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REGULAR ARTICLE

Microbial Impact on Epoxy Nanocomposites in Oil and Gas Transportation Systems

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This article presents the results of an assessment of the effect of microorganisms on epoxy composites intended for the protection of oil and gas transportation equipment. The relevance of the study stems from the need to ensure the long-term service life of protective coatings in biologically active, aggressive environments. Experimental data on the fungal resistance of the tested materials were obtained during their expo-sure to media containing well-known cultures of destructive microorganisms. The spectrum of metabolic products formed after contact with the specimens with aggressive biological media was analyzed, which made it possible to evaluate the degree of their biodegradation. It was established that the composite based on epoxy resin with the addition of a modifier (d-ascorbic acid) exhibits pronounced fungistatic properties. It was proved that the introduction of a dispersed filler in the form of a synthesized aluminium-copper charge into the composite imparts fungicidal activity to the material as well. It was shown that the presence of discrete fibres further enhances the barrier properties of the composite with respect to microorganism penetration. Thus, the developed epoxy composite is characterized by the ability not only to inhibit fungal growth but also to exert a lethal effect on fungi. These properties render the material resistant to biodeterioration over an extended service life. The results obtained allow the investigated composite to be recommended for the reliable protection of parts and products of oil and gas transportation equipment. The application of such a material holds promise for conditions involving constant contact with biologically active, aggressive environments.

Keywords: Nanomodified epoxy composites, Polymeric bandage systems, Thermal stability, Thermogravimetric analysis, Thermal destruction kinetics, Activation energy, Physical interaction, Chemical interaction.

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1. INTRODUCTION

The modern development of gas and oil transportation infrastructure demands materials with enhanced performance characteristics capable of withstanding the aggressive effects of service environments and associated biological factors. Polymer composite materials based on epoxy resins hold a leading position in the creation of protective coatings and structural components for the oil and gas industry. High adhesion to metals, chemical resistance, and relative ease of application make epoxy composites attractive for the protection of pipelines, storage tanks, and auxiliary equipment. However, the long-term service of such materials in contact with soil, industrial water, and petroleum products reveals new aspects of their degradation [1, 2]. One such aspect is biodeterioration – the destruction of polymer materials resulting from the vital activity of microorganisms, predominantly bacteria and fungi. Despite the seemingly "inert" nature of cross-linked polymers, practical experience demonstrates that they can undergo biodegradation under the influence of specific degrading microorganisms. This process becomes particularly critical for gas and oil transportation systems, where leaks caused by the failure of protective coatings may lead to environmental disasters and substantial material losses. Biodeterioration is a multistage process that begins with microbial cell adhesion to the polymer surface and the formation of a biofilm. Subsequently, microbial metabolites, notably organic acids and extracellular enzymes, initiate the chemical destruction of the polymer binder, altering its structure and properties. The presence of fillers and fibers introduces additional factors that can either accelerate or decelerate the biodegradation of the composite. On the one hand, mineral microdispersed fillers reduce the volume fraction of the polymer matrix, thereby potentially decreasing the target for microbial attack. On the other hand, surface relief irregularities on equipment coatings can serve as sites for local microbial adhesion and trigger the destruction process. Discrete fibers, in particular basalt or carbon fibers, create a developed internal surface and may promote the capillary ingress of moisture and microorganisms into the bulk of the composite coating through interfacial defects. However, there is also a positive aspect: fibers are capable of forming a barrier framework that mechanically impedes the propagation of biodeterioration into the material bulk. An analysis of the literature [3-17] indicates that the biostability of

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epoxy composites has been insufficiently studied, especially with respect to naturally occurring microorganisms. The majority of investigations have focused on evaluating resistance to specific defined microorganisms, which does not fully capture the actual pattern of biodeterioration. Systematic data are lacking in the influence of the type and concentration of microdispersed filler on the biostability of epoxy composites under conditions close to those encountered in service. Furthermore, it has not been established how discrete reinforcement affects the kinetics of microbial degradation and the temporal evolution of physical and mechanical properties. It remains unclear whether a synergistic or antagonistic effect exists between the filler and the fibers in determining the bioresistance of the composite. This issue is particularly pressing for the gas and oil transportation sector, where materials are in prolonged contact with soil microbiota and formation waters saturated with microorganisms. An understanding of the mechanisms of biodeterioration will enable the targeted design of epoxy composites with a predictable service life in biologically active environments. Hence, the aim of this work is to evaluate the impact of microorganisms on epoxy composites filled with microdispersed powder and reinforced with discrete fibers. To achieve this aim, it was decided to examine the surface structure after long-term exposure of the materials in commonly encountered model environments. A separate objective is the identification of the isolated degrading microorganisms and the assessment of their enzymatic activity toward the previously developed composites. The findings of the study will provide a basis for selecting optimal epoxy composite formulations to enhance their biostability under the critical operating conditions of gas and oil transportation complex equipment.

