DOI: 10.31471/2311-1399-2022-2(18)-45-52

Multifunctional polymer composites for oil and gas production complex equipment

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Received: 08.08.2022 Accepted: 17.09.2022

Abstract

For the repair of pipeline transport, a polymer adhesive has been developed, which provides high indicators of adhesive strength. For its formation, ED-20 brand epoxy dian oligomer and plant filler with a dispersion of 400–600 nm and a content of q = 0.050–0.500 pts.wt. (per 100 pts.wt. of ED-20 epoxy oligomer) were used. Polyethylene polyamine PEPA hardener (q = 10 pts.wt.) was used to crosslink the epoxy binder. It is shown that the maximum value of tearing adhesive strength (σ_a = 33.6 MPa), shearing (τ_a = 11.2 MPa) and the minimum value of residual stresses (σ_{res} = 0.80–0.88 MPa) are characterized by materials containing filler of plant origin q = 0.075–0.100 pts.wt. Comprehensive studies of physical and mechanical properties were conducted, based on the results of which the optimal filler content in the reactoplastic matrix was established, which is q = 0.075–0.100 pts.wt. per 100 pts.wt. of epoxy oligomer ED-20 and 10 wt.h. PEPA hardener. Such composites are characterized by mechanical properties at bending: destructive stresses σ_{fl} = 102.9–118.7 MPa, modulus of elasticity E = 3.0–3.2 GPa. The content of additives of various dispersity in the ED-20 epoxy binder was determined by the method of mathematical planning of the experiment. This made it possible to obtain polymer coatings with a complex of improved mechanical properties: σ_{fl} = 107.3 MPa, E = 4.6 GPa.

Keywords: destructive bending stresses, epoxy matrix, mathematical planning of the experiment, modulus of elasticity.

Introduction

Pipeline transport accounts for 11–15 % of the volume of global freight turnover. One of the components of the oil and gas transportation equipment are pipes, which combine various components of the technological chain into a single production complex. In this way, pipelines are used to transport strategic liquids from places of extraction or production to the consumer. The network of pipelines allows to efficiently move natural gas, oil and petroleum products over long distances without intermediate processes of their overloading. However, the operation of pipeline transport under the influence of aggressive external factors leads to the formation of various types of destruction, which disrupts the continuity of its functioning [1–3].

In this regard, a topical task is to find ways to ensure the continuity of operation of pipeline transport, as well as to develop new materials for carrying out repair works in order to ensure the reliable operation of the oil and gas production complex. High adhesive strength to the metal base, chemical resistance to the influence of aggressive environments, increased wear resistance allows to use reactive plastic polymers in the form of coatings to protect multifunctional equipment of the oil and gas production complex. To ensure reliable operation of coatings, as well as during repair work (coupling-glue restoration of pipeline sections), not only high values of adhesive strength, but also indicators of physical and mechanical properties are important [4–7]. At the same time, taking into account the possibility of operating pipeline transport under conditions of influence of the marine ecosystem (underwater pipelines), the environmental friendliness of the components of protective coatings is relevant. Therefore, additives of plant origin are increasingly used [8, 9], which makes it possible not only to increase mechanical strength indicators, but also to ensure environmental friendliness. Taking into account all the above, the development of a multifunctional polymer composition intended for protection, as well as suitable for use during repair work of the equipment of the oil and gas production complex, is up-to-date.

The purpose of the work is to determine the optimal content of plant origin filler in the epoxy binder for the formation of a multifunctional composition.

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Materials and research methodology

ED-20 epoxy resin was used to form epoxy nanocomposites. Polyethylene polyamine (PEPA) cold-hardening hardener was used to crosslink the epoxy binder.

An additive of plant origin was used as a filler for experimental studies. The primary form of the additive are pressed briquettes based on secondary products of plant processing with the size: d = 60 mm, l = 80–150 mm (Fig. 1). Ash content is 2.8 %, density is 0.855 g/cm^3. Pressed material of plant origin is used in boiler houses for heating residential premises. The final product is a filler (Fig. 2), obtained as a result of thermal decomposition of pressed organic material.

