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ANTI-CORROSION POLYMER COATINGS FOR VEHICLES PROTECTION

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Summary. Epoxy diene oligomer ED-20, polyethylene polyamine PEPA hardener, powdered synthesized iron/titanium carbide mixture with dispersion of 10...12 μm, and the filler of plant origin with dispersion of 0.4...0.6 μm were used to form anticorrosive coatings. The investigation of the change in the value of resistivity and specific capacity in the diesel fuel environment was carried out, taking into account the rational ratio of differently dispersed fillers in the epoxy binder. The decrease in the resistivity of protective coatings by 1.5...1.6 times relative to the epoxy matrix was achieved. At the same time, the correlation with the value of the capacitance, which decreases by 1.8...2.0 times, respectively was established. Additionally, visual analysis of the surface of the developed coatings was carried out. They were kept for 6552 h in the river water at variable temperatures – $T = 263...293 \pm 2$ K. For coatings containing the rational combination of two fillers, no defects in the form of cracks, peeling, and swelling were observed.

Key words: epoxy binder, adhesion, filler, mathematical design of the experiment, corrosion, defect analysis.

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Statement of the problem. Corrosion destruction is a serious problem in the operation of machine parts and mechanisms of vehicles. Corrosion can cause damage and loss of functionality of metal structures and vehicle parts, resulting in the need for repair or replacement of elements. This represents a significant economic cost, especially in industries where special materials or expensive equipment are used. Localized corrosion is particularly dangerous because it can cause significant weakening of the metal in limited areas of the surface (even when a small surface area is affected). This can result in the appearance of cracks, chipping or peeling of the material layer, which affects the strength and reliability of the units or vehicle surfaces as a whole [1–3].

Analysis of the available investigation results. In order to counteract the above mentioned negative factors, the application of epoxy-based composite coatings is important [3–6]. While choosing epoxy composite protective coatings and their components, a number of factors, particularly: the ability of the coating to adhere reliably to the metal substrate; stability of its properties at elevated or low temperatures; type of environment with which the coating will be in contact (fuels and lubricants, acid solutions, salts, alkalis, river or sea water) should be taken into account [4–6]. Therefore, it is important to choose ingredients that will make it possible to improve adhesion and cohesive strength. Their combination will increase the anti-corrosion properties.

The objective of the paper is to determine the optimal content of two fillers with different physical and chemical nature to ensure improved anticorrosive properties of epoxy coatings.

Materials and methods of the investigation. Epoxy resin ED-20 was used to form epoxy composites. For the epoxy binder crosslinking, cold hardening solidifier polyethylene polyamine (PEPA) was used.

The following fillers were used to improve adhesion strength and corrosion resistance:

- synthesized powdered iron-carbide-titanium charge (ICTC) with $d = 10...12 \mu\text{m}$ dispersion was used. The final fraction of the filler was produced by high-voltage electrodischarge (HVED) synthesis according to the method described in papers [5, 6]. The starting material is the mixture of powders with the following initial composition: Fe (75 %) + Ti (25 %). After HVED treatment, the following composition was obtained: 70% Fe + 5% Ti + 20 % TiC + 5 % Fe₃C.

- filler of plant origin (FPO), dispersion $d = 0.4...0.6 \mu\text{m}$. The additive is obtained as the result of thermal decomposition of pressed plant material.

The formation of epoxy composite materials was carried out according to the methods given in papers [7, 8].

In order to optimize the content of fillers, orthogonal central compositional planning by STATGRAPHICS® Centurion XVI application package was used. The content of the main filler was chosen based on the previous results of adhesive properties [9] of epoxy composites investigations. The choice of the additional filler is related to its availability and cost in Ukraine.