2. MATERIALS AND RESEARCH METHODS

2.1 Materials

To form the polymer matrix, the ED-20 grade epoxy resin (ISO 18280), widely used in global practice, was utilised. The epoxy resin has a molecular weight of 390-430 with an epoxy group content of 20.0-22.5 % and a hydroxyl group content of 1.25 %, while the average epoxy functionality is 2.0 [8]. This indicates an increased activity of the side groups and main chain segments of the epoxy resin macromolecules towards homogeneous and heterogeneous interfacial interaction with the groups of modifiers or plasticisers in the compositions, as well as with active centres on the surface of dispersed or fibrous fillers. Additionally, it should be noted that the viscosity of the epoxy resin is 13-20 Pa·s, and its density is 1.16 g/cm^3.

Materials based on such a binder exhibit improved adhesive, physico-mechanical, and thermophysical properties. Together, these factors ensure a high degree of gelation in polymer composites, resulting in enhanced corrosion resistance in aggressive environments, particularly when the materials are subjected to dynamic loads and a broad range of temperature fluctuations. It should be noted that epoxy resins are thermosetting polymers that can be cured at room temperature. Based on this, and considering the industrial technology for forming composites and protective coatings, it was deemed appropriate to use polyethylenepolyamine (PEPA) (CAS 68131-73-7) as the curing agent for the epoxy binder. Polyethylenepolyamine has a molecular weight of 230–250; the viscosity of the curing agent is 0.9 Pa·s, and its density is 1.05 g/cm^3 [8]. The general chemical formula of PEPA is as follows: [– CH_2–CH_2–NH–]$_n$. D-ascorbic acid (DAA) is soluble in water and degrades upon prolonged boiling. It actively participates in redox reactions via hydrogen proton transfer. It exhibits antioxidant properties, thereby ensuring the stability of the properties in homogeneous systems. Furthermore, it influences iron exchange. Specifically, during physicochemical interfacial interactions with active centers on the surface of additives, it can reduce trivalent iron (Fe^{3+}) to divalent iron (Fe^{2+}).

The structural formula and general appearance of the modifier are presented in Fig. 1 and Fig. 2.

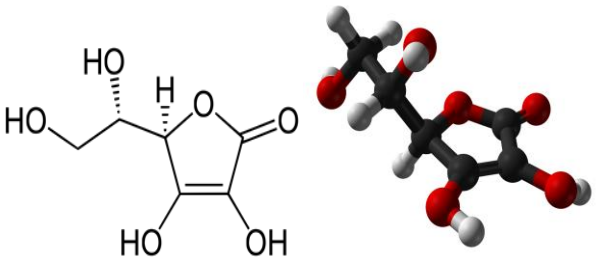


Fig. 1 – General representation of the chemical bonds of the D-ascorbic acid modifier [18]

Fig. 2 – General appearance of the D-ascorbic acid modifier (× 16)

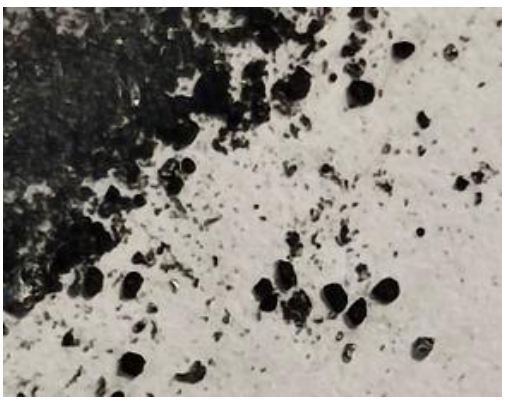
To improve the mechanical and thermophysical properties of the modified epoxy composites, a microdispersed filler was introduced at a low concentration in the form of a synthesized aluminum-copper charge (SACC). It should be noted that this filler was specifically synthesized for this application. To activate interfacial interactions, facilitate the formation of additional high-modulus compounds, and reduce the size of the dispersed particles, high-voltage electric discharge (HVED) synthesis was performed using a custom-developed test bench in a kerosene medium [19]. The precursor material for the HVED treatment was a microparticle mixture with the following component ratio: Al (87.5 %) and Cu (12.5 %). During the synthesis, the accumulated energy of a single discharge (W) was 1 kJ, while the integral specific processing energy (W_{spec}) was 5 MJ/kg.