Figure 1 – Pressed briquettes based on the secondary products of plant processing

The technology of forming composite materials (CM) was carried out in a certain sequence, which is indicated in the scientific works [10, 11].

In the work, the following was studied: tearing adhesive strength, shearing adhesive strength, residual stresses, modulus of elasticity at bending, destructive stresses at bending. The size of the filler and the nature of the adhesive joint tearing were determined by optical microscopy.

The adhesion strength of the coatings to the metal base was determined by measuring the destructive stress ("fungi method") with uniform tearing of a pair of glued samples according to ASTM D897−08. The study of adhesive strength at shearing was carried out similarly by measuring the pull-off force of adhesive joints of steel samples on an automated tearing machine UM-5 at a loading speed of υ = 10 N/s. The diameter of the working part of the steel samples at tearing was d = 25 mm. The gluing area of the samples, which were studied at tearing and shearing, was the same.

Residual stresses in the matrix were determined by the cantilever method [10]. The coating with a thickness of δ = 0.3–0.5 mm was formed on a steel base. Base parameters: total length l = 100 mm, operating length l_0 = 80 mm, thickness δ = 0.3 mm.

Breaking stresses and flexural modulus at bending were determined according to ASTM D 790−03. Parameters of the samples: length l = 120 ± 2 mm, width b = 15 ± 0.5 mm, height h = 10 ± 0.5 mm.

The deviation of the values during the study of the adhesive strength indicators of the physical and mechanical properties of CM was 4–6 % from the nominal.

To improve the physical and mechanical properties of protective coatings, two fillers were used in a complex (a mixture of discrete fibers based on cotton and polyester and a filler of plant origin), the optimal content of which was determined by the method of mathematical planning of the experiment.

Research results and discussion

Previously, a polarizing microscope POLAM R-211 was used to determine the size of the particles of plant-based filler (PBF). Based on the obtained research results, the size of the filler particles was determined (Fig. 2), which is 400–600 nm.

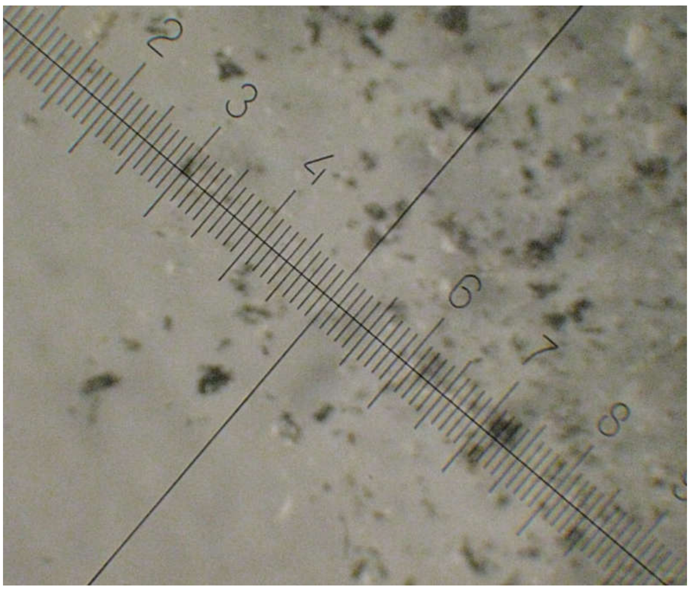


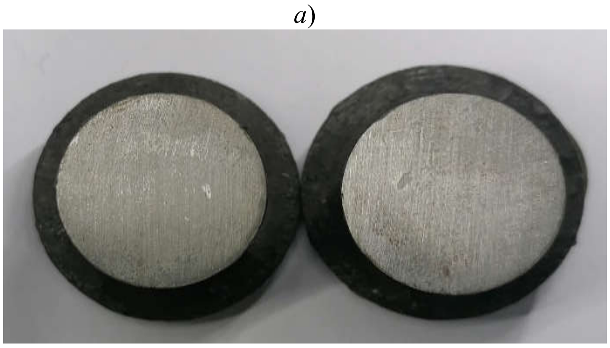
Figure 2 – General view of PBF particles on a polarizing microscope POLAM R-211 (graduation is 0.5 μm)