The corrosion resistance of the protective coatings was determined by two methods. The first method involved experimental investigations under laboratory conditions, and as the result the change in the resistivity and specific capacitance of samples over time under the influence of aggressive environment was analyzed. RCL meter type E7-22 was used to measure the resistance and capacitance of protective coatings. The device was connected to the measuring cell where samples in the form of coatings applied to the metal base were placed. For 30 days at temperature $T = 293 \pm 2 \text{ K}$, the resistivity and specific capacitance of the coatings were measured, and their values were calculated by the following formulas:

$$\rho = \rho \cdot S, \text{ Om}\cdot\text{m}^2; c = c \cdot S, \text{ n}\Phi/\text{m}^2; S = \pi D^2/4, \text{ m}^2 \quad (1)$$

To obtain the average values of the coatings resistance and capacitance, 5 samples with 20 cm² working area were used.

When investigating corrosion resistance of the developed coatings in the laboratory, diesel fuel that meets the requirements of Euro 5 EN 590 with the following characteristics: cetane index – 51; polyaromatic hydrocarbon content – 11 %; sulfur content – 0.001 % was used as aggressive medium.

The second method involved experimental investigations of samples in natural conditions under the influence of river water and variable temperatures. The duration of exposure of samples with dimensions of 90×90×2.0 mm in the aggressive environment of river water was $t = 6552 \text{ h}$ (9 months) at the temperature of $T = 263...293 \pm 2 \text{ K}$. The coating with $h = 700...800 \mu\text{m}$ thickness was applied to the samples made of structural steel – St 2 by mechanical method.

For visual analysis of defects, 4 coatings (each of which was applied to 4 sample plates), with 20 cm² working area were used.

Results of the investigations and discussions. Based on the results of the investigation of composite materials adhesive strength [9], where ICTC as the main filler and FPO as additional one were used, we presented the main levels of change in the content of protective coating components in Table 1.

According to the experiment planning scheme, 9 experiments ($N = 9$) were carried out [10]. To exclude systematic errors, each experiment was repeated five times ($p = 5$) [11]. The extended planning matrix of the complete factor experiment and its results are shown in Table. 2.

Table 1

Levels of variables in conditional and natural scales

Components	Factor	Intermediate level, <i>q</i> , wt. p..	Variation step, Δq , wt.p.	Values of variable levels (wt. p.), corresponding to conventional units		
				-1	0	+1
ICTC	x_1	0.050	0.025	0.025	0.050	0.075
FPO	x_2	20	10	10	20	30

Table 2

Adhesive strength of the coating with two-component filler

Investigation No	Component content, <i>q</i> , wt.p.		Adhesive strength at breakaway (σ_a), MPa
	x_1	x_2	
1	0.025	10	59.3
2	0.075	10	49.8
3	0.025	30	58.3
4	0.075	30	49.4
5	0.050	20	61.2
6	0.075	20	52.4
7	0.025	20	51.3
8	0.050	30	55.8
9	0.050	10	60.1

Coefficients of the regression equation (Table 3) were determined according to papers [10–11].

Table 3

Coefficients of the regression equation

b_0	b_1	b_2	b_{11}	b_{22}	b_{12}
58.87	-2.97	-1.07	-5.70	0.50	0.28

At the same time, the following regression equation was obtained:

$$y = 58,87 - 2,97x_1 - 1,07x_2 - 5,70x_1^2 + 0,50x_2^2 + 0,28x_1x_2.$$

For statistical processing of the obtained experimental results, the reproducibility of the experiments was checked according to Cochran criterion G [10, 11]. In this case, to define the calculated value of Cochran criterion, it is necessary to predetermine the values of adequacy dispersions (S_{ui}^2) and of reproduction dispersions ($S^2 \{y\}_i$) according to the methods given in paper [10] (Table 4).

After performing mathematical calculations, it was found that the condition $G = 0.273 \leq G_{3;8; 0.95} = 0.478$ (for the adhesive strength at breakaway) is met.