The authors of [19] demonstrated that following electric discharge synthesis, a reduction in the size of the microdispersed particles to an average diameter of d_{avg} = 13 ± 1 μm is observed. Additionally, their phase composition changes, leading to the formation of high-modulus compounds, specifically $CuAl_2$ and Al_4C_3 (Table 1). The general appearance of the synthesized SACC microdispersed particles after the HVED treatment is shown in Fig. 3.

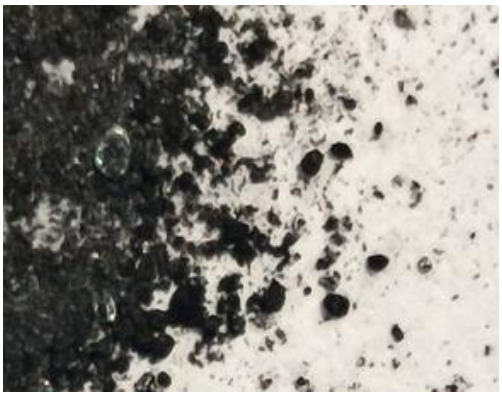
Table 1 – Results of the high-voltage electric discharge (HVED) synthesis of the filler

Initial composition	Post-treatment composition	Electrode system	Diameter after treatment, d, μm		
			d_{min}, μm	d_{max}, μm	d_{avg}, μm
Al (87.5 %) + Cu (12.5 %)	Al (81 %) + Cu (9 %) + $CuAl_2$ (6 %) + Al_4C_3 (4 %)	1	0.2	95	13

To increase the impact strength and plasticity of the composites, discrete fibers (DF) were additionally introduced into the epoxy binder as a filler, comprising the following composition (%): polyester – 52, viscose – 40, and elastane – 8. The dimensions of the discrete fibers were: l = 0.8-1.2 mm, d = 22-26 μm.



a



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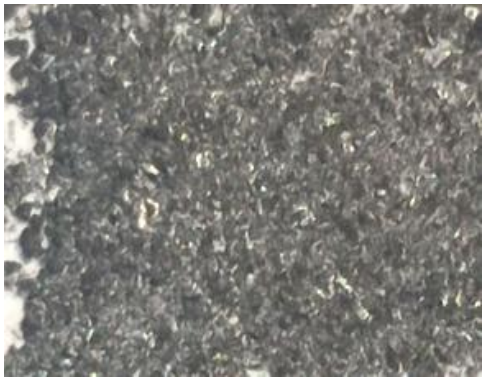

c

d

Fig. 3 – General view of SACC particles (a-d) following HVED treatment (× 16)

2.2 Research Methods

The investigations were conducted in accordance with ISO 846:2019 ("Plastics – Evaluation of the action of microorganisms") [20]. This standard outlines test methods for determining the degradation of plastics caused by the action of fungi, bacteria, and soil microorganisms. The testing protocol involves the inoculation of samples with a spore suspension, incubation of the inoculated samples, evaluation of any physical effects on the specimens, and analysis of the fungal growth dynamics. The scope of the testing and the specific test strains employed depend on the intended application of the plastic. Under specific climatic and environmental conditions, microorganisms colonize the surfaces of plastic products and proliferate rapidly. However, their presence and

metabolic by-products not only degrade the polymer composite but also exert a detrimental effect on the metal components and mechanisms they are intended to protect. The standard specifies methods for evaluating the degradation of plastics caused by fungi and bacteria. Its objective is to determine the biodegradability of plastics and to establish the type and extent of the induced deterioration. Within the framework of this standard, the type and extent of degradation are assessed based on the following criteria:

- visual inspection;
- change in mass;
- changes in physical properties.

