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# THE USE OF COPPER NANOMODIFIED CALCIUM CARBONATE AS A BACTERICIDAL ADDITIVE FOR CONCRETE

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**Abstract:** Experimental studies of the resistance of cement stone and concrete to the influence of mold fungi have been carried out. The effectiveness of the use of nanomodified calcium carbonate and the PHMG biocide as bactericidal additives for concrete has been investigated. The influence of Glenium and C-3 plasticizers on the effectiveness of the action of bactericidal additives has been investigated. Both investigated biocidal additives showed a fungicidal effect on concrete specimens. The use of nanomodified calcium carbonate as a bactericidal additive for concrete is effective as the use of PHMG biocide. The introduction of this bactericidal additives does not reduce the strength of the hardened concrete.

**Keywords**: concrete, mold fungi, biocidal additives, PHMG, calcium carbonate.

# ПРИМЕНЕНИЕ НАНОМОДИФИЦИРОВАННОГО КАРБОНАТА КАЛЬЦИЯ В КАЧЕСТВЕ БАКТЕРИЦИДНОЙ ДОБАВКИ ДЛЯ БЕТОНА

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**Аннотация:** Проведены экспериментальные исследования стойкости образцов из цементного камня и бетона к воздействию плесневых грибов. Исследована эффективность применения наномодифицированного карбоната кальция и биоцида ПГМГ-ГХ в качестве бактерицидных добавок для бетона. Дополнительно исследовано влияние пластификаторов Glenium и С-3 на эффективность действия бактерицидных добавок. Обе исследованные биоцидные добавки показали фунгицидный эффект на бетоне. Применение наномодифицированного карбоната кальция в качестве бактерицидной добавки для бетона не менее эффективно, чем применение биоцида ПГМГ-ГХ. Введение исследованных биоцидных добавок не снижает прочность затвердевшего бетона.

**Ключевые слова**: бетон, плесневые грибы, биоцидные добавки, ПГМГ, карбонат кальция.

## INTRODUCTION

Biocorrosion is an important problem in modern construction. The growth of organisms such as bacteria, lichens and building materials can negatively effect on the structure of reinforced concrete. Microbes destroy the building material, not only manifesting themselves on its surface. They reduce strength characteristics of reinforced concrete penetrating inside the structure and causing metal corrosion. The destruction of structures by biocorrosion can cause colossal damage and repairing of such structures leads to high financial costs [1,2].

The degree of mold growth depends on environmental factors, material properties and the characteristics of the mold itself. Biocorrosion research aims to predict whether mold will grow on a specific material under favorable conditions, certain humidity and temperature. To prevent the growth of mold fungi, such research must be carried out at the stage of development of new building materials and must be considered in the design, construction and operation of the building. The knowledge gained will help to protect the future building structure from biocorrosion.

The most susceptible building materials to mold are wooden structures. Sugar and starch in the wood are suitable for the growth and reproduction of fungi [3, 4]. But it is necessary to understand that fungal mycelium can become a breeding ground for other fungi or for other microorganisms. The destructive effect of this microorganisms will manifest itself on other materials, in particular on concrete. There are foreign studies of biocorrosion of concrete structures, which show that the most dangerous for cement are sulfur-oxidizing and sulfate-reducing bacteria [5, 6].

Molds reproduct by spores, so it is usual to Cladosporium, Penicillium and Aspergillus to have their spores in the air. Mold fungi can stay on the surface of the material for a long time in the form of spores and do not appear in any way until conditions for their favorable development arise. The most important factor for the development of mold fungi is the humidity of the environment, which depends not only by the climatic features, but also by the operating conditions of the rooms in buildings or by short-term exposure to moisture on the building structure [7, 8]. Indoor mold growth is also harmful to humans and it can cause adverse health effects.

A large number of works are devoted to the study of the biological resistance of cement composites aimed of the effect of the type of binders, fillers and biocidal additives on the mold fouling and resistance of materials to molds [9, 10, 11].

Theme of bacterial protection became especially relevant in 2020, when the epidemic of the SARS-CoV-2 virus had arrived. Scientists were faced with the need to create antibacterial surfaces capable of destroying bacteria [14]. Chinese scientists [12] conducted an analysis of studies of inorganic and organic antibacterial additives, which showed that inorganic additives with metal ions are more effective than organic due to a longer duration of action. It was concluded that the use of antibacterial nanoadditives is a promising way to increase the biostability of building materials [13].