Table 1 – Adhesive properties of composites filled with PBF

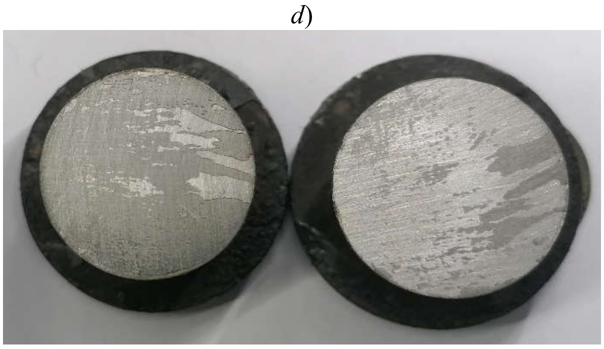
Content of the PBF q, pts.wt.	Tearing adhesive strength σ_a, MPa	Shearing adhesive strength τ_a, MPa	Residual stresses σ_{res}, MPa	Tearing nature of adhesives
0.050	24.6	8.8	1.19	adhesive and cohesive
0.075	33.6	10.9	0.88	cohesive
0.100	27.6	11.2	0.80	cohesive
0.300	22.3	9.4	0.85	adhesive and cohesive
0.500	23.5	8.0	0.96	adhesive

a)

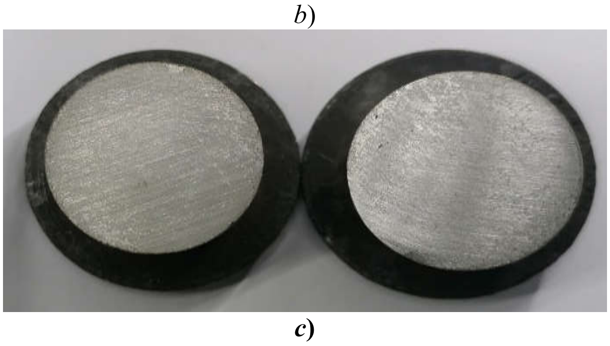
d)



b)



e)



c)

a) 0.050; *b*) 0.075; *c*) 0.100; *d*) 0.300; *e*) 0.500
Figure 3 – Tearing nature of epoxy composites with different content of PBF *q*, pts.wt.

Based on the analysis of the considered aspects of connecting and carrying out repair activities (restoration) of pipelines [8, 9, 12], in particular, polymer pipelines, the development of glued couplings, as well as bandage products using epoxy compositions, is up-to-date. For this, it is necessary to develop polymer compositions with increased indicators of adhesive strength. Therefore, further the influence of PBF with a dispersion of 400–600 nm on adhesive strength indicators (tearing adhesive strength, shearing adhesive strength, residual stresses) was studied. It is shown that the introduction of PBF particles with a content of q = 0.050 pts.wt. does not significantly affect the indicators of adhesive strength at tearing and shearing (Table 1), since their value is at the level of the unfilled epoxy matrix [10]. The obtained values of the residual stresses σ_{res} = 1.19 MPa (Table 1) and the mixed nature of the tearing (Fig. 3, *a*) allow to state that there is minor chemical interaction of the adhesive with the substrate. Then, as the introduction of PBF particles with a content of q = 0.075 pts.wt. provides an increase in the tearing adhesive strength to the maximum value σ_a = 33.6 MPa, as well as an increase in the shearing adhesive strength to τ_a = 10.9 MPa. However, the maximum value of shearing adhesive strength was observed when PBF particles were introduced with a content of q = 0.100 pts.wt. It is obvious, that in this case there is a different mechanism of tearing, which is associated with a different amount of influence on the process of destruction of the adhesive joint by normal and tangential stresses [13, 14].

Table 2 – Mechanical properties of composites filled with PBF

Content of the PBF q, pts.wt.	Destructive stresses at bending σ_{fl}, MPa	Modulus of elasticity at bending E, GPa
0.050	87.4	2.5
0.075	118.7	3.0
0.100	102.9	3.2
0.300	76.5	2.9
0.500	82.8	2.8

At the same time, the introduction of PBF with a content of q = 0.075–0.100 pts.wt. provides a monotonous decrease in residual stresses (Table 1), which makes it possible to use such adhesives without peeling and cracking. The tearing nature of such adhesives is cohesive (Fig. 3, *b*, *c*), which indicates a significant chemical interaction of the polymer with the metal base. Further introduction of the filler (q = 0.300–0.500 pts.wt.) leads to a decrease in the adhesive strength of the reactive plastic polymer σ_a = 22.3–23.5 MPa, τ_a = 8.0–9.4 MPa. A decrease in adhesive strength indicators and an increase in the value of residual stresses (Table 1) may indicate an increase in the viscosity of the polymer, and, therefore, the possibility of the formation of heterogeneous structures in the adhesive film. Therefore, the transitional nature of the tearing was observed, namely the transition from adhesive and cohesive (Fig. 3, *d*) to adhesive (Fig. 3, *e*).

When using polymer compositions as a surface layer of a protective coating, it is necessary to ensure an increase in the indicators of physical and mechanical properties. Therefore, the influence of plant-based filler on the indicators of cohesive strength at bending (destructive stresses, modulus of elasticity) was further studied. It is shown (Table 2), that the maximum indicators of destructive stresses at bending are characterized by the material with the content of PBF q = 0.075–0.100 pts.wt. With this content, a composite material is formed, the destructive stresses of which are σ_{fl} = 102.9–118.7 MPa (Table 2). A 2.4-times increase of the investigated property, compared to the epoxy matrix, indicates an increase in the density of the spatial grid of the filled polymer, and, therefore, a change in the stress-strain state of the material. In this case, the optimal amount of plant-based additive ensures moderate flexibility of the polymer chain. Due to this, the material is characterized by an increased ability to resist deformation under the load. A further increase in the filler content within q = 0.300–0.500 pts.wt. leads to deterioration of the strength parameters of the composite material (σ_{fl} = 76.5–82.8 MPa).

Based on the obtained research results of the additive effect on the value of the modulus of elasticity, a similar dependence was observed (Table 2). The minimal amount of the additive (q = 0.050 pts.wt.) does not significantly affect on the elasticity of the polymer (E = 2.5 GPa), which indicates an insufficient number of contacts of the functional groups of the polymer with the active additive centers. Then, as the introduction of PBF with a content of q = 0.075–0.100 pts.wt. provides increased elasticity of the composite. The modulus of elasticity increases to E = 3.0–3.2 GPa. However, the values of the modulus of elasticity are relatively not high. Therefore, one of the methods of increasing the elasticity of the polymer is the combination of fillers of different physical and chemical nature and dispersion. Thus, in the previous work [10] it was shown, that composites filled with a mixture of discrete fibers based on cotton and polyester with a content of q = 0.50–0.75 pts.wt. are characterized by increased strength indicators. per 100 pts.wt. epoxy oligomer ED-20 and 10 wt.h. PEPA hardener. Such composites are characterized by the following properties: impact strength W = 12.7–13.2 kJ/m^2; destructive stresses at bending σ_{fl} = 56.0–62.0 MPa, modulus of elasticity at bending E = 3.15–3.30 GPa. Thus, a rational combination of additives of different physico-chemical nature and dispersion will ensure an increase in the degree of crosslinking of the composite material and, accordingly, will improve the operational characteristics of the developed protective coatings, in particular the modulus of elasticity.

Therefore, in order to improve the operational characteristics of protective coatings, an experimental study was conducted, taking into account fillers of different dispersions in the epoxy binder. Previously, on the basis of complex studies of the physical and mechanical properties of CM, the rational content of the main filler was chosen – a mixture of discrete fibers based on cotton and polyester [10] and an additional filler of plant origin (Table 2).

According to the experiment planning scheme, 9 experiments (N = 9) were conducted [15, 16]. To exclude systematic errors, each experiment was repeated three times (p = 3) [15]. The extended planning matrix of the full factorial experiment and its results are given in Table 3.

For statistical processing of the obtained results of the experiment, the reproducibility of the experiments was checked based on the Cochrane G criterion [15, 16]. At the same time, in order to determine the estimated value of the Cochran criterion, firstly it is necessary to determine the values of the variances of adequacy (S_{ui}^2) and variances of reproduction (S^2 $\{y_i\}$) according to the methods given in the work [15, 16] (Table 4).

After making mathematical calculations, it was established that the condition $G = 0.394 \leq G_{3;8;\,0.95} = 0.478$ (for destructive stresses at bending) is fulfilled.

Further, the significance of the coefficients of the regression equation was determined by analyzing the results according to the experimental plan (Table 4). The calculated values of the Student's test t_{0p}, t_{1p}, t_{2p},

Table 3 – Research results of the cohesive strength of CM

Experiment	Components content q, pts.wt.		Destructive stresses σ_{fl}, MPa	Modulus of elasticity E, GPa
	x_1	x_2	y_1	y_2
1	0.25	0.050	82.2	3.2
2	0.75	0.050	86.4	3.4
3	0.25	0.100	100.2	4.4
4	0.75	0.100	76.8	3.3
5	0.50	0.075	122.2	3.8
6	0,75	0.075	89.2	3.6
7	0.25	0.075	90.7	3.6
8	0.50	0.100	107.3	4.6
9	0.50	0.050	84.6	3.4

Table 4 – Experimental results of the study of destructive stresses at bending materials

Experiment	Destructive stresses at bending σ_{fl}, MPa			Average value σ_{fl}, MPa	Variances of adequacy S_{ui}^2, MPa^2	Variances of reproduction $S^2\{y_i\}$, MPa^2
	1	2	3			
1	82.60	82.20	81.80	82.2	0.16	0.32
2	86.40	86.00	86.80	86.4	0.36	0.72
3	100.20	100.20	100.20	100.2	0	0
4	77.00	76.80	76.60	76.8	0.04	0.08
5	122.20	122.00	122.40	122.2	0.04	0.08
6	89.00	88.80	89.80	89.2	0.28	0.56
7	90.70	90.60	90.80	90.7	0.01	0.02
8	107.20	107.40	107.30	107.3	0.01	0.02
9	84.50	84.60	84.70	84.6	0.01	0.02

t_{12p}, t_{11p}, t_{22p} are greater than the tabular t_T, so it was believed that all coefficients of the regression equation $b_0 = 112.11$, $b_1 = -3.45$, $b_2 = 5.18$, $b_{12} = -6.9$, $b_{11} = -17.12$, $b_{22} = -11.12$ are significant. Taking into account the given provisions, the regression equation remains unchanged:

$$y = 112.11 - 3.45x_1 + 5.18x_2 - 17.12x_1^2 - \\ -11.12x_2^2 - 6.9x_1x_2 \ .$$

According to the Fisher's criterion [15], it was established that the presented equation adequately describes the content of the composition.

The process of interpreting the obtained mathematical model, as a rule, is not reduced only to determining the influence of factors. A simple comparison of the absolute value of the linear coefficients does not determine the relative degree of influence of the factors, since there are also quadratic terms and pairwise interactions. In the detailed analysis of the obtained adequate model, it is necessary to take into account the fact that for the quadratic model the degree of influence of the factor on the change of the initial value is not constant. The transformation of coded values of the regression equation into natural ones was performed according to the methodology presented in the work [16], on the basis of which the following regression equation was obtained:

