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Assessment of the Durability of Epoxy Composites in a Model Environment Mycelial Fungi

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Abstract:

The object of research is to determine the degree of durability of polymer epoxy-based composite samples under the influence of mycellial fungi metabolitic products. Such products have high degree of destructive activity as well as ability to adapt to materials with various chemical compositions. Asbestos-based fillers were used to create durable epoxy-based polymer composites. To characterize microbiotic durability rating the following methods were used. Experimental data were used to construct a mathematical model of the durability of epoxy composites. The regression equation is used as an objective function to find the global maximum of the resistance coefficient of the studied composites. The results for composites with fillers are compared with a control sample without filler. The composition of model environment to influence epoxy-based polymer compositions was determined. The realization of planning matrix provides mathematical of durability for modified epoxy-based polymer compositions. The specific effects of micromycete fungi were determined depending on the content and type of filler. Asbestos-based fillers of various nature derived from industrial waste were shown to be effective in creating durable epoxy-based polymer compositions.

1 Introduction

The object of the study is the biological stability of epoxy composites filled with fine construction waste (FDCW) in the form of ground slate, fibrous chemical waste (WCP), and quartz sand.

Biological corrosion is a determining factor in the reliability and durability of buildings and structures [1-9]. The damage from bio-damage accounts for more than 5% of the world's gross output. Microorganisms play a significant role in biological destruction: mycelial fungi and bacteria.Microbial colonization of the surface of structures is accompanied by a change (destruction) of its structure as a result of direct exposure to microbiota – mainly through the products of their metabolism or under the influence of chemically active substances formed as a result of the interaction of microbes and pollutants.

Of the microorganisms, mycelial fungi have the most significant damaging effect on industrial and building materials, the high destructive activity of which is due to the ability to adapt to materials of different chemical nature, which is primarily due to the presence of a well-developed and mobile enzyme complex. In this regard, it is of interest to study the behaviour of epoxy composites in the environment of various types of microorganisms [6]. According to the Russian State Standard GOST 9.049-91 «Unified system of protection against corrosion and aging. Polymer materials and their components. Methods of laboratory tests for resistance to the effects of mold fungi» [7].

There is a wide range of technological methods that make it possible to purposefully control the structure and, consequently, the properties of epoxy composites, one of which is the use of fillers of various nature and fractional composition [10-21]. Fillers participate in the formation of the microstructure of the matrix base and the contact zones of the composite. However, the use of certain types of finely ground additives can lead to a heterogeneous structure of the composite. According to the statistical processing of the experiments carried out, the global optimization problem is formulated and solved on the basis of the regression equations obtained, the result of which is determined by the values of the factors at which the maximum resistance coefficient is achieved.

The purpose of this study is an optimal assessment of the coefficient of resistance of polymer composites according to the data of a multifactorial experiment. To achieve this goal, the following tasks were solved:

– conducting experiments with test polymer composites;
– coding of the actual values of the factors affecting the resistance coefficient of the studied polymer composites;
– determination of the experiment planning matrix and experimental response function;
– according to the data of a multifactorial experiment, the determination of the regression equations / objective functions (the coefficient of resistance of polymer composites);
– formulation and solution of the global optimization of the objective function of three variables;
– ranking of the studied polymer composites according to the maximum resistance coefficient.