Further, we determined the significance of the regression equation coefficients by analyzing the results according to the experimental plan (Table 4). The calculated values of

Student's criterion $t_{0p}, t_{1p}, t_{2p}, t_{12p}, t_{11p}, t_{22p}$ are greater than tabular ones t_T , so it was considered that all the regression equation coefficients are significant. Taking into account the above mentioned conditions, the regression equation remains unchanged:

$$y = 58,87 - 2,97x_1 - 1,07x_2 - 5,70x_1^2 + 0,50x_2^2 + 0,28x_1x_2$$

Table 4

Experimentale results of the investigations of adhesive strength at breakaway

Investigation No	Adhesive strength at breakaway (σ_a), MPa					Average value of adhesive strength (σ_a), MPa	Adequacy dispersion S_{ui}^2 , MPa	Reproduction dispersions $S^2\{y\}_i$, MPa
	1	2	3	4	5			
1	59.8	60.0	59.5	59.9	59.3	59.3	0.023	0.047
2	49.5	50	49.9	49.9	49.7	49.8	0.013	0.027
3	58.5	58	58.4	58.2	58.4	58.3	0.013	0.027
4	49.2	49.3	49.5	49.4	49.6	49.4	0.010	0.020
5	61.6	61.4	61.6	61.5	61.1	61.2	0.003	0.007
6	52.2	52.1	52.5	52.6	52.6	52.4	0.003	0.007
7	51	51.2	51.5	51.4	51.4	51.3	0.003	0.007
8	56	55.9	55.7	55.6	55.8	55.8	0.010	0.020
9	60.4	60.1	60.5	60.1	60.2	60.1	0.030	0.060

The adequacy of the obtained model was tested by Fisher's criterion [10, 11]:

$$F_p = \frac{S_{u \max}^2}{S_y^2} \leq F_{(0,05; f_{ao}; f_y)} \quad (2)$$

The calculated value of Fisher's criterion is less than the tabulated one, which at 5 % significance level is $F_{(t)} = 3.25$. That is, condition (2) is met. It can be assumed that the equation adequately describes the composition.

The conversion of coded values into natural ones was performed according to the formula [10, 11]:

$$x_i = \frac{q_i - q_{i0}}{\Delta q_i} \quad (3)$$

After performing mathematical transformations using formula 3, we obtained the following regression equation with natural values of the variable parameters:

$$\sigma_a = 70,069 - 141,196q_1 - 0,363q_2 - 0,036q_1^2 + 0,005q_2^2 + 1,12q_1q_2$$

On the basis of the mathematical planning of the experiment (Table 4), it was found that polymeric materials containing the following components: ED-20 binder – 100 wt. p, PEPA hardener – 10 wt. p, basic filler (ICTC) ($d = 10 \dots 12 \mu\text{m}$) – 0.050 wt.%; additional filler (FPO) ($d = 0,4 \dots 0,6 \mu\text{m}$) – 20 wt. p. were characterized by the maximum adhesive strength. Such

composition is reasonable to use for the formation of protective coatings with improved adhesive properties, as well as for the formation of coatings resistant to aggressive environments.

When developing coatings resistant to corrosion destruction, their ability to preserve their properties under the influence of external factors such as humidity, temperature, and aggressive media is taken into account. In addition, the rate of the aggressive medium penetration through the polymer coating to the metal base is an important characteristic of corrosion-resistant coating. This makes it possible to assess the manufacturability of the coating and its reliability during operation. If the coating provides rapid penetration of the aggressive medium to the metal base, this can result in the weakening of protective effect and accelerated exfoliation of such materials. Therefore, based on the mathematical planning of the experiment and the results of the adhesion strength investigation, four coatings containing different content of components were identified and tested under conditions of exposure to the aggressive environment – diesel fuel:

- Coating 1 – matrix (coating without filler);
- Coating 2 – (ICTC (0,025 wt.p.) + FPO (10 wt.p.)).
- Coating 3 – (ICTC (0,050 wt.p.) + FPO (20 wt.p.));
- Coating 4 – (ICTC (0,050 wt.p.) + FPO (10 wt.p.));

Experimental tests of the resistance of the developed protective coatings to aggressive environments were carried out in the following sequence:

1) Metal test specimens, 20×20×2.0 mm in size, coated on all sides with protective coating, were glued to the bottom of the plastic container (Fig. 1) using Loctite 5923 fuel-resistant sealant. A hole was made on one side of the coating and in the bottom of the container. A hole was made in the coating on one side in the bottom of the container. The hole was made in such a way that the contact of E7-22 type RCL-meter device could be connected to the metal base.

2) The tightness of the container was checked for 48 hours.

3) Containers with coatings were filled with aggressive medium (diesel fuel that meets the requirements of Euro-5 EN 590 standard).

4) The resistance and capacitance of the coatings were measured by E7-22 type RCL meter for 30 days at the test temperature $T = 293 \pm 2$ K.

5) The obtained values were calculated according to formulas (1) and graphs of the obtained dependencies were built.

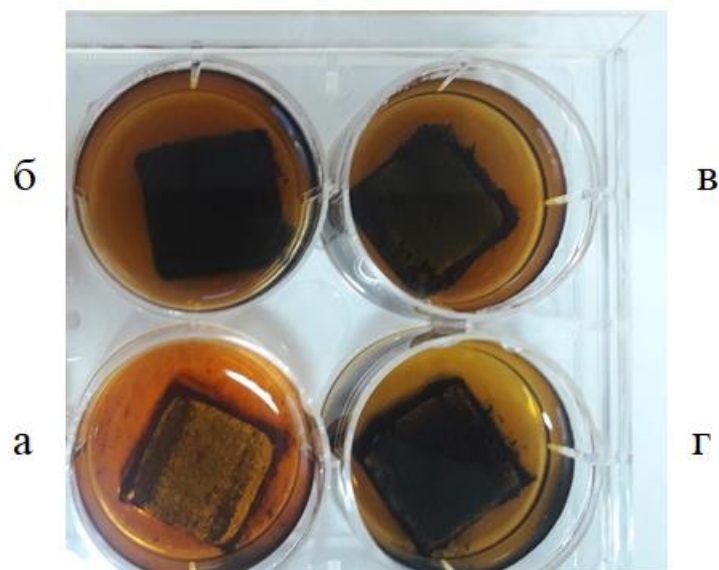
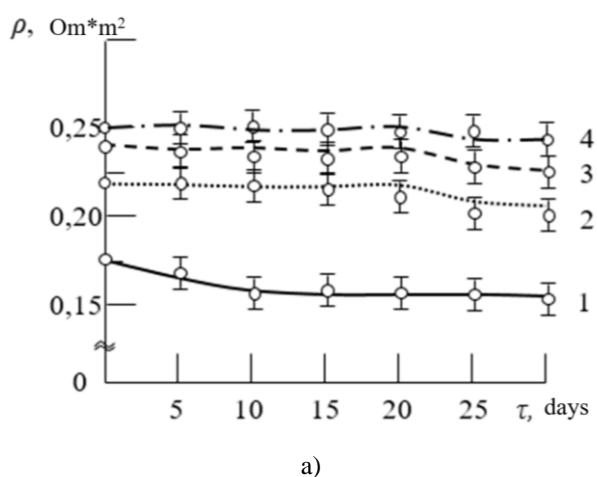
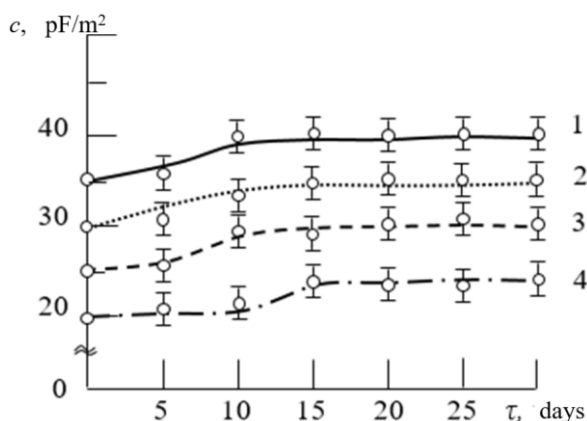