Antibacterial nanoadditives contain nanoparticles of heavy metals in their composition. However, in some cases, the resulting product becomes toxic and unusable that is why the most common metals are silver or copper. Copper and silver nanoparticles show a complete absence of toxic effects on humans [15]; therefore, the use of copper ions is most promising for the development of bactericidal nanoadditives. Copper has not only antibacterial but also antiviral properties. [16,17]. British scientists [18] conducted a study showing that no viable SARS-CoV-2 was found on the copper surface after 4 hours.

Modern nanotechnology makes it possible to increase the resistance of the material to biodegradation that will lead to increasing the durability of the concrete structure.

Carbonate rocks are the basis for the production of building materials. Introduction of carbonate into the composition of concrete does not reduce its strength [19, 20]. Calcium carbonate nanomodified with copper particles can become a promising bactericidal nanoadditive for mortars. This additive is currently used in the production of antibacterial silicone rubber and plastics. The surfaces of materials made with this additive will retain antibacterial properties until the copper nanoparticles are not removed mechanically.

## MATERIALS AND METHODS

The purpose of this experimental study was to determine the effectiveness of the use of nanomodified calcium carbonate as a bactericidal additive for concrete. The effectiveness of the additive was researched through the determination of the resistance of cement stone and concrete to the effect of mold fungi.

For experimental specimens, Portland cement CEM I 42.5 R produced by LLC

«Gornozavodskcement» was used as a binder. Fine-grained river sand sifted to a fraction of 0.315-0.2 mm was used as a fine aggregate for concrete. Plasticizing additives «Glenuim ACE 440» manufactured by LLC «BASF» and plasticizer «C-3» manufactured by JSC «GK Polyplast» were introduced in the dosage recommended by manufacturers into concrete compositions together with mixing water. All experimental compositions had a normal solution consistency according to GOST 310.4-81 standart, because the type of plasticizer influenced to the W/C ratio of solution.

Researched antibacterial additive was a calcium carbonate modified with copper nanoparticles manufactured by GV Holdings Co (South Korea). This additive was used as the main bactericidal nanoadditive for the research. As an alternative bactericidal additive, the additive «PHMG» produced by LLC «Alterkhim-Pro» (Dzerzhinsk, Russia) was chosen. To ensure uniform distribution of the additives in the concrete mixture, they were previously introduced into the mixing water. The number of components by weight is given for 1 kg of cement in table 1.

Concrete specimens were molded in the form of small briquette bars with size dimensions 1x1x3 cm. This size of specimens allows to put them in Petri dishes for testing for fungal resistance and then use to determine the strength of hardened concrete. For the manufacture of formwork these forms were modeled in the Autodesk 3dsmax software (Fig. 1A). The created model was printed (Fig. 1B) on a 3d-printer in the laboratory of Perm Polytechnic University (Perm, Russia). The use of additive technologies made possible to reduce the cost of molds production and increased the accuracy of the concrete specimen. Forming of concrete specimen was carried out in the laboratory of Perm Polytechnic University (Perm, Russia).