The tests conducted in accordance with this standard are applicable to all plastic materials that feature a flat, easily cleanable surface. In accordance with the standard, the following test methods were employed: Method A (fungal growth test) and Method B (determination of fungistatic effects). The experimental procedure for Method A was conducted as follows. Square-shaped specimens with dimensions of 50 ± 1 mm were prepared, with a recommended thickness of 0.5-2.0 mm. The specimens were placed on the surface of an incomplete agar nutrient medium. The agar surface, as well as the test tubes, were inoculated with a spore suspension. A single specimen was placed in each test tube (or Petri dish), and all experimental variants were evaluated using five replicate specimens. Incubation was carried out at 28 ± 1 °C for 28 days. During this period, the tested microorganisms utilized the polymer as a nutrient source for their development. This test involves exposing the material to the test strains of fungi and bacteria under specific temperature conditions for a defined period of time. For Method B, the specimens were placed in Petri dishes containing a nutrient medium and transferred into specialized environmental chambers operating at a temperature of 28 ± 1 °C and a relative humidity exceeding 90%. In accordance with the ISO 846:2019 standard, the following test fungal strains were utilized:

- Aspergillus niger (ATCC 6275);
- Penicillium funiculosum (ATCC 36839);
- Chaetomium globosum (ATCC 6205);
- Trichoderma virens (ATCC 9645);
- Paecilomyces varioti (ATCC 18502).

The specimens were incubated for 3 months. A single specimen was placed in each dish, and all experimental variants were evaluated using five replicate specimens. Changes in mass and the biological resistance coefficient after 1, 2, and 3 months of exposure were utilized as the evaluation criteria. The tests and testing conditions described in the ISO 846:2019 standard are experimental and encompass most, if not all, potential applications. For specific applications and long-term testing, it is necessary to employ procedures that reflect performance under real-world conditions. Primarily, the impact of microorganisms on the investigated materials occurs via two distinct mechanisms:

– direct action: degradation of the polymer, which serves as a nutrient source for microbial growth;

– indirect action: the influence of microbial metabolites, resulting in phenomena such as discoloration or the further deterioration of properties.

3. DISCUSSION OF RESEARCH RESULTS

The ISO 846:2019 standard provides various protocols for evaluating the proliferation of fungi and bacteria, both independently and in combination. The following test methods were selected for this study (Fig. 4.):

- Method A: Fungal growth test;
- Method B: Determination of fungistatic effects.

Method A: Fungal growth test. The prepared plastic specimens are exposed to a mixed fungal spore suspension containing an adequate amount of an incomplete (carbon-free) nutrient medium.

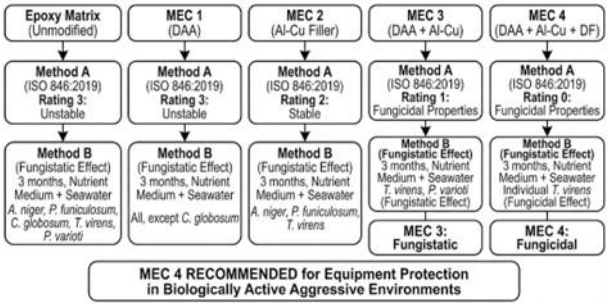

Fig. 4 – Results of assessing microbial action on epoxy composites

The fungi may utilize the polymer as a nutrient source. If fungal growth is observed on the tested specimen, it can be concluded that the polymer exhibits low resistance to fungi, indicating that the material is susceptible to direct fungal attack. Following a predetermined incubation period, one of the following is established: either the prepared specimen contains no nutritive components, or fungal growth occurs on the material, inducing some deterioration of its properties. At the conclusion of the experiment (as detailed in the "Research Methodology" section), the test specimens were evaluated via visual inspection both before and after cleaning, and the changes in mass or other physical properties were determined. If the specimens lack nutrient components, the fungi are unable to form mycelium, and no degradation of the polymer occurs. Evaluation test: The assessment of microbial growth on the surface of the specimens is visual. The rating scale ranges from 0 (no growth) to 5 (substantial growth):

– 0 – no fungal growth observed (under a microscope); thus, the specimen exhibits excellent resistance to fungal growth;

– 1 – germinated spores and the presence of weakly developed mycelia observed (under a microscope);

– 2 – branched mycelia observed (under a microscope);

– 3 – barely visible fungal growth observed (with the naked eye), while being clearly visible under a microscope;

– 4 – fungal growth observed (with the naked eye), covering up to 25 % of the specimen's surface area;

– 5 – fungal growth observed (with the naked eye), covering more than 25 % of the specimen's surface area.