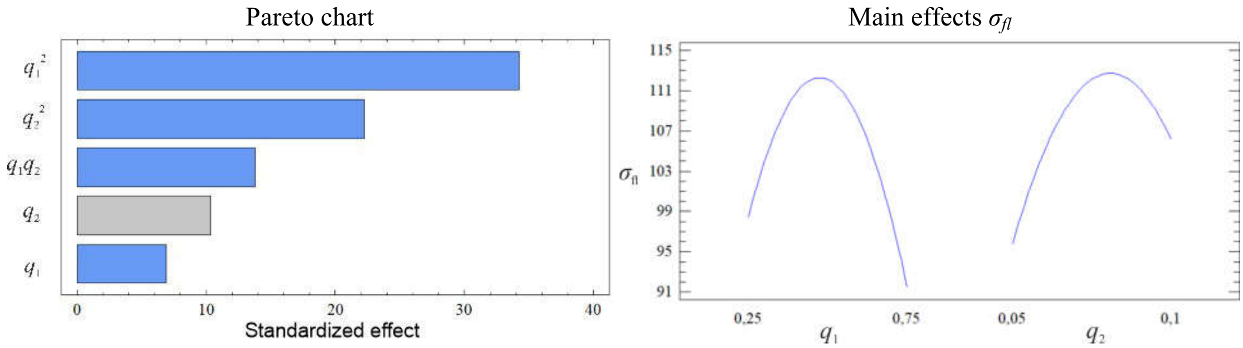
$$\sigma_{fl} = -106.456 + 342.867q_1 + 3427.33q_2 - \\ -273.867q_1^2 - 1104.0q_2^2 - 17786.7q_1q_2 \ .$$

The carried out transformation allows predicting the value of the initial value for any point in the middle of the factor variation area. Also, with its help, you can plot graphs of the dependence of the initial value (destructive stresses at bending) on any factor (or two factors). The geometric interpretation of the response surface is shown in Fig. 4, 5.

Based on the experimental studies, it was established, that both factors are significant. It should be noted that the influence of the content of additional filler on the destructive stresses indicators at bending is higher compared to the main one (according to the Pareto chart). Analyzing the calculated response surface, it was determined that the developed epoxy composite with the following content of dispersed filler has the optimal indicators of destructive stress at bending: a mixture of discrete fibers based on cotton and polyester 0.50 pts.wt., filler of plant origin 0.075 pts.wt. (σ_{fl} = 122.2 MPa).

Similarly to the above scheme of calculations, the composition of the CM was optimized according to the modulus of elasticity at bending. The coding of the natural values of the components and the scheme of planning the experiment are selected according to the Table 3.

On the basis of checking the reproducibility of the experiments according to the Cochran criterion, taking into account the variances of adequacy and variances of reproduction (Table 5), it was established that the condition $G_{calcul} = 0.267 \leq G_{table} = 0.478$ (for the modulus of elasticity at bending) is fulfilled.



a) *b)*

Figure 4 – Pareto chart (*a*) and main effects σ_{fl} (*b*)

Table 5 – Experimental research results of the modulus of elasticity at bending materials

Experiment	Modulus of elasticity at bending E, GPa			Average value E, GPa	Variances of adequacy S_{ui}^2, MPa^2	Variances of reproduction $S^2\{y_i\}$, MPa^2
	1	2	3			
1	3.20	3.10	3.30	3.2	0.01	0.02
2	3.40	3.30	3.50	3.4	0.01	0.02
3	4.50	4.30	4.40	4.4	0.01	0.02
4	3.20	3.30	3.40	3.3	0.01	0.02
5	3.80	3.70	3.90	3.8	0.01	0.02
6	3.60	3.80	3.40	3.6	0.04	0.08
7	3.60	3.80	3.40	3.6	0.04	0.08
8	4.50	4.60	4.70	4.6	0.01	0.02
9	3.40	3.50	3.30	3.4	0.01	0.02

