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REGULAR ARTICLE

Structural and Chemical Features of Epoxy Composites Filled with Biogenic Lignocellulosic Filler Obtained from Agro-Industrial Waste

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The work investigated the effect of organic biogenic microdispersed lignocellulosic filler on the physical, mechanical and thermophysical properties of epoxy composites. Attention is paid to the analysis of structural and interfacial effects that occur at the micro- and nanolevels and determine the performance characteristics of materials. The elemental composition of the filler was investigated using X-ray fluorescence analysis, which enabled the identification of mineral components capable of forming active centers that facilitate interfacial interaction with the epoxy matrix. The organic component of the filler was investigated by gas chromatography with mass spectrometric detection. At the same time, the presence of fatty acids and their glycerol esters with polar functional groups capable of participating in intermolecular interactions was revealed. It has been proven that the introduction of a filler with a content of q = 1,0…1,5 wt.% provides an increase in the destructive stresses during bending, the modulus of elasticity, the impact strength of epoxy composites, and a complex of thermophysical properties. This is due to the action of various mechanisms, in particular physical reinforcement and stress relaxation in the polymer volume. The results of comprehensive studies confirm that the biogenic lignocellulosic filler performs not only the role of a dispersed reinforcing component, but also the function of a structure-forming element that provides directed control of the properties of epoxy composites due to nanolevel interfacial interaction. This opens up prospects for creating functional polymer materials with predicted physical, mechanical and thermophysical characteristics.

Keywords: Epoxy nanocomposites, Coating, Biogenic filler, Epoxy resin, Gas chromatography, X-ray fluorescence analysis, Nanostructures, Performance characteristics.

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1. INTRODUCTION

1.1 Statement of the Problem

The modern development of transport technology involves the use of polymer composites with a complex of enhanced properties, in particular physical, mechanical and thermophysical. This is due to the use of such materials in structural elements and transport parts where variable loads, temperatures, and aggressive external environments are present [1-5]. At the same time, the disadvantages of polymer composites include fragility, which affects their service life. Therefore, one of the promising ways of purposefully regulating the physical, mechanical and thermophysical properties of epoxy materials is the introduction of active fillers, in particular those obtained from waste from the agricultural and food industries. The implementation of such an approach will ensure the rational use of secondary raw materials in the creation of epoxy composites and will allow obtaining new materials with specified physical, mechanical and thermophysical characteristics [6-11].

1.2 Analysis of Recent Research and Publications

In works [3, 4] it is shown that the introduction of differently dispersed fillers into the epoxy matrix allows influencing the physical, mechanical and thermophysical properties by changing the mechanism of deformation and fracture of composites due to the formation of a multilevel polymer structure. The authors showed [4] that an increase in the bending strength of epoxy composites filled with limestone waste particles containing $CaCO_3$ (97.12 %), MgO (1 %), SiO_2 (1.50 %), Fe_2O_3 (0.08 %), Al_2O_3 (0.30 %) can be achieved with a content of 7 mass fraction. At this content, the value of the bending strength is 65 MPa, while the Shore hardness value (85-88) is affected by a linear increase in this additive (7-29 wt. %). However, the results of electron microscopy have proven that an increase in the content of limestone powder leads to the formation of a sedimentary layer (0.9…1.0 mm), which negatively affects the performance characteristics of such polymer composites.

No less effective is the introduction of lignocellulosic

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additives (kenaf, jute, ramie, pineapple fiber, flax, sisal, etc.) into the polymer binder, which significantly increase the value of the bending strength and elastic modulus of polymers [6-14]. At the same time, the results of the authors' research [10-15] indicate that an excessive content (more than 10 wt. %) of biogenic additives leads to agglomeration and local stress concentrators, which negatively affects the mechanical and thermophysical characteristics of composites. Considering the above, it is relevant to search for alternative functional fillers capable of ensuring the formation of an ordered polymer structure without defects in the form of agglomerates, which lead to the emergence of local stress concentrators in the polymer volume. At the same time, it is promising to study the influence of biogenic microdispersed fillers with a polyfunctional chemical nature and a developed surface, which combine the ability for active interfacial interaction with the epoxy matrix.

The aim of the work is to study the nature and influence of the content of biogenic lignocellulosic filler on the properties of epoxy composites.

2. MATERIALS AND RESEARCH METHODS

To create epoxy composites and coatings based on them, the following were used: binder-epoxy oligomer ED-20 (ISO 18280:2010); hardener-polyethylene polyamine, PEPA (TU 6-05-241-202-78). In order to improve the performance characteristics of epoxy composites and protective coatings, a microdispersed organic biogenic lignocellulosic filler (OBLF) synthesized by mechanical grinding and fractionation of coffee industry waste was used. The average size of the filler particles is 10…20 microns. The filler is characterized by a multicomponent organic nature, i.e. the presence of a lignocellulosic component and active functional groups, which determines its ability to actively interact with the epoxy oligomer ED-20. Epoxy composites were formed using the technology described in [6-8].

X-ray fluorescence analysis (XRF). The elemental composition of the organic biogenic lignocellulosic filler was determined by the method of X-ray fluorescence analysis, which is based on the registration of characteristic X-ray radiation induced by the primary X-ray excitation of the sample atoms. Qualitative identification of elements was carried out by the energy position of the characteristic lines, while their quantitative content was determined by the intensity of the corresponding peaks, taking into account matrix corrections. The relative error in determining the concentration of elements of average atomic mass did not exceed ±3…5 %, for light elements – ±7…10 %, which is typical for energy-dispersive X-ray fluorescence analysis in the analysis of biogenic materials. The repeatability of the results was assessed based on the results of a series of parallel measurements. The coefficient of variation of the intensity of the main peaks did not exceed 5 %. The detection limit of elements depended on their atomic number and matrix composition of the sample and was approximately 10-3…10-2 wt. % for elements with $Z \geq 13$. For light elements, the detection limit was higher, which was taken into account when interpreting the results.

Gas chromatography with mass spectrometric detection (GC-MS). The method is based on the separation of volatile and semi-volatile organic components in a chromatographic column with subsequent identification of compounds by their mass spectra. The reproducibility of gas chromatographic analysis results was assessed by repeated measurements. The relative standard deviation of the areas of the main peaks did not exceed 5 %. Mass spectrometric identification was considered reliable if the coincidence coefficient with library spectra was not lower than 80…90 %. The detection limit of organic components depended on their volatility and ionization ability and was approximately 10^{-6}…10^{-5} wt. % for typical organic compounds. The systematic error of quantitative assessment of the relative content of components was taken into account when interpreting the results.

The tensile strength and flexural modulus of polymeric materials were tested according to ASTM D790-03.

The impact strength of polymeric materials was tested according to ASTM D6110-18.

The Martens heat resistance of polymeric materials was tested according to ISO 75-2.

The thermal coefficient of linear expansion of polymeric materials was tested according to ISO 11359-2.

3. DISCUSSION OF RