 0.16 to 0.315 mm	100	100	100	100

Note: Values represented as mass fraction.

When preparing compositions based on caustic magnesite, the dry components (caustic magnesite and filler) were first thoroughly mixed. After that, a bischofite solution was added to the working mixer, and the resultant mixture was mixed until a homogeneous mass. Samples of 1x1x3 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. The composites' bioresistance was defined against fouling values and changes in the materials' properties after exposure to a standard biological medium in accordance with the GOST 9.049-91. The investigated parameters included changes in mass content and the composites resistance coefficient after exposure to the biological medium.

The samples were exposed to a standard set of filamentous fungi: *Aspergillus oryzae* (Ahlburg) Cohn, *Aspergillus niger* vgn Tieghem, *Aspergillus terreus* Thom, *Chaetomium globosum* Kunze, *Paecilomyces variotii* Bainier, *Penicillium funiculosum* Thom, *Penicillium chrysogenum* Thom, *Penicillium cyclopium* Westling, and *Trichoderma viride* Pcis, ex Fr. The tests were conducted using methods 1 and 3. In method 1, samples were cleaned and inoculated with a water suspension of fungal spores and then incubated under optimal growth conditions for 28 days. In method 3, samples were inoculated with a spore suspension in the presence of a nutrient medium, and incubated for 90 days under optimal growth conditions.

RESULTS AND DISCUSSION

Caustic magnesite-wood composites with compositions listed in Table 1 were tested. The control sample for the filled composites was a magnesium stone composition hardened with magnesium chloride, as were the composites with fillers. Two filler fractions, less than 0.16 mm and 0.16 to 0.315 mm, were used in the experimental analysis. Table 2 presents compressive strength values for the wood-filled composites, both unexposed and exposed to a standard biological medium for 30, 60, and 90 days.

Table 2. Test Results

Number	Compressive Strength (MPa)			
	Control	After Fungal Exposure		
		30 days	60 days	90 days
1	13.80	12.83	9.92	9.00
2	6.62	6.68	8.04	8.42
3	9.42	9.87	10.11	10.28
4	8.68	8.99	9.43	9.83
5	10.00	10.06	10.09	10.12
6	7.85	7.11	7.03	6.90
7	7.77	7.05	5.81	5.25
8	6.63	5.98	5.01	4.38
9	7.83	6.18	4.11	3.38

The optimal filler content and dispersion creates an improved matrix in composites with filler particles and a film phase (Bobryshev *et al.* 2012). This filling, spanning from nano- to microstructures, typically determines the key properties of the resulting material. Adding the filler forms boundary layers and a film-like state of the matrix. The latter exhibits higher density and strength than the bulk matrix phase. It is in the formation of this film structure in the matrix that the composite exhibits increased strength and reduced permeability (Bobryshev *et al.* 2012).

Comparing the strength properties of caustic magnesite composites filled with various wood powders revealed higher values when using ash as a filler. Table 2 also indicates that, in terms of loss of strength and resistance in the standard biological medium, the wood fillers ranked as follows: ash, aspen, linden, pine.

Table 3 presents colonization rates after exposure to the standard medium, and the calculated bio-resistance coefficients for the samples. Figure 1 shows the appearance of the composite specimen after testing. The relative units (rel. units) of the resistance coefficient in Table 3 were obtained by dividing the control values for time exposure by the values of each composition before the tests began. Therefore, the resistance coefficient value for each composition prior to testing is equal to one. Exposure of the samples to a standard medium, using methods 1 and 3, revealed that the caustic magnesite composites were fungus-resistant, but not fungicidal. In case of external contamination, they were susceptible to biodegradation.

Table 3. Bio-resistance of Composites, Depending on the Type of Wood Filler

Number of Composition				Assessment of Fungal Colonization in Points		As per GOST
	30 days	60 days	90 days	1	3	
1	0.93	0.72	0.65	0	4	Fungus-resistant
2	1.01	1.21	1.27	0	3	Fungus-resistant
3	1.05	1.07	1.09	1	4	Fungus-resistant
4	1.04	1.09	1.13	0	4	Fungus-resistant
5	1.01	1.01	1.01	0	4	Fungus-resistant
6	0.91	0.90	0.88	0	3	Fungus-resistant
7	0.91	0.75	0.68	1	4	Fungus-resistant
8	0.68	0.45	0.41	1	4	Fungus-resistant
9	0.79	0.52	0.43	0	4	Fungus-resistant

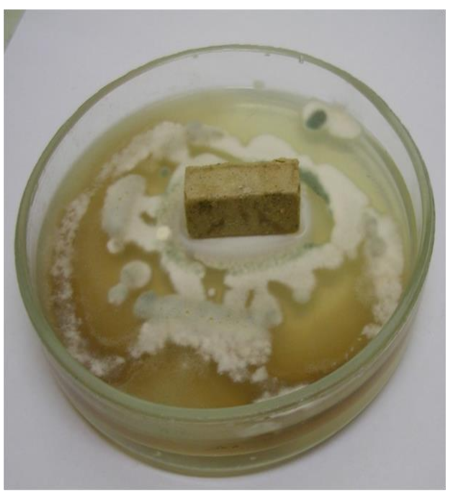

Fig. 1. The appearance of the composite specimen after testing

Method 1 was used to evaluate bio-resistance, with the samples constantly exposed to microorganisms in Petri dishes without a nutrient medium. Table 2 results show both decreases and increases in the strength of caustic magnesite composites when exposed. Comparison of compositions were made easier using a diagram (Fig. 2) in which the diagram's vertices for each composition are connected by a single curve. The strength of the wood-based composites is impacted by the particle size of the filler. The finer the particles the more optimal conditions are created for the formation of an improved matrix in composite structure composed of filler particles and a film phase. Increasing the filler size from less than 0.16 mm to 0.16 to 0.315 mm resulted in a decrease in strength for all types of fillers.

Figure 1 clearly shows that the compressive strength of all compositions, including those with larger filler fractions and the control sample, decreased as a result of exposure to the biological medium. In contrast, the strength of composites filled with wood powders of less than 0.16 mm fraction increased somewhat. What is important is the change in the resistance properties of the wood-filled compositions compared to those materials without any filler (Fig. 3). Given this, diagrams of the biological resistance coefficient for the aforementioned compositions are presented (Fig. 3).

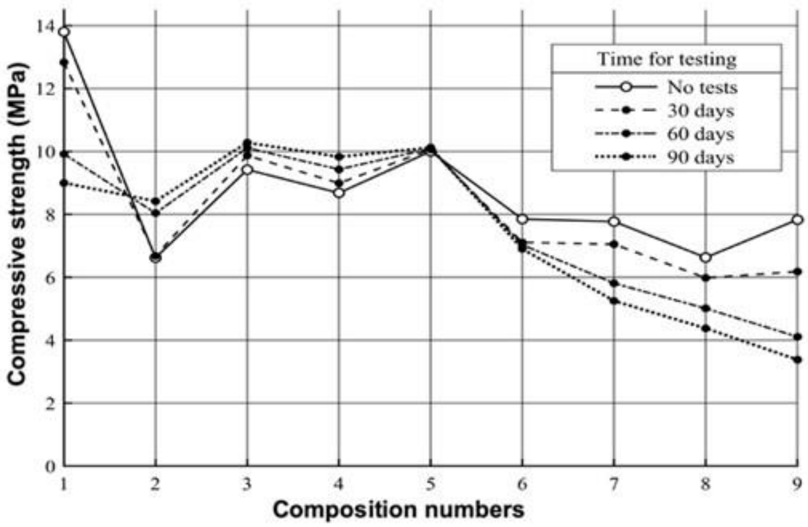

Fig. 2. Change in compressive strength of test compositions

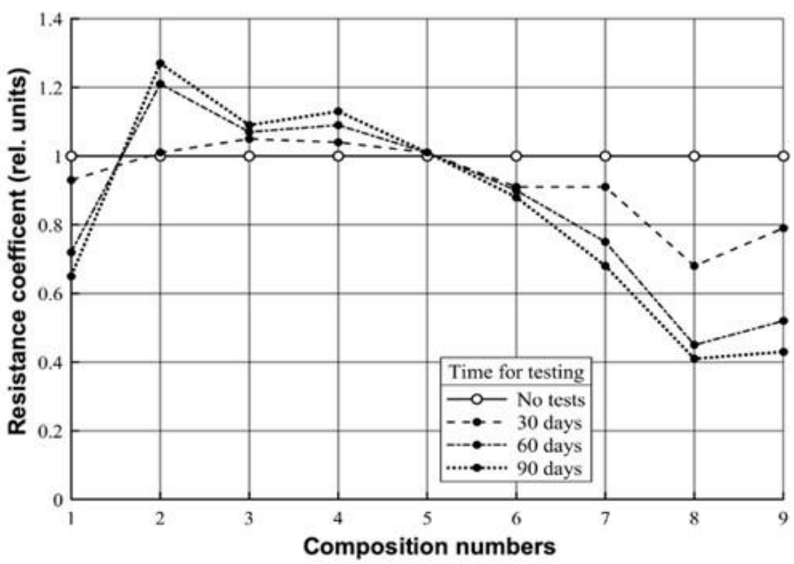

Fig. 3. Change in the resistance coefficient of the test compositions

Figure 3 shows the change in the resistance coefficient of the test compositions. The lower limit in the resistance coefficient is set at 1.0, representing the values for the control samples. The resistance coefficient values for the tested compositions over time were divided by the values for the control compositions at each specific time point. It follows from the diagram that compositions 2, 3, 4, and 5 had a higher bioresistance coefficient compared to composition 1. Wood fillers from pine, aspen, lime, and ash tree with a fine fraction (passed through a 0.16 mm sieve), improved their bio-resistance.

Table 3 and Fig. 3 show that composition 2 shows the greatest increase in the resistance coefficient. Compositions 6 through 9 have lower bio-resistance coefficients compared to the unfilled control samples.

To assess the overall impact on the compressive strength of the compositions contaminated with fungi, it is proposed to introduce an impact metric (K_m) equal to the ratio of the area (S) of the convex hull to the square of the perimeter (P) of the convex hull, as follows.

$$K_m = S/P^2 \quad (1)$$

The convex hull is determined for the compressive strength values or resistance coefficient values of both the control compositions and the compositions exposed to filamentous fungi. Figure 4 shows an example of plotting the convex hull for the compressive strength values of the compositions, based on values in Table 2.

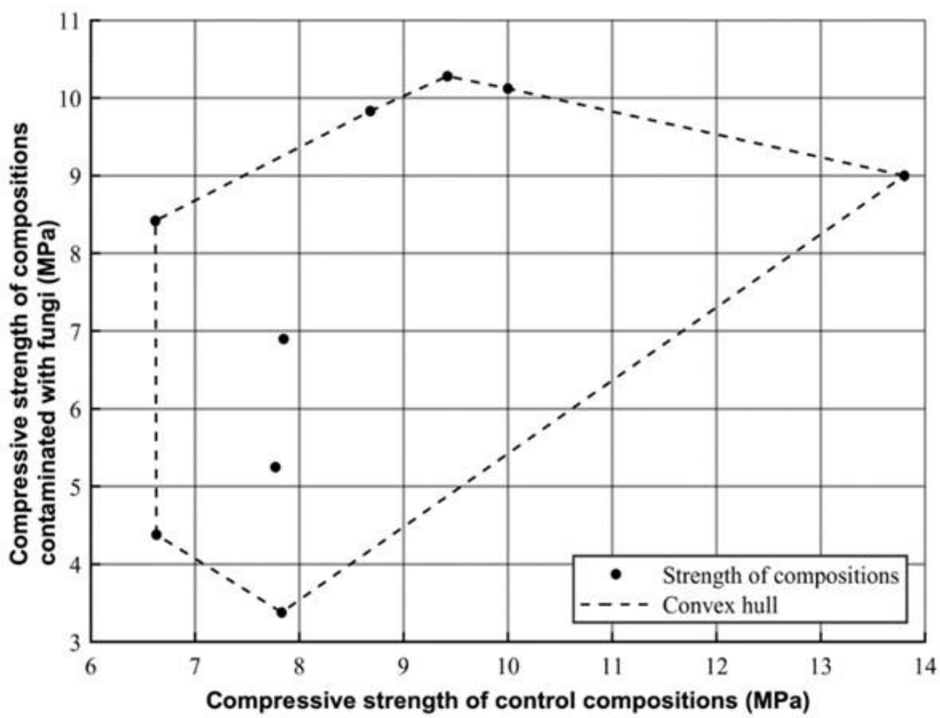

Fig. 4. Convex hull of compressive strength for test compositions

Using Eq. 1 and Fig: 4, the following results were obtained:

- Area of the convex hull after 90 days of testing: 26.807900
- Perimeter of the convex hull: 21.726867
- Impact metric for compression: 0.056790

Figure 5 shows an example of plotting the convex hull for the resistance coefficients of the test compositions, based on values in Table 3.