2 Materials and Methods

Exposure to microorganisms can cause changes in the strength characteristics of building materials and products [10-12], [14-21]. In this regard, the biological resistance of, for example, building composites is of interest. Studies of the biological resistance of epoxy composites filled with fine-dispersed construction waste were carried out in the form of ground slate, fibrous waste of chemical production, as well as quartz sand. Equally, mobile mixtures were made, while the amount of hardener was fixed and remained constant throughout the experiment.The tests were carried out according to GOST 9.049-91 by two methods 1 and 3 [7]. Their essence is to withstand materials infected with mould fungi spores in conditions optimal for their development, followed by an assessment of the fungal resistance and fungicidal properties of the samples. In the first case, without the presence, and in the second – with the presence of a nutrient medium for microorganisms.As a characteristic for determining the microbiological resistance of materials, their fouling with microscopic fungi was considered, which was established 14 days after the start of the experiment.The assessment of the fungus resistance of the products was carried out on a six-point scale: 0 is when viewed under a microscope, the growth of mold fungi is not visible; 1 is when viewed under a microscope, sprouted spores and slightly developed mycelium are visible in the form of non-branching hyphae; 2 is when viewed under a microscope, mycelium is visible in the form of branching hyphae, sporulation is possible; 3 is when viewed with the naked eye, the growth of fungi is barely noticeable, but clearly visible under a microscope; 4 is when viewed with the naked eye the growth of fungi is clearly visible and covers up to 25% of the test sample; 5 is when viewed with the naked eye, the growth of fungi covering more than 25% of the surface is clearly visible.The material is considered to be fungal-resistant if it receives a score of 0-2 points according to method 1 and has fungicidal properties if there is a zone of no fungal growth around the sample on the nutrient medium or fungal growth is observed on the surface and on the edges of the samples, estimated by 0 and 1 points. The compositions accepted for testing are given in Table 1.

Table 1. The effect of fillers on the fouling of epoxy composite materials

n/a	The composition of the composite in mass parts	The degree of mushroom growth in points according to the method		Characteristics according to GOST9.049-91
		1st	3rd	
1	Unfilled composition	3	5	Non-fungus resistant
2	Composition based on quartz sand (100)	3	5	Non-fungus resistant

3	Composition based on FDCW (100)	2	4	Mushroom resistant
4	WCP-based composition (100)	1	3	Mushroom resistant
5	Composition based on quartz sand (60)	3	5	Non-fungus resistant
6	WCP-based composition (60)	0	1	Mushroom resistant

The probability of biological damage to polymer materials is due to the impact of microbial metabolic products on them. In the process of their vital activity, microorganisms secrete various acids, water and enzymes that have a negative effect on various materials. At the same time, it was revealed that the main processes of destruction of various materials are caused by the action of acids: from organic, both polyatomic (humic, pyruvic), and simpler in structure (acetic, lactic, tartaric, oxalic, malic, citric), to inorganic.It has been established that such acids as oxalic and citric can accumulate fungi in large quantities (up to 10%). The maximum production of acids by mushroom cultures is observed at temperatures up to 40 °C and low pH values of the medium. According to [10], [12], the environment of mycelial fungi during research can be modelled with an aqueous solution of organic acids and hydrogen peroxide.The determining factor in the process of spreading microorganisms and their waste products is the diffusion of culture fluid into the structure of the building material. We have conducted studies to establish the influence of the type of filler and the composition of an aggressive medium that simulates the effect of mycelial fungi on the resistance of epoxy composites in terms of changes in mass content and hardness on the surface of samples. The samples were exposed in model environments for 90 days.The compositions of unfilled and filled epoxy composites were used as the material for the study. Portland cement, chrysotile asbestos, as well as FDCW and WCP were used as fillers. Combinations of citric and oxalic acids, as well as hydrogen peroxide with a concentration of up to 5%, were used as suspected agents of chemical corrosion caused by exposure to the waste products of mycelial fungi.

As an optimized indicator, the resistance coefficient (*Rc*) was taken according to the change in hardness.The experiment was implemented using methods of mathematical planning of experiments [22, 23]. A symmetrical plan in a cubic planning area containing 13 observation points is selected. As variable factors were assigned: X1 is concentration of citric acid; X2 is concentration of oxalic acid; X3 is concentration of hydrogen peroxide. The planning matrix and the working matrix are shown in Table. 2. The results of the experiment are shown in Table 3.