Figure 1. Investigation of the corrosion resistance of the developed coatings in the laboratory for 30 days at test temperature $T = 293 \pm 2$ K: a – Coating 1 (epoxymatrix); b – Coating 2 (ICTC (0.025 wt.p.) + FPO (10 wt. p.); c – Coating 3 (ICTC(0.050 wt. p.) + FPO (20 wt. p.)); d – Coating 4 (ICTC (0.050 w. p.) + FPO (10 wt. p))

Thus, based on the dynamics of changes in the resistivity and specific capacitance, it was determined experimentally that the epoxy matrix is characterized by the lowest corrosion resistance in the aggressive environment (Fig. 2). During 30 investigation days the resistance of the developed coatings to the effects of the aggressive environment is: the value of resistivity – $\rho = 0.155 \dots 0.175 \text{ Ohm}\cdot\text{m}^2$ (Fig. 2, a, curve 1) and the specific capacitance – $c = 35 \dots 40 \text{ pF}/\text{m}^2$ (Fig. 2, b, curve 1). It should be noted that the values of resistivity and specific capacitance stabilize on the 10th day of testing, which is the inhibition of the diffusion process. For the developed coatings, no changes in the resistivity and resistivity values were observed during 20 days of experimental studies (Fig. 2, a, b, curves 2–4). This indicates the complication of the electric current passage through the developed polymer coatings. At the same time, coating 4 is characterized by the highest value of resistivity ($\rho = 0.240 \dots 0.250 \text{ Ohm}\cdot\text{m}^2$). It was believed that the obtained investigation results indicate the maximum compaction of the polymer's spatial grid, which ensures the slowdown of electrochemical reactions on the substrate surface. That is, the maximum compaction of the polymer structure contributes to the formation of hydrophobic surface, which reduces the penetration of moisture into the polymer, and, consequently, increases its hydrophobic properties. After the 20th day of experimental studies, insignificant changes in resistivity within the range of $\rho = 0,05 \text{ Ohm}\cdot\text{m}^2$ were observed for the developed coatings. within the range of $\rho = 0,05 \text{ Ohm}\cdot\text{m}^2$. This fact additionally confirms the previously put forward provisions about creating the barrier to the penetration of aggressive water molecules into the base.



a)

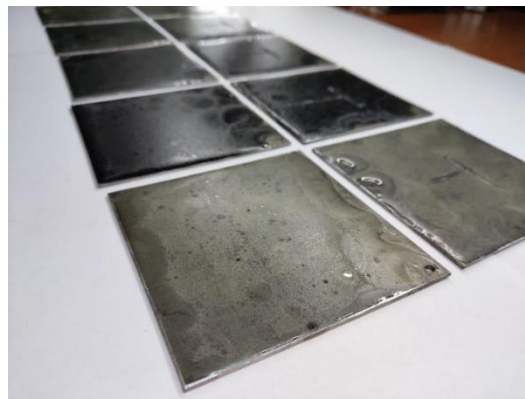


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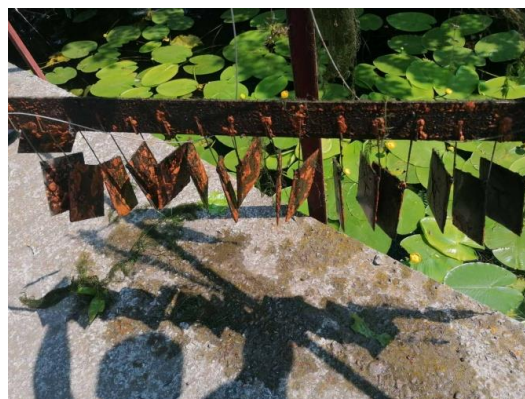
Figure 2. Change in resistance (a) and capacitance (b) over time of the developed epoxy protective coatings at frequency of 1 kHz under conditions of exposure to the aggressive environment (diesel fuel) for 30 days at test temperature of $T = 293 \pm 2 \text{ K}$: 1 – Coating 1 (epoxymatrix); 2 – Coating 2 (ICTC (0.025 wt. p.) + FPO (10 wt. p.)); 3 – Coating 3 (ICTC (0.050 wt. p.) + FPO (20 wt. p.)); 4 – Coating 4 (ICTC(0.050 wt. p.) + FPO (10 wt. p.))

However, the obtained values of the specific capacity of coatings makes it possible for us to state that coatings 4 containing the following components: epoxy binder ED-20 – 100 wt. p; polyethylene polyamine PEPA hardener – 10 wt. p.; basic filler (ICTC) - 0.050 wt. p.; additional filler (FPO) – 10 wt. p. are characterized by improved anticorrosive properties. That is, coating 4 is characterized by the lowest value of the specific capacitance - $c = 20 \dots 24 \text{ pF/m}^2$. At the same time, for such polymeric coatings (coating 4), the value of the capacitance increases in the interval of 10...15 days of testing, while for coatings 1–3, the specific capacitance increases at the beginning of the experiment.

Simultaneously, we carried out the experimental investigations of the corrosion resistance of the developed coatings in natural conditions under the influence of river water and variable temperatures ($T = 263 \dots 293 \pm 2 \text{ K}$) for $t = 6552 \text{ h}$ (9 months). The coating was applied to metal plates made of structural steel – St 2 ($90 \times 90 \times 2.0 \text{ mm}$) by mechanical method on one side (Fig. 3, a). To accelerate the course of physicochemical processes of corrosion destruction and to examine defects on the surfaces of the formed coatings, «T»-shape cuts were made (Fig. 3). The protective coatings applied to the metal sample-plate were immersed to the depth of 2.0...2.5 meters into the river water environment (Dnipro River, Kherson).



a)



b)

Figure 3. The general view of coatings that were tested under the influence of river water and variable temperatures ($T = 263 \dots 293 \pm 2 \text{ K}$): a – developed coatings before testing; b – developed coatings that were tested under the influence of river water for $t = 6552 \text{ h}$ and variable temperatures ($T = 263 \dots 293 \pm 2 \text{ K}$)

Based on the data obtained from the visual image of the surface of the applied protective coatings, which were kept in natural conditions under the influence of river water and variable temperatures for $t = 6552 \text{ h}$ (9 months), the following was determined: the surface of the coating, which does not contain fillers (matrix), is characterized by significant defects in the form of cracks and exfoliation (Fig. 4, a, b).

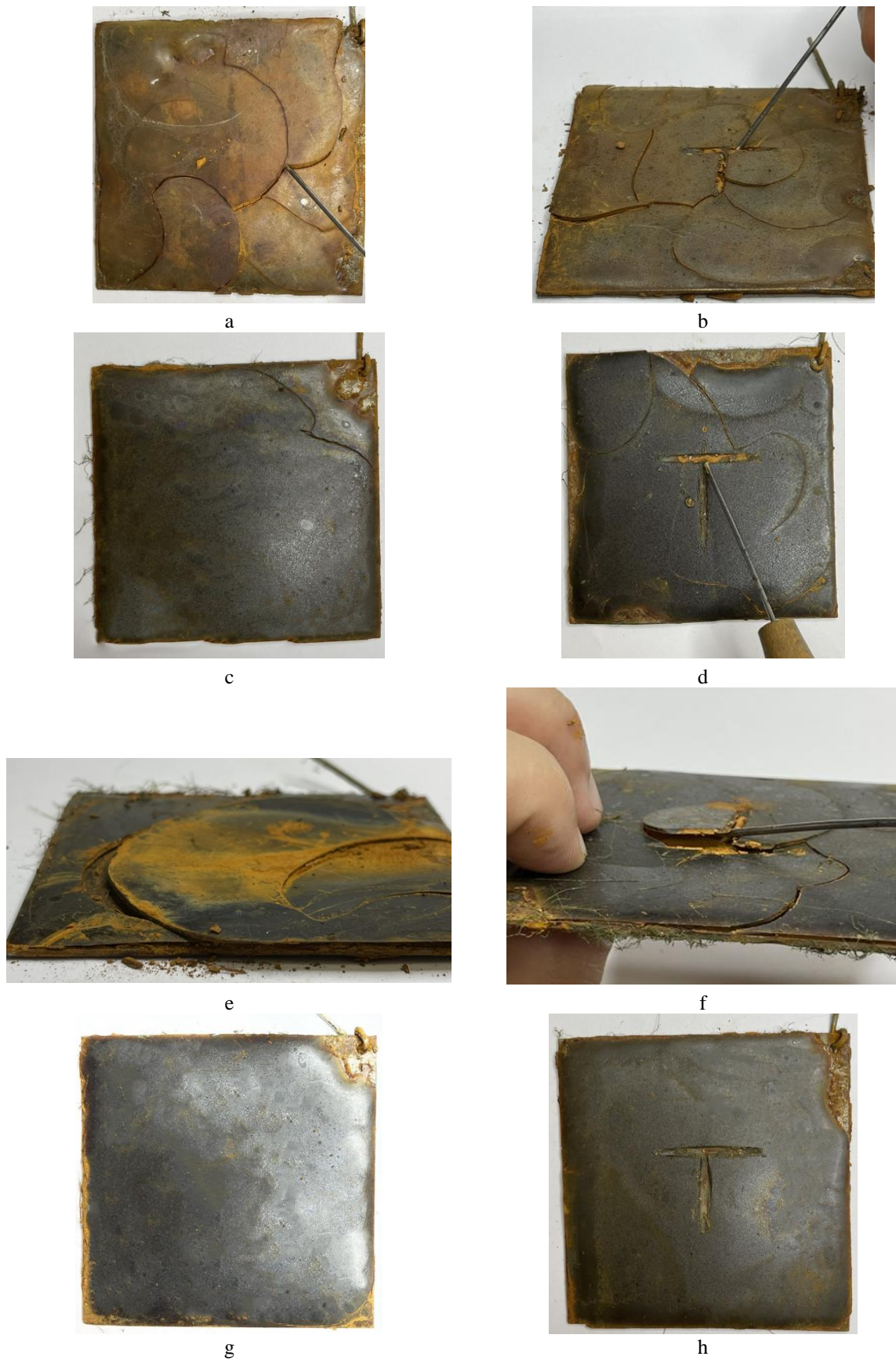


Figure 4. General view of surface defects of the developed coatings, which were tested under the influence of river water and variable temperatures ($T = 263 \dots 293 \pm 2 \text{ K}$) during $t = 6552 \text{ h}$: a, b – coating 1; c, d – coating 2; d, e – coating 3; f – coating 4

It was considered that the formed defects were associated with a slight degree of polymer cross linking, resulting in the sorption of aggressive environment. Similar defects (cracking and exfoliation) were observed for coating 3 (Fig. 4, e, f). It was believed that the swelling and subsequent exfoliation of such materials was associated with the course of the sorption process, which indirectly indicated the structural heterogeneity of the coating. Taking into account the content of fillers present in the protective coatings, it was assumed that the heterogeneity of the polymer structure was caused by the oversaturation of the filler of plant origin, the content of which is the maximum value among the developed coatings – 20 wt. p. Obviously, at the stage of forming the protective coating, the viscosity of the polymer system increases, and this, in turn, creates conditions for incomplete wetting of fillers with epoxy binder.

Analysis of the surface of coating 2 (Fig. 4, c) revealed its continuity with the presence of small crack, which could indicate the technological aspects of the application. This indicates sufficiently high protective ability of the developed coating. Simultaneous comparison of the same coating, but with «T»-shape cut, revealed minor cracks on the surface as well. However, it should be noted that in «T»-shape cut (Fig. 4, d), no swelling or exfoliation of the coating, but traces of corrosion damage to the substrate were observed. Coating 4 (Fig. 4, f, g) is worthy of attention. Visual analysis of such coating showed no signs of exfoliation, cracking, or swelling of the coating, which was consistent with the results of the corrosion resistance investigation conducted in the laboratory.

Conclusions. By the method of mathematical planning of the experiment using the STATGRAPHICS® Centurion XVI application package, the content of additives with different physical and chemical nature in the epoxy binder was optimized in order to obtain protective coatings with improved performance characteristics. It has been proved that the introduction of the synthesized powdered iron-carbide-titanium charge ($d = 10...12 \mu\text{m}$) with the content of $q = 0.050$ wt. p. and the filler of plant origin ($d = 0.4...0.6 \mu\text{m}$) with the content of $q = 20$ wt. p. per 100 wt. p. of ED-20 oligomer and 10 wt. p. of PEPA hardener ensures the formation of the material with adhesive strength at breakaway $\sigma_a = 61.2$ MPa. That is, optimization of the content of fillers with different physical and chemical nature in the epoxy binder provides the increase in adhesive strength of the composite by 2.4 times (compared to the unfilled matrix), which makes it possible to use such materials in the formation of protective coatings for functional purposes.

On the basis of the investigation of the corrosion durability of the developed protective coatings in the river water environment (Dnipro River, Kherson) and diesel fuel (meets the requirements of Euro-5 EN 590), it was found that the coatings with the following composition of components were characterized by improved corrosion durability: epoxy binder ED-20 – 100 wt. p., polyethylene polyamine PEPA hardener – 10 wt. p.; the main filler of synthesized powdered iron-carbidotitanium charge – 0.050 wt. p.; additional filler of plant origin – 10 wt. p. Such coatings are characterized by the maximum (among the investigated materials) value of resistivity – $\rho = 0.240...0.250 \text{ Ohm} \cdot \text{m}^2$ and the lowest value of specific capacitance – $c = 20...24 \text{ pF}/\text{m}^2$. Additionally, the visual analysis of the surface of the developed coatings did not reveal any defects after testing for $t = 6552$ h and exposure to variable temperatures – $T = 263...293 \pm 2 \text{ K}$.

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АНТИКОРОЗИЙНІ ПОЛІМЕРНІ ПОКРИТТЯ ДЛЯ ЗАХИСТУ ЗАСОБІВ ТРАНСПОРТУ

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Резюме. Для формування антикорозійних покриттів використано епоксидний діановий олігомер марки ЕД-20, твердник поліетиленполіамін ПЕПА, синтезовану порошкову залізо-карбідотитанову шихту дисперсністю 10...12 мкм, наповнювач рослинного походження дисперсністю 0,4...0,6 мкм. Методом математичного планування експерименту з використанням прикладного пакета STATGRAPHICS® Centurion XVI оптимізовано вміст різних за фізико-хімічною природою добавок у епоксидному зв'язувачі для отримання захисних покриттів з поліпшеними експлуатаційними характеристиками. На основі математичного планування експерименту визначено 4 різних захисних покриттів, які випробовували на стійкість до агресивних середовищ: річкової води (р. Дніпро, м. Херсон) і дизельного пального (відповідає вимогам стандарту Євро-5 EN 590). Випробовування розроблених захисних покриттів проводили за двома незалежними методами. Перший метод передбачав визначення питомого опору й питомої ємності у лабораторних умовах з використанням приладу RCL-метр типу Е7-22. Таким чином, проведено дослідження зміни значення питомого опору й питомої ємності у середовищі дизельного пального з урахуванням раціонального співвідношення різнодисперсних наповнювачів у епоксидному зв'язувачі. Досягнуто зменшення значення питомого опору захисних покриттів у 1,5...1,6 рази відносно епоксидної матриці. При цьому встановлено кореляційну залежність значення ємності, яке зменшується у 1,8...2,0 разів відповідно. Другий метод передбачав візуальний аналіз поверхні розроблених покриттів, які витримували впродовж 6552 год. у середовищі річкової води при змінних температурах – $T = 263 \dots 293 \pm 2$ К. Для покриттів, що містять у своєму складі раціональне поєднання двох наповнювачів, спостерігали відсутність дефектів у вигляді тріщин, відшарування й набухання.

Ключові слова: епоксидний зв'язувач, адгезія, наповнювач, математичне планування експерименту, корозія, аналіз дефектів.

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