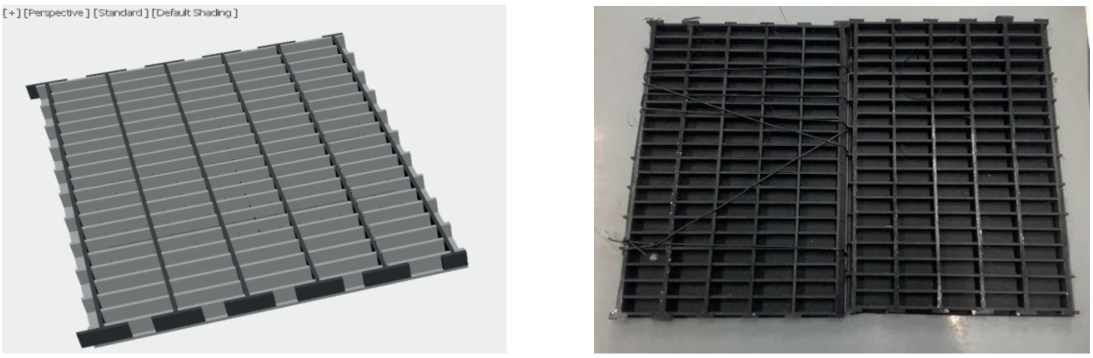



*Figure 1. 3D model of formwork and 3D printed formworks*

*Table 1. Types of experimental compositions*

| № | C, kg | S, kg | W, kg | Plastisizer | | Antibacterial additive | | Type of composition |
|---|---|---|---|---|---|---|---|---|
| | | | | type | mass, kg | type | mass, kg | |
| 1 | 1 | | 0,32 | | | | | Cements stone – no additives |
| 2 | 1 | | 0,32 | | | CaCO3 | 0,005 | Cements stone with 0,5%C CaCO3 |
| 3 | 1 | 3 | 0,34 | Glenium | 0,01 | CaCO3 | 0,005 | Experimental compositions with 0,5%C dosage of additive |
| 4 | 1 | 3 | 0,34 | Glenium | 0,01 | PHMG | 0,005 | |
| 5 | 1 | 3 | 0,36 | C-3 | 0,005 | CaCO3 | 0,005 | |
| 6 | 1 | 3 | 0,36 | C-3 | 0,005 | PHMG | 0,005 | |
| 7 | 1 | 3 | 0,34 | Glenium | 0,01 | | | Concrete – no additives |
| 8 | 1 | 3 | 0,36 | C-3 | 0,005 | | | |
| 9 | 1 | 3 | 0,34 | Glenium | 0,01 | PHMG | 0,02 | Experimental compositions with 2%C dosage of additive |
| 10 | 1 | 3 | 0,34 | Glenium | 0,01 | CaCO3 | 0,02 | |

After 28 days of hardening, the concrete specimens were packed into groups according to the compositions and then transported to the laboratory of microbiological analysis of Lobachevsky University (Nizhny Novgorod, Russia). They were tested for resistance to mold fungi.

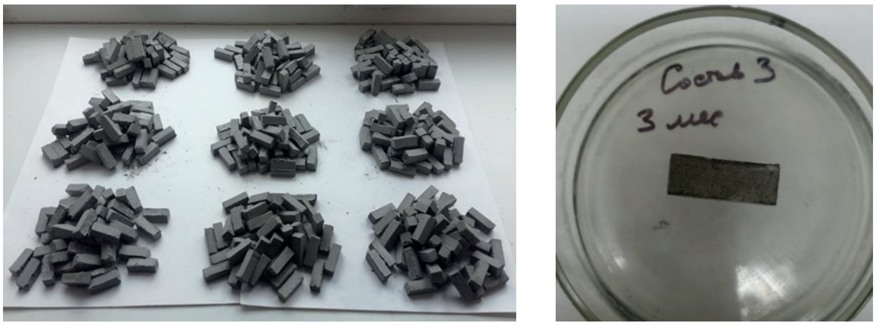



*Figure 2. Concrete specimens before experiment and in Petri dish*

The tests were carried out in accordance to GOST 9.049-91 according to methods 1 and 3. When tested according to method 1, the material is contaminated with mold spores in water and placed in a sterile Petri dish. In this case, molds grow only on the nutrients contained in the material. If the material is not a nutrient medium for molds, then the growth of molds will not occur, therefore, the material is fungus-resistant. When tested according to method 3, the material is contaminated with mold spores and then exposed in a Petri dish with a nutrient medium for fungi. In this case, fungi will grow on the nutrient medium, but if the material is fungicidal, then mold fungi will not grow on it, and as a result of the diffusion of the biocide into the nutrient medium, an inhibition zone is formed in which mold growth will not be observed.

Specimens were exposured in Petri dishes for 1 and 3 months at temperature 28 ± 2 °C and humidity of more than 90%. Thus, the specimens were kept in an environment favorable for the development of molds. 5 concrete specimens were taken for each composition and method.

After the experiment each of the tested groups was packed separately according to the methods and transported to Ogarev Mordovia University (Saransk, Russia), where the specimens were tested for compressive strength.

*Figure 3. Mold fungi growth*

The compressive strength of the samples was determined from the results of testing 5 specimens for each composition. Specimens were placed in a special tooling that allowed the specimens of this size to be tested using standard test equipment. Flexural strength was not determined due to the low strength of the fine-grained concrete specimens caused by their small size. The average compressive strength of the 5 specimens tested is taken to display the result.