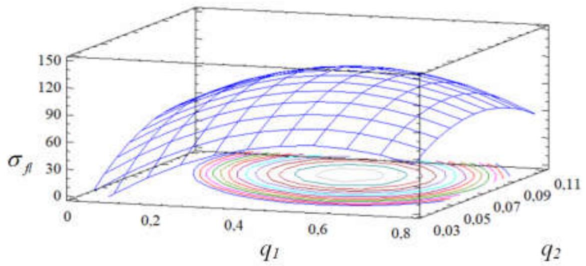


Figure 5 – Estimated response surface $\sigma_{fl} = f(q_1, q_2)$

Further, the significance of the coefficients of the regression equation was determined by analyzing the results according to the experimental plan (Table 5). The estimated values of the Student's test t_{0p}, t_{1p}, t_{2p}, t_{11p}, t_{12p} except for t_{22p}, are greater than t_T, so it was considered that only the coefficient of the regression equation $b_{22} = 0.05$ is not significant. As a result, the following regression equation was obtained:

$$y = 3.90 - 0.15x_1 + 0.38x_2 - 0.35x_1^2 - 0.33x_1x_2 .$$

According to Fisher's criterion [15], it was established that the presented equation adequately describes the content of the composition.

After converting the coded values of the regression equation into natural values [16], the following equation was obtained:

$$E = 0.15 + 8.90q_1 + 29.33q_2 - 5.60q_1^2 + 80.00q_2^2 + 52.00q_1q_2 .$$

The surface response geometric interpretation is shown in Fig. 6, 7.

Analyzing the estimated response surface, it was determined that the maximum modulus of elasticity at bending is characterized by the epoxy composite with the following additive content: a mixture of discrete fibers based on cotton and polyester 0.50 wt. parts, filler of plant origin (0.100 pts.wt. (E = 4.6 GPa).

Conclusions

1. The maximum adhesive strength is characterized by polymer materials with a content of q = 0.075–0.100 pts.wt. filler of plant origin. At the same time, a kinetically balanced adhesive film is formed, expressed by the cohesive nature of the separation of the joint surface and insignificant values of residual stresses (σ_{res} = 0.80–0.88 MPa). This makes it possible to use such materials in the repair of pipelines, operating in many industries, as well as the formation of an adhesive layer of functional coatings.

2. The optimal content of filler of plant origin, which provides a significant increase in the mechanical strength of the composite by 1.2–2.4 times (compared to an unfilled matrix), is q = 0.075–0.100 pts.wt. This makes it possible to use such materials when forming the surface layer of functional protective coatings.

3. The content of different physical and chemical additives in the epoxy binder was optimized to obtain multifunctional coatings. The optimal content of the main (the mixture of discrete fibers based on cotton and polyester) and additional (plant filler) fillers for the formation of composites with increased physical and mechanical properties was established. It has been proven

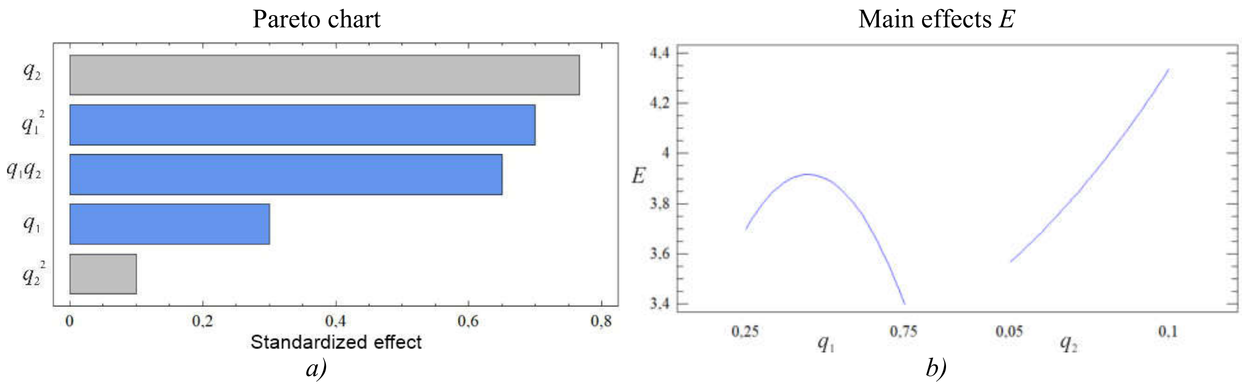


Figure 6 – Pareto chart (*a*) and main effects *E* (*b*)