Table 2. Planning matrix and working matrix of the environment modelling the effects of mycelial fungi metabolism products

Experience No.	Planning matrix of coded factors			Working matrix characterizing the percentage of components in an aqueous solution		
	X1	X2	X3	citric acid	oxalic acid	hydrogen peroxide
1	0	+1	+1	2.5	5	5
2	+1	0	+1	5	2.5	5
3	–1	0	+1	0	2.5	5
4	0	–1	+1	2.5	0	5
5	+1	+1	0	5	5	2.5
6	–1	+1	0	0	5	2.5
7	0	0	0	2.5	2.5	2.5
8	+1	–1	0	5	0	2,5
9	–1	–1	0	0	0	2.5
10	0	+1	–1	2.5	5	0

11	+1	0	–1	5	2.5	0
12	–1	0	–1	0	2.5	0
13	0	–1	–1	2.5	0	0

Table 3. Dependence of the change in the resistance coefficient on the change in the hardness of epoxy composites in aggressive model media

Experience No.	Resistance coefficient, conl. units.					
	Without filler	Portland cement	serpentinite	FDCW	WCP	quartz sand
1	1.15	1.14	1.36	1.00	1.44	1.17
2	1.05	1.20	1.42	1.22	1.25	0.63
3	1.39	1.20	1.25	1.16	1.51	1.29
4	0.96	1.17	1.37	1.40	1.29	0.98
5	1.02	1.04	1.43	1.13	1.09	0.82
6	0.90	1.40	1.14	0.92	1.05	0.63
7	1.02	1.04	1.33	1.21	1.30	0.99
8	0.50	1.48	1.56	1.32	1.54	1.00
9	1.83	1.66	1.26	1.33	1.35	1.23
10	0.63	1.15	1.44	1.13	1.20	0.88
11	0.50	1.41	1.49	1.08	1.28	1.11
12	0.93	1.37	1.27	1.31	1.30	0.97
13	0.97	1.38	1.36	1.34	1.29	1.02

Fig. 1 can give an idea of the change in the resistance coefficient.

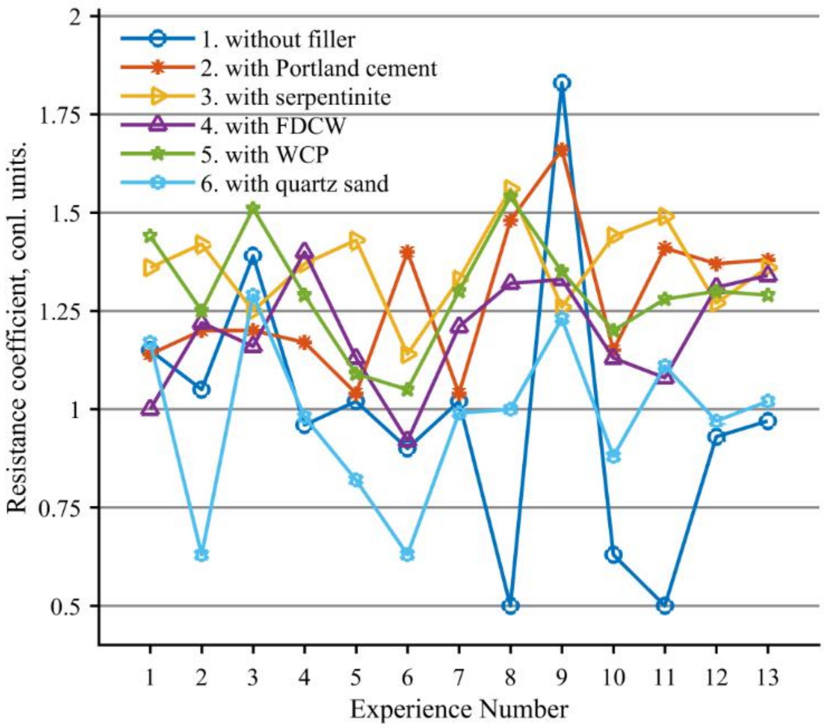

Fig. 1- Diagram of changes in resistance coefficients

As can be seen from Fig. 1 or data from Table 3, there are noticeable changes in the coefficient of resistance from experience to experience, as well as from the filler of a particular composite.

Based on the analysis of the results of Table 3, we will compare the resistance of filled compositions with the resistance of unfilled composites, taking into account typical kinetic regimes [23].