## RESULTS

Results according to method 1 testing show that all researched compositions of concrete and cement stone were found to be fungus-resistant. This happened because cement stone and concrete are not a breeding ground for molds, therefore, their growth was not observed on the specimens. But the fungal resistance of a material does not itself guarantee that the material will be protected from mold. During the operation of the concrete structure, various contaminants brought by water and winds settle on the surface of material. Thus, various bacteria, algae and other contaminants can appear on the surface of the concrete and they will already be a breeding ground for mold. They will contribute to biocorrosion and destruction of building material, therefore it is important to provide the building material to be not only fungal resistant, but also fungicidal. That can be determined by method 3 experiment.

Table 2 shows the results of testing specimen according to method 3. The points indicate the degree of mold fungi growth, where R is the size of zone of inhibition in the presence of a fungicidal effect. The introduction of biocidal additives caused a fungicidal effect.

*Table 2. Results after 4 weeks of exposition in mold fungi by methods 1 and 3*

| № | Type: Cem - cement C – conctrete A - additive | Plasticizer | Antibacterial additive | | Degree of mold fungi growth, points | | Result |
|---|---|---|---|---|---|---|---|
| | | | Type | %C | M 1 | M 3 | |
| 1 | Cem | | | | 0 - 1 | 2 | Fungi resistant |
| 2 | Cem + A | | CaCO3 | 0,5 | 0 | 0 (R = 4-5 mm) | Fungicidal |
| 3 | C + A | Glenium | CaCO3 | 0,5 | 0 | 0 (R = 2-4 mm) | Fungicidal |
| 4 | C + A | Glenium | PHMG | 0,5 | 0 | 0 (R = 8-10 mm) | Fungicidal |
| 5 | C + A | C-3 | CaCO3 | 0,5 | 0 - 1 | 0 (R = 1-5 mm) | Fungicidal |
| 6 | C + A | C-3 | PHMG | 0,5 | 0 | 0 (R = 12-15 mm) | Fungicidal |
| 7 | C | Glenium | | | 0 | 0 (R = 1-2 mm) | Fungi resistant |
| 8 | C | C-3 | | | 0 - 1 | 2 | Fungi resistant |
| 9 | C + A | Glenium | PHMG | 2 | 0 | 0 (R = 8-15 mm) | Fungicidal |
| 10 | C + A | Glenium | CaCO3 | 2 | 0 - 1 | 0 (R = 1-4 mm) | Fungicidal |

The degree of mold growth after 4 months of exposure can be observed in photos of Petri dishes with the studied specimens. The most indicative result of the effect of nanomodified calcium carbonate is noticeable on specimens of cement stone.

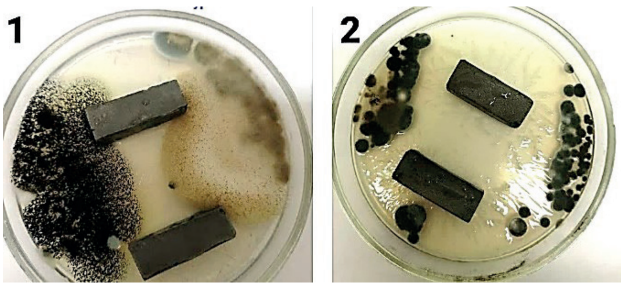



*Figure 4. Degree of mold growth on cement stone: 1 – no additive, 2 – with nanomodified $CaCO_3$*

Photos are shown Specimens with the biocidal nanomodified calcium carbonate show a fungicidal effect with an inhibition zone of 4-5 mm after 1 month of exposure. After 3 months of exposure, the inhibition zone decreased to 2-3 mm, however, the specimens continued to demonstrate fungicidal effect. Compositions No.3- No.10 were a specimens of concrete with a plasticizer. This building material is used in building structures, therefore the results of testing of these specimens for fungal resistance will be more practically applicable. Compositions No.7 and No.8 were prepared to assess the effect of the plasticizing additive on fungal resistance. Fig.5 shows the results of mold growth of concrete specimens made with different plasticizers but without biocidal additives.