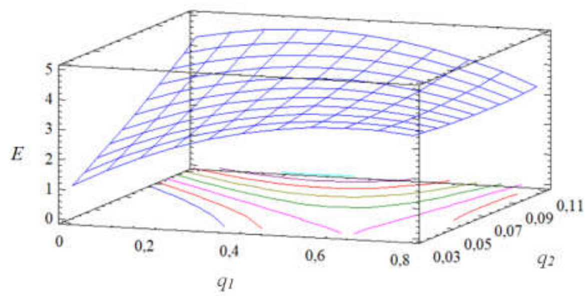


Figure 7 – Estimated response surface $E = f(q_1, q_2)$

that the introduction of a mixture of discrete fibers based on cotton and polyester with a content of q = 0.50 pts.wt. and filler of plant origin with a content of q = 0.100 pts.wt. per 100 pts.wt. oligomer ED-20 and 10 pts.wt. PEPA hardener ensures the formation of a material with the following properties: destructive bending stress σ_{fl} = 107.3 MPa, modulus of elasticity at bending E = 4.6 GPa. The rational combination of fillers of different physical and chemical nature and dispersion made it possible to increase the elasticity of the composite material by 1.4 times, with a slight decrease in the value of the destructive stresses at bending, which indicates a significant interphase interaction of the polymer components, and, therefore, high indicators of operational characteristics.

In order to expand the area of use of the developed materials, it is planned to carry out complex studies of the structure of such composites and resistance to the influence of various aggressive environments.

Publication contains research results, funded within the framework of: a named scholarship of the Verkhovna Rada of Ukraine for young scientists - doctors of science for 2022 (Decree of the Verkhovna Rada of Ukraine dated December 1, 2022 2791-IX); R&D of young scientists "Directed control of the structure formation of nanocarbon-containing polymer composites to improve the operational characteristics of transport» (0121U107610).

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УДК 667.64:678.026

Багатофункціональні полімерні композити для устаткування нафтогазовидобувного комплексу

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Для проведення ремонту трубопровідного транспорту розроблений полімерний адгезив, що забезпечує високі показники адгезійної міцності. Для його формування використовували епоксидний діановий олігомер марки ЕД-20 і наповнювач рослинного походження дисперсністю 400–600 нм за вмісту $q = 0.050$–0.500 мас.ч. (на 100 мас.ч. епоксидного олігомеру ЕД-20). Для зшивання епоксидного зв'язувача використано твердник поліетиленполіамін ПЕПА ($q = 10$ мас.ч.). Показано, що максимальним значенням адгезійної міцності при відриві ($\sigma_a = 33.6$ МПа), зсуві ($\tau_a = 11.2$ МПа) і мінімальним значенням залишкових напружень ($\sigma_{res} = 0.80$–0.88 МПа) характеризуються матеріали, що містять наповнювач рослинного походження $q = 0.075$–0.100 мас.ч. Проведено комплексні дослідження фізико-механічних властивостей, за результатами яких встановлено оптимальний вміст наповнювача у реактопластичній матриці, який становить $q = 0.075$–0.100 мас.ч. на 100 мас.ч. епоксидного олігомеру ЕД-20 і 10 мас.ч. твердника ПЕПА. Такі композити характеризуються механічними властивостями при згині: руйнівні напруження $\sigma_{fl} = 102.9$–118.7 МПа, модуль пружності $E = 3.0$–3.2 ГПа. Методом математичного планування експерименту встановлено вміст різнодисперсних добавок у епоксидному зв'язувачі ЕД-20. Це дозволило отримати полімерні покриття з комплексом поліпшених механічних властивостей: $\sigma_{fl} = 107.3$ МПа, $E = 4.6$ ГПа.

Ключові слова: епоксидна матриця, математичне планування експерименту, модуь пружності, руйнівні напруження при згині.