- For epoxy composites without filler, the highest resistance is noted in model media No. 2,5, 9; the least high resistance in model media No. 3,6, 10. In other media, the value of resistance takes intermediate values.
- For epoxy composites with Portland cement filler, compared with epoxy composites without filler, the resistance changes as follows: it decreases sequentially in wet environments №№ 13, 1,4, 2, 5, 3; slightly higher in model environments No. 6, 10, 12; does not change significantly in model environments No. 7, 8, 11.
- For epoxy composites with serpentinite filler, compared with epoxy composites without filler, the resistance changes as follows: decreases in model environments №№ 13, 3, 9, 7, 8, 6, 1, 12, 5, 4, 2; consistently increases in model environments No. 10, 11.
- For epoxy composites with filler from FDCW, compared with epoxy composites without filler, the resistance changes as follows: it decreases sequentially in model environments №№ 3, 5, 1, 4, 6, 2, and in model environment, No. 13, the decrease in resistance is associated with weight loss; it does not change significantly in model environments №№ 12, 7, 10, 11, 9, 8.
- For epoxy composites with WCP filler, compared to epoxy composites without filler, the resistance changes as follows: it decreases sequentially in model environments №№ 13, 7, 11, 4, and in model environment No. 3, the decrease in resistance is associated with weight loss; it does not change significantly in model environments №№ 1, 2, 5, 6, 8, 9, 10, 12.
- For epoxy composites with quartz sand filler, in comparison with epoxy composites without filler, the resistance changes as follows: it decreases sequentially in wet environments №№ 6, 9, 1, 3, 5, 2, 7, and in model medium No. 4, the decrease in resistance is associated with weight loss; it increases sequentially in model media No. 11, 10; it does not change significantly in model media No. 8, 12, 13.
- For epoxy composites without filler, the durability is consistently increased in model environments №№ 8, 11, 10, 6, 12, 4, 13, 5, 7, 2, 1, 3, 9. The minimum value of the resistance coefficient is 0.5 for media No. 8 and 11; the maximum is 1.83 for medium No. 9.
- For epoxy composites with Portland cement filler, compared with epoxy composites without filler, the resistance changes as follows: exceeds the resistance of composites without filler and consistently increases in model environments №№ 7, 5, 2, 4, 13, 12, 6, 10, 11, 8; the durability of composites without filler is less and consistently decreases in model media No. 1, 9, 3.
- For epoxy composites with serpentinite filler, compared with epoxy composites without filler, the resistance changes as follows: exceeds the resistance of composites without filler and consistently increases in model environments №№ 1, 6, 7, 12, 2. 13, 5, 4, 10,11, 8; the durability of composites without filler is less and consistently decreases in model media No. 3, 9.
- For epoxy composites with filler from FDCW, compared with epoxy composites without filler, the resistance changes as follows: exceeds the resistance of composites without filler and consistently increases in model environments №№ 6, 5, 2, 7, 13, 12, 4, 10, 11, 8; the durability of composites without filler is less and consistently decreases in model media No. 1, 3, 9.
- For epoxy composites with filler from WCP, compared with epoxy composites without filler, the resistance changes as follows: exceeds the resistance of composites without filler and consistently increases in model environments №№ 5, 3, 6, 2, 7, 1, 13, 4, 12, 10, 11, 8; less durability of composites without filler in model medium No. 9.
- For epoxy composites with quartz sand filler, compared with epoxy composites without filler, the resistance changes as follows: exceeds the resistance of composites without filler and consistently increases in model environments №№ 4, 1, 12, 13, 10, 8, 11; less durability of composites without filler and always decreases in model environments №№ 3, 5, 6, 2, 9.

The analysis of the obtained results allows statistical processing of the experimental results in order to get analytical regression equations describing the dependences of the resistance coefficient (*Rc*) of epoxy composites on the type of filler and the composition of the model medium with organic acids and hydrogen peroxide.The calculated dependencies are summarized in Table 4.