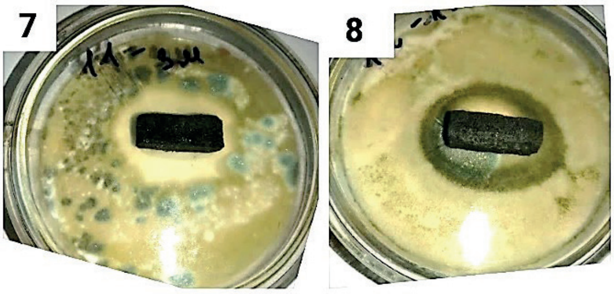


*Figure 5. Degree of mold growth on concrete:*
*7 – plasticizer Glenium ACE 440;*
*8 – plasticizer C-3*

The results show that Glenium ACE 440 polycarboxylate superplasticizer performs much better than C-3 plasticizer. Specimens with Glenium plasticizer showed a negligible fungicidal effect with an inhibition zone of 1-2 mm, while specimens with C-3 plasticizer show a mold growth rate of up to 2 points according to method 3. Concrete with C-3 plasticizer is fungus resistant, but it is no longer fungicidal, like concrete with Glenium plasticizer.

For experimental compositions No.3- No.6, the dosage of biocidal additives was chosen the same and equal to 0.5% of the cement mass (0,5%C). This made it possible to assess the effect of the additive on the fungicidal properties of concrete with a plasticizer. Despite the fact that concrete with a plasticizer Glenium and without a biocidal additive already shows a fungicidal effect, it was important to find out how the joint introduction of biocidal additives and plasticizers will affect the fungicidal properties of concrete. The results in Table 2 show that concrete with both types of additives show a fungicidal effect, but in compositions with PHMG (No.4 and No.6) the zone of inhibition is much larger and reaches 15 mm.

For compositions with nanomodified CaCO3, the inhibition zone does not exceed 5 mm. This difference is also noticeable in the photographs shown in Fig.6 and Fig.7.

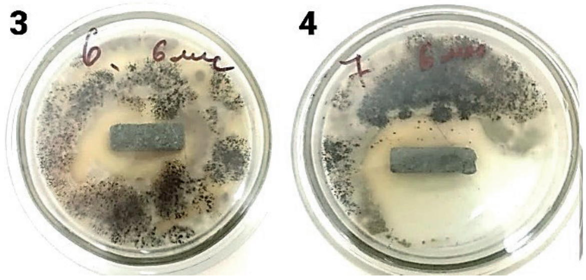


*Figure 6. Degree of mold growth on concrete with a Glenium plasticizer:*
*3 – nanomodified $CaCO_3$ additive;*
*4 – PHMG additive*

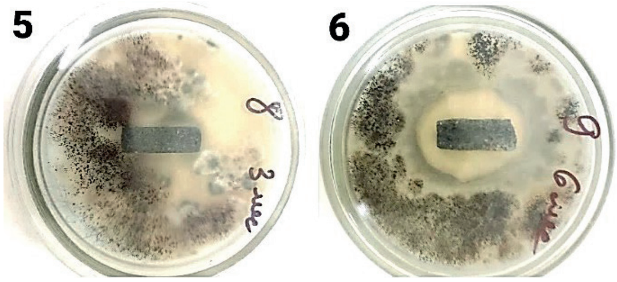


*Figure 7. Degree of mold growth on concrete with a C-3 plasticizer:*
*5 – nanomodified $CaCO_3$ additive;*
*6 – PHMG additive*

For experimental compositions No.9 and No.10, an increased dosage of biocidal additives was chosen. It was equal to 2% by weight of the cement. These 2 compositions were made in order to understand the effect of an increased dosage of a biocidal additive on the fungicidal properties of concrete. Concrete specimen were made with Glenium plasticizer. The results in Table 2 show that increasing the dosage of the biocidal additive does not enhance the fungicidal effect on concrete. The inhibition zone did not exceed 5 mm with an increase in the dosage of nanomodified calcium carbonate. With an increased dosage of the PHMG biocide, the inhibition zone was 8-15 mm, which also corresponds to the result obtained at a dosage of 0.5%. It can be assumed that the increasing of dosage of researched additive does not make sense, since positive results have already been obtained with dosages of 0.5% of additive.