When a score of 0 to 2 is obtained according to the evaluation test criteria, the material is considered resistant to fungi. If a rating of 0 or 1 is assigned, the investigated material is concluded to possess fungicidal properties.

Based on the results of the corrosion resistance tests, materials with the following compositions were selected for the fungal resistance study:

– initial epoxy matrix (ED-20 epoxy resin: PEPA curing agent – 100 : 10);
– MEC 1 (epoxy resin : DAA modifier: curing agent – 100 : 1.5 : 10);
– MEC 2 (epoxy resin : dispersed filler: curing agent – 100 : 2 : 10);
– MEC 3 (epoxy resin : DAA modifier: dispersed filler: curing agent – 100 : 1.5 : 2 : 10);
– MEC 4 (epoxy resin : DAA modifier : dispersed filler: DF filler : curing agent – 100 : 1.5 : 2 : 0.02 : 10).

Table 2 presents the fungal resistance test results for the developed epoxy composites evaluated according to Method A.

Table 1 – Evaluation results of fungal growth dynamics on the surface of the developed polymer composites

Material	Rating according to Method A	Evaluation test results
Epoxy matrix	3	Not resistant to fungi
MEC 1	3	Not resistant to fungi
MEC 2	2	Resistant to fungi
MEC 3	1	Fungicidal properties
MEC 4	0	Fungicidal properties

It can be concluded that the materials based on the initial epoxy matrix and the modified polymer, which received a rating of 3, contain a sufficient amount of nutrients to promote fungal growth. A slight fungal growth was observed. Consequently, based on the evaluation results, these materials are classified as not resistant to fungi.

The material containing the dispersed filler (MEC 2) also contains nutritive substances. However, their quantity is negligible, and fungi were detected only under high magnification and in minimal amounts. Therefore, it can be asserted that this material exhibits resistance to fungi.

The composites containing a combination of the modifier and filler (MEC 3), as well as those additionally incorporating discrete fibers (MEC 4), received ratings of 1 and 0, respectively. It can be concluded that these composites do not serve as a nutrient source for fungi. They are either neutral towards fungi – although germinated spores were observed in some localized areas (MEC 3) – or exhibit fungistatic properties (MEC 4). Overall, these composites are characterized by fungicidal properties. Therefore, they can be recommended for the protection of components and products utilized in biologically active aggressive environments.

Method B: Determination of fungistatic effects.

The prepared specimens are exposed to a mixed fungal spore suspension containing a nutrient medium (with a carbon source). Even if the polymer lacks nutritive substances, fungi can grow on the specimens, and their metabolic by-products – produced from the nutrient agar medium – can attack the material. Prior to this, the surfaces of the specimens were inoculated with an aqueous suspension of the test fungi (*Aspergillus niger*, *Penicillium funiculosum*, *Chaetomium globosum*, *Trichoderma virens*, *Paecilomyces varioti*) by applying it evenly using an atomizer. Subsequently, the specimens were placed in Petri dishes containing a nutrient medium (seawater) and incubated for three months. Any inhibition of growth on both the polymer composite and the nutrient agar medium (zone of inhibition) indicates either a fungicidal effect (death of the fungus) or fungistatic activity (cessation of fungal growth and reproduction).

An analysis of the fungal species that developed on the surfaces of the specimens revealed the following. The specimen made of the initial epoxy matrix is characterized by a broad spectrum of fungi that proliferated both within the bulk and on the surface of the polymer. Specifically, the presence of all tested fungal species was identified: *Aspergillus niger*, *Penicillium funiculosum*, *Chaetomium globosum*, *Trichoderma virens*, and *Paecilomyces varioti*. In the specimen containing the modifier (MEC 1), all the aforementioned fungal species are similarly present, with the exception of *Chaetomium globosum*. The MEC 2 material exhibited the presence of the fungi *Aspergillus niger*, *Penicillium funiculosum*, and *Trichoderma virens*.