Using Eq. 1 and Fig. 5, the following results were obtained:

- Area of the convex hull after 90 days of testing: 0.098200
- Perimeter of the convex hull: 1.934173
- Impact metric for resistance coefficients: 0.026249 rel. units

Based on the calculated impact metrics, the strength of the compositions in Tables 2 and 3 are more susceptible to fungal attack compared to the relative changes in the coefficients of the same compositions. Given the relative units (rel. units) of the impact metrics, they are proposed as numerical quality assessments for compositions exposed to various treatments, such as fungal infestation.

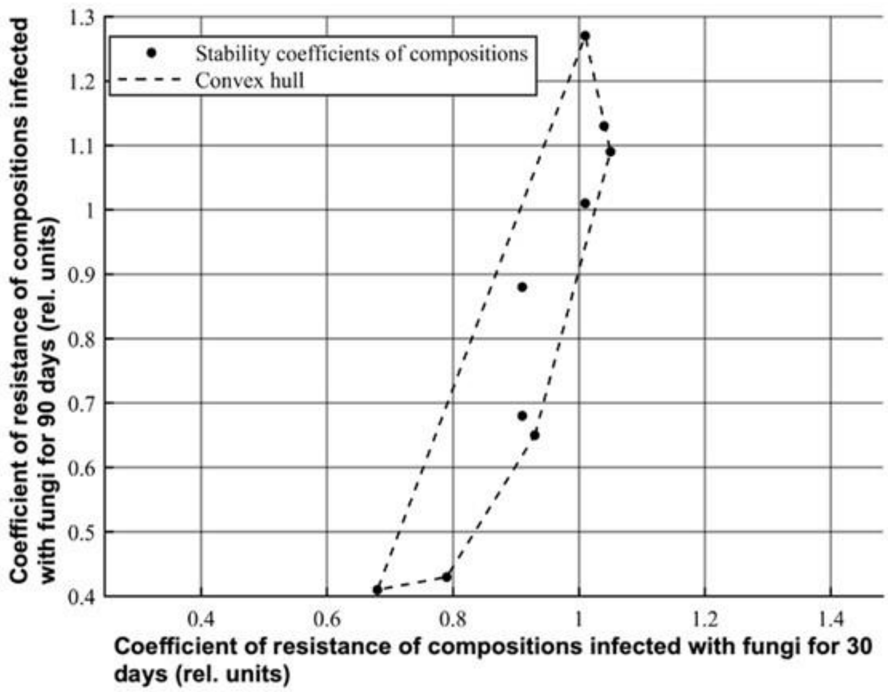

Fig. 5. Convex hull of the resistance coefficient for test compositions

CONCLUSIONS

1. Compared to other building materials, caustic magnesite composites are less studied regarding bio-resistance to microscopic environments and the metabolites they produce. Caustic magnesite composites filled with fine-fraction wood powders exhibited improved strength and bio-resistance. This is because optimal conditions are created for the formation of an improved matrix consisting of filler particles and a film phase.
2. Caustic magnesite composites of various compositions are fungistatic materials, meaning that, in the absence of contamination (nutrients), the material samples do not become colonized by microorganisms. Prolonged exposure of the samples to microorganisms (achieved during testing by continuously injecting microorganisms into Petri dishes containing the samples) leads to a decrease in strength properties.
3. Using method 3, caustic magnesite composites are not fungicidal materials and do not become colonized by microorganisms in the presence of nutritive media. The

calculations and plotting of convex hulls introduced into analysis allow to define in relative metric units of impact the compressive strength and the resistance coefficient for the investigated compositions after exposure to filamentous fungi medium during controlled intervals of time.

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