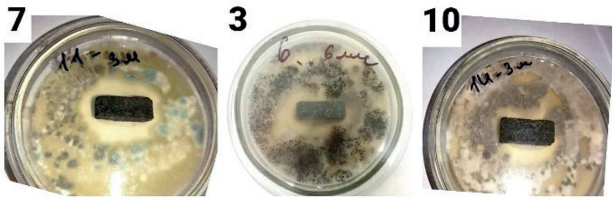



*Figure 8. Degree of mold growth on concrete with a Glenium plasticizer with antibacterial $CaCO_3$ additive dosage: 7 – 0%; 3 – 0,5%C; 10 – 2%C*

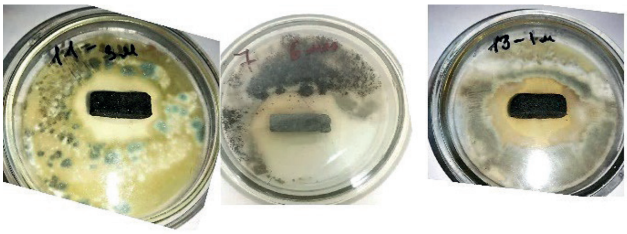
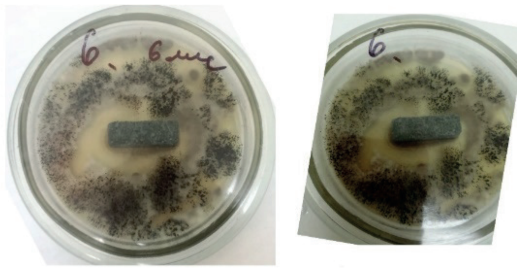


*Figure 9. Degree of mold growth on concrete with a Glenium plasticizer with PHMG additive dosage: 7 – 0%; 4 – 0,5%C; 9 – 2%C*

Fig.8 and Fig.9 clearly demonstrate that the degree of mold growth does not depend on the dosage of the bactericidal additive in the selected ranges of 0.5–2%. For a visual comparison, the figures show the test results of specimen from the composition No.7, where only the Glenium plasticizer was used and no biocidal additives were introduced.

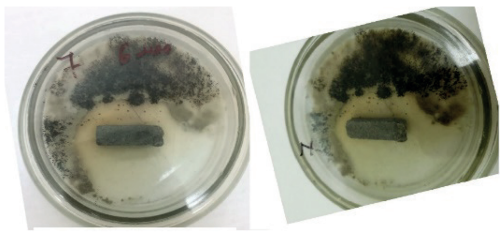
Mold growth rate depends on the exposure time. All of the above results describe the degree of mold growth after 4 weeks of exposure. This time is sufficient to assess the fungicidal properties of the material in accordance to GOST 9.049-91. In practice, mold growth can manifest itself at a much later time, since mold, like any living creature, will adapt to environmental conditions for some time and then grow and develop over time. Fungicidal properties of the material may appear in the form of a zone around the specimen only at the early time of exposure, but later this zone may overgrow, since one species of fungi will become a breeding ground for other type of fungi. In this case, the fungicidal property of the material will not allow mold fungi to cause biocorrosion.

In this study, Petri dishes with specimens of compositions No.3- No.6 were additionally photographed after 3 months of exposure. This allows a visual assessment of the growth rate of molds over time. Fig.10 and Fig.11 show that concrete compositions with Glenium plasticizer and two investigated biocidal additives after 3 months of exposure continue to demonstrate a fungicidal effect. No significant mold growth was observed in the inhibition zone and mold did not grow on the specimens themselves. This allows us to conclude that the nanomodified CaCO3 additive shows the same efficiency as the PHMG biocidal additive even after 3 months of exposure to molds. Fig.12 show that the composition of concrete with the plasticizer C-3 and the nanomodified CaCO3 after 3 months show a fungicidal effect, but there is noticeable overgrowing of the zone after 3 months. If PHMG was chosen as a biocidal additive, even after 3 months, there is a clear absence of additional mold growth after 3 months. It can be concluded that the combined use of PHMG and the Glenium plasticizer is the most effective way for ensuring the durability of a concrete structure to the extent of its resistance to the destructive effects of molds.



*A B*

*Figure 10. Degree of mold growth on concrete with a Glenium plasticizer with antibacterial CaCO3 additive (composition No.3).*
*A – after 1 month, B – after 3 months*



*A B*

*Figure 11. Degree of mold growth on concrete with a Glenium plasticizer with antibacterial PHMG additive (composition No.4).*
*A – after 1 month, B – after 3 months*

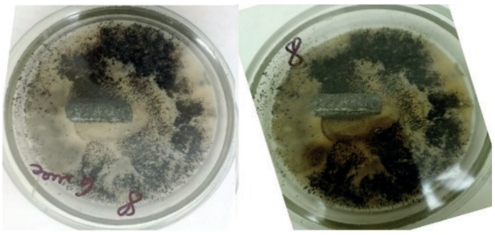



*Figure 12. Degree of mold growth on concrete with a C-3 plasticizer with antibacterial CaCO3 additive (composition No.5).*
*A – after 1 month, B – after 3 months*