Specimens MEC 3 and MEC 4 merit specific attention. Specifically, the MEC 3 specimen is characterized by the presence of the fungi *Trichoderma virens* and *Paecilomyces varioti*, whereas MEC 4 exhibits only *Trichoderma virens*. Furthermore, their concentration is relatively low. It can be asserted that the MEC 3 material, due to the presence of the modifier and dispersed filler, is characterized by a fungistatic effect; that is, these ingredients prevent the growth and reproduction of almost all tested fungi. In contrast, the MEC 4 material additionally incorporates discrete fibers, which induces a fungicidal effect. This fungicidal activity manifests in the death of the fungi, as confirmed experimentally by both methods.

Thus, based on the conducted experiments, information regarding the biological resistance of the developed materials to common fungi was obtained. The obtained results enable the prediction of the materials' behavior during chemical and biological corrosion processes. The spectrum of microbial metabolites was identified following the exposure of the specimens to aggressive environments, which allowed for the determination of the extent of their fungal resistance. It has been proven that the MEC 4 material, which contains the epoxy resin, DAA modifier, dispersed filler, and BDF filler, is fungistatic. It is also characterized by fungicidal properties. Such a composite can be recommended for the protection of components and products utilized in biologically active aggressive environments.

4. CONCLUSIONS

Information regarding the biological resistance of the developed materials to common fungi was obtained. The spectrum of microbial metabolites was identified following the exposure of the specimens to aggressive environments, which allowed for the determination of the extent of their fungal resistance. It has been proven that the material containing an epoxy resin, a modifier (D-ascorbic acid), a dispersed filler (synthesised aluminium-copper mixture), and a filler in the form of discrete fibres is fungistatic. It is also characterised by fungicidal properties. The developed epoxy nanocomposite MEC 4 (ED-20 + 1.5 phr D-ascorbic acid + 2 phr Al-Cu charge + 0.02 phr discrete fibres + PEPA) is characterised by both fungistatic and fungicidal properties.

It does not serve as a nutrient source for fungi and remains stable under biologically active, aggressive conditions. Therefore, it is recommended for use as a protective coating for components and equipment in oil and gas transportation systems, especially in environments with constant contact with soil microbiota, formation waters, and other biologically aggressive media. Future work should focus on long-term field tests under real operating conditions (e.g., buried pipelines, offshore platforms) and on evaluating the stability of the fungicidal effect after mechanical damage or prolonged thermal cycling.

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Оцінка дії мікроорганізмів на епоксидні нанокомпозити для обладнання газо-нафтотранспортного комплексу

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У статті наведено результати оцінки дії мікроорганізмів на епоксидні композити, призначені для захисту обладнання газо-нафтотранспортного комплексу. Актуальність дослідження зумовлена необхідністю забезпечення тривалої експлуатації захисних покриттів в умовах біологічно активних агресивних середовищ. Експериментально отримано інформацію щодо грибостійкості досліджуваних матеріалів під час їх експозиції у середовищах, що містять загальновідомі культури мікроорганізмів-деструкторів. Проаналізовано спектр продуктів метаболізму, які утворюються після контакту зразків з агресивними біологічними середовищами, що дозволило оцінити ступінь їх біодеструкції. Встановлено, що композит на основі епоксидної смоли з додаванням модифікатора (d-аскорбінової кислоти) проявляє чітко виражені фунгістатичні властивості. Доведено, що введення до складу композиту дисперсного наповнювача у вигляді синтезованої алюміній-мідної шихти забезпечує матеріалу також і фунгіцидну дію. Показано, що присутність дискретних волокон додатково посилює бар'єрні властивості композиту щодо проникнення

мікроорганізмів. Таким чином, розроблений епоксидний композит характеризується здатністю не лише пригнічувати ріст грибків, але й чинити на них летальну дію. Зазначені властивості роблять матеріал стійким до біопошкоджень впродовж тривалого часу експлуатації. Отримані результати дозволяють рекомендувати досліджений композит для надійного захисту деталей та виробів газо-нафтотранспортного обладнання. Застосування такого матеріалу є перспективним для умов постійного контакту з біологічно активними агресивними середовищами.

Ключові слова: Наномодифіковані епоксидні композити, Полімерні бандажні системи, Термічна стабільність, Термогравіметричний аналіз, Кінетика термічного руйнування, Енергія активації, Фізична взаємодія, Хімічна взаємодія.