Table 4.Computational regression equations

No. 1	For composites without filler
	$Rc = 1.020 + 0.247X_1 + 0.070X_2 - 0.190X_3 + 0.041X_1^2 + 0.363X_1X_2 + + 0.023X_1X_3 + 0.001X_2^2 + 0.132X_2X_3 - 0.094X_3^2;$
No. 2	For composites filled with Portland cement
	$Rc = 1.040 + 0.025X_1 + 0.157X_2 + 0.075X_3 + 0.182X_1^2 + 0.030X_1X_2 - - 0.010X_1X_3 + +0.097X_2^2 + 0.050X_2X_3 + 0.072X_3^2;$
No. 3	For composites filled with serpentinite
	$Rc = 1.330 - 0.122X_1 + 0.023X_2 + 0.020X_3 - 0.004X_1^2 - 0.003X_1X_2 - - 0.013X_1X_3 + +0.021X_2^2 - 0.022X_2X_3 + 0.031X_3^2;$
No. 4	For composites filled with FDCW
	$Rc = 1.210 - 0.004X_1 + 0.151X_2 + 0.010X_3 - 0.030X_1^2 + 0.055X_1X_2 + + 0.073X_1X_3 - 0.005X_2^2 - 0.047X_2X_3 + 0.012X_3^2;$
No. 5	For composites filled with WCP
	$Rc = 1.300 + 0.006X_1 + 0.086X_2 - 0.052X_3 - 0.006X_1^2 - 0.037X_1X_2 - - 0.060X_1X_3 - 0.036X_2^2 + 0.060X_2X_3 + 0.041X_3^2;$
No. 6	For composites filled with quartz sand
	$Rc = 0.900 + 0.070X_1 + 0.091X_2 - 0.011X_3 - 0.041X_1^2 + 0.105X_1X_2 - - 0.200X_1X_3 - 0.029X_2^2 + 0.082X_2X_3 + 0.051X_3^2.$

The regression equations given in Table 4 are proposed to be considered as objective functions of three variables, followed by a search for the maximum of the Rc resistance coefficients depending on the combination of coded or actual factors. Then the optimization problem can be formulated as

$$Rc(X_1, X_2, X_3) \Rightarrow \max; \ \ =1 \le X_1 \le +1, \ \ =1 \le X_2 \le +1, \ \ =1 \le X_3 \le +1. \qquad (1)$$

The problem of optimizing the composition and properties of composites is quite relevant [25-27]. In this regard, an attempt is also made here to optimize the coded/valid factors at which the resistance coefficient of the form (1) reaches its maximum.

As can be seen from Table 4, the objective function of each regression equation is twice continuously differentiable under given constraints on variables. Since the objective function is a function of three variables, it is impossible to visualize it. Therefore, it is necessary to turn to the methods of global optimization [28], [29]. In its numerical calculations of the search for the global maximum Rc, the MATLAB Global Search method/function (R2010a and higher) was used. Global Search Method performs a global minimum of a function of several variables under a variety of variable constraints.The Global Search method performs a global minimum of a function of several variables under a variety of variable rules. To find the maximum of the objective function, it is enough to change the sign in front of it. This will allow us to determine the variables (factor values) at which the minimum of the objective function taken with the inverse sign is achieved. Subsequently, before the found minimum value, it will be necessary to replace the sign of the resulting number.

For the transition from coded factors to real ones, the following relations are applied [22]:

$$\begin{gathered} dMax = \max(Xreal), \\ dMin = \min(Xreal), \\ dM = (dMax - dMin)/2, \\ dC = (dMax + dMin)/2, \\ Xd1 = X_{\max}(1) \cdot dM + dC, \\ Xd2 = X_{\max}(2) \cdot dM + dC, \\ Xd3 = X_{\max}(3) \cdot dM + dC, \end{gathered} \qquad (2)$$

whereXrealis all the values of the actual factors of the working matrix from Table 2, *Xd*1, *Xd*2, *Xd*3 are the actual values of the factors at which the maximum resistance coefficient of the corresponding composite is achieved.