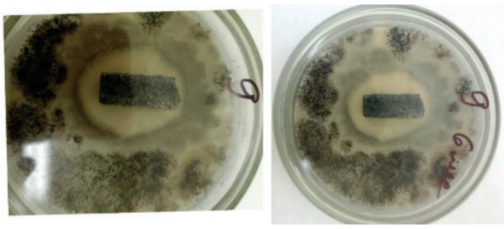



*Figure 13. Degree of mold growth on concrete with a C-3 plasticizer with antibacterial PHMG additive (composition No.6).*
*A – after 1 month, B – after 3 months*

But we should know that PHMG can be toxic for human and cannot be used in construction, that is why nanomodified CaCO3 can be used to protect concrete structure.

Compressive strength is an important characteristic of concrete. Biocorrosion leads to a deterioration of the strength characteristics of concrete structures. The decrease in strength of a structure from the effects of mold can pass slowly and imperceptibly. If it is not detected in time, the destruction of the structure can occur suddenly. In this study, all tested concrete specimens proved to be fungus-resistant after 1 month of exposure. Therefore, a noticeable decrease in the strength of the samples as a result of the destructive effect of molds should not be observed.

For the practical applicability of this study and substantiation of the effectiveness of the use of nanomodified additives, the compressive strength of specimens from all compositions was additionally investigated. Due to small size of the tested specimens (1x1cm), the results obtained are relative, however, they allow to make a comparative analysis.

Specimens were divided into 3 groups. The first and second group of specimens after 28 days of hardening were exposed to mold fungi according to methods 1 and 3, respectively. The third group of samples after 28 days of hardening was left for storage at a temperature of 20 ± 2 °C and a humidity of 60%.

After the experiment on fungal resistance with specimens of groups 1 and 2, specimens of all groups were tested for compressive strength at one day. For compositions No.3- No.6, samples were additionally tested after 3 months of exposure. Fig.14 show the compressive strength results of samples stored in room conditions compared to samples stored in a Petri dish at 28 ± 2 °C and humidity of above 90%.

The increased temperature and humidity during tests according to methods 1 and 3 led to an acceleration of the compressive strength of concrete. That is evident from the results of tests of specimens tested according to method 1, where the compressive strength increased by 3-6 times compared to specimens stored at room temperature.

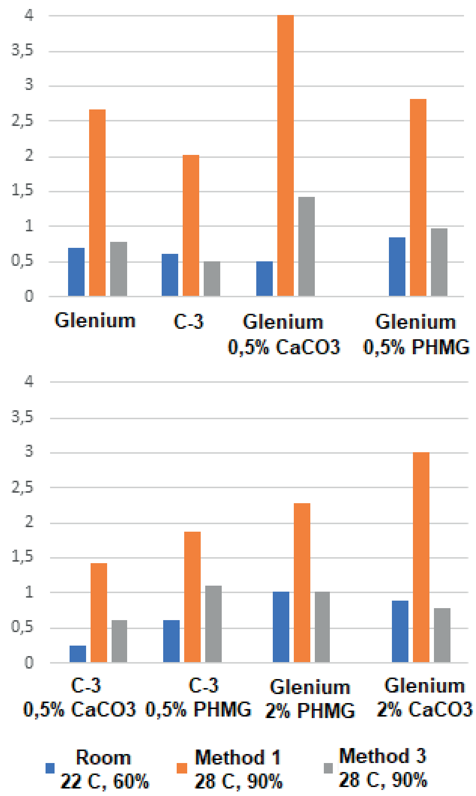



*Figure 14. Relative compressive strength of concrete specimens*

Concrete with C-3 plasticizer has lower compressive strength compared to concrete with Glenium plasticizer. This is clearly seen on compositions with a biocidal additive at a dosage of 0.5%C. Compositions with Glenium plasticizer have 1.2 - 2.5 times greater compressive strength than compositions with C-3 plasticizer. This is observed both on specimens stored at room temperature and on specimens exposed to mold, where much better conditions for the concrete curing were created.
The introduction of biocidal additives together with the Glenium plasticizer leads to a slight increase in the compressive strength of the concrete.
For compositions No.3 - No.6 were obtained results that allow to compare the compressive strength of the specimens after 1 and 3 months of exposure. The results show that the additional increasing of strength of the specimens during the second and third months of exposure is already insignificant and at 3 months of exposure all compositions have the same strength as they had after 1 month of exposure. Glenium plasticizer accelerates early strength development, which is also evident in the results of these tests. Strength diagrams are shown in Fig. 15.

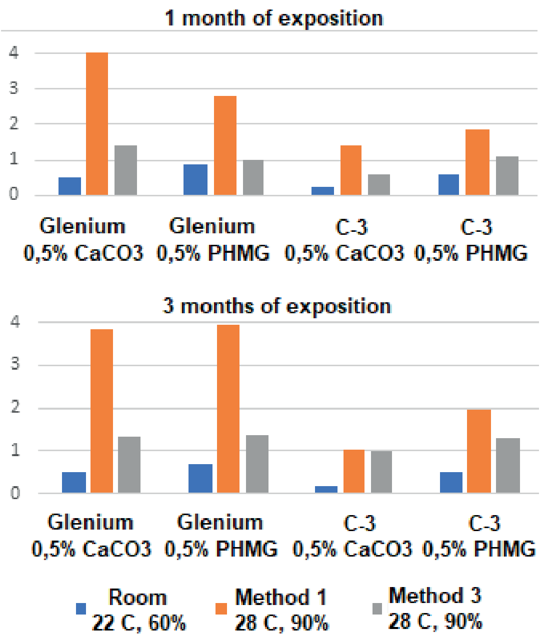



*Figure 15. Relative compressive strength of specimens*

## DISCUSSION

The results of the study show the effectiveness of usage of each type of additive as a bactericidal additive and as a hardening additive.
Cement stone specimens showed a fungicidal effect, so the introduction of nanomodified calcium carbonate into the cement stone can be effective.
Composition of concrete with Glenium ACE 440 plasticizer also showed a fungicidal effect, which can be explained by the high quality of this type of plasticizer. The manufacturer indicates that this plasticizer increases the durability of concrete structures, therefore, it is likely that the manufacturer includes fungicidal additives to the plasticizer, which led to an insignificant fungicidal effect of concrete. Mold already begun to grow up to 2 points on concrete specimens only with C-3 plasticizer and while tested according to method 3.
The introduction of PHMG into the concrete composition gives greater efficiency than the introduction of nanomodified calcium carbonate in the same dosage. This is noticeable from the results of measuring the zone of inhibition. All compositions with PHMG show an inhibition zone of up to 15 mm, while concrete samples with the addition of nanomodified calcium carbonate show fungicidal activity with an inhibition zone of maximum 5 mm.
Increasing of the fungicidal effect is not observed while increasing of the dosage of biocidal additives above 0.5%. Concrete retained its fungicidal properties, but the inhibition zone of the material remained the same size as it was at a dosage of 0.5%. Therefore, increasing the dosage of the biocidal additive above 0.5% is not advisable.
The compressive strength of concrete specimens with Glenium plasticizer is in all cases higher than the strength of specimens made of concrete with C-3 plasticizer. This can be explained by the high quality of the plasticizer and the presence of hardening accelerators in its composition.
The introduction of PHMG slightly increases the compressive strength of concrete specimens,

which corresponds to the manufacturer's description. However, the 2-3 times increase in compressive strength declared by the manufacturer was not achieved. At a dosage of PHMG equal to 0.5% - 2%, the strength of concrete specimens increased by an average of 14-18%.

## CONCLUSION

In this study the resistance to mold fungi was determined for cement stone specimens and samples of concrete with different plasticizing additives. Two types of antibacterial additives were investigated: calcium carbonate modified with copper nanoparticles and the biocidal additive PHMG. All studied specimens were found to be fungus resistant. Concrete compositions with biocidal additives are fungicidal or at least fungistatic for 1 month of exposure.

The introduction of nanomodified additives into concrete may be relevant from the point of view of novelty, however, this study shows that the introduction of the biocidal additive PHMG on the studied concrete compositions gives the same. At the same time, an increase in the dosage of biocides is not advisable since it will only lead to an increase in the cost of concrete. Its compressive strength and biocidal characteristics will remain the same.

In further studies, reduced dosages of biocidal additives and various plasticizing additives for concrete should be considered, since they can affect the biocidal properties of the concrete in different ways. Polymeric materials are actively used in modern construction. The use of nanomodified additives can be effective precisely in them; however, this topic requires further research.

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