Thus, to solve the optimization problem, numerical values of the planning matrix of coded factors and numerical values of the working matrix of Table 2 are required, as well as the type of regression equations from Table 4 with the constraints specified in the conditions of the optimization problem (1). To move to the fundamental factors, the relations (2) are used.

Determination of numerical values of resistance coefficients makes it possible to rank them depending on the type of filler. In addition, it is also possible to determine the percentage of change in the resistance coefficient of composites with fillers in relation to the composite without filler.

The listed operations are entirely algorithmic, and therefore their software implementation is possible for an automated search of optimal solutions in accordance with the task (1) based on the GlobalSearch library function.

3 Results and Discussion

In accordance with the proposed approach to solving the problem of optimizing (maximizing) the resistance coefficient *Rc* of polymer composites, the results presented in tables 5 and 6 were obtained, in which the designations of factors that maximize the objective function (1) are denoted as X1max, X2max, X3max. They display the values of the coded and real factors separated by a slash.

Table 5. Results of maximization of resistance coefficients

Composites	X1max	X2max	X2max	Rc, conl. units
1. For composites without filler	1.000/5.000	1.000/5.000	–0.186/2.035	1.745
2. For composites filled with Portland cement	1.000/5.000	1.000/5.000	1.000/5.000	1.718
3. For composites filled with serpentinite	–1.000/0.000	1.000/5.000	1.000/5.000	1.537
4. For composites filled with FDCW	1.000/5.000	1.000/5.000	1.000/5.000	1.425
5. For composites filled with WCP	–1.000/0.000	1.000/5.000	1.000/5.000	1.484
6. For composites filled with quartz sand	1.000/5.000	1.000/5.000	–1.000/0.000	1.366

Table 6.Ranking of composites by the relative value of the resistance coefficient ΔRc

Composites	X1max	X2max	X2max	ΔRc, %
1. For composites without filler	1.000/5.000	1.000/5.000	–0.186/2.035	0.000
2. For composites filled with Portland cement	1.000/5.000	1.000/5.000	1.000/5.000	1.562
3. For composites filled with serpentinite	–1.000/0.000	1.000/5.000	1.000/5.000	11.933
5. For composites filled with WCP	–1.000/0.000	1.000/5.000	1.000/5.000	14.970
4. For composites filled with FDCW	1.000/5.000	1.000/5.000	1.000/5.000	18.350
6. For composites filled with quartz sand	1.000/5.000	1.000/5.000	–1.000/0.000	21.731

In Table 6, the numerical values of Rc are determined based on the data in Table 5 by the formula

$$\Delta Rc = \left(1 - \frac{Rc_{k\max}}{Rc_{1\max}}\right)100\%, \qquad (3)$$

where k = 2, 3, 4, 5, 6.

Formula (3) determines the percentage of deviation of the maximum values of the resistance coefficients of composites with fillers relative to the resistance coefficient of the composite without filler, which is indicated under number 1.

4 Conclusions

1 Based on the data obtained during the tests, the features of fouling of samples with cultures of micromycetes depending on the type of filler were revealed. It has been established that, compared with the samples filled with quartz sand, the compositions filled with asbestos-containing waste (FDCW and WCP) showed fungi-resistant properties. The susceptibility of epoxy composites in the presence of external contamination according to method 3 was also revealed. In this case, it is important to reduce the permeability and increase the durability of the composites when kept in the metabolic products of bacteria and filamentous fungi.

2 The composition of the model medium for assessing the resistance of epoxy polymer concretes in the metabolic products of filamentous fungi, which includes various concentrations of citric and oxalic acids, as well as hydrogen peroxide, in an aqueous solution, is substantiated. The implementation of the experiment planning matrix made it possible to obtain mathematical and graphical dependences of the change in the mass content and resistance of composites during exposure to the environment on the percentage of acids and hydrogen peroxide in an aqueous solution of an aggressive environment.

3 The problem of maximizing the durability coefficient for each composite participating in experimental studies with their subsequent ranking, both in terms of the conditional value of the durability coefficient and in relation to its change in comparison with the composite without filler, has been solved.This approach can be extended to other cases of studying the properties of polymer/cement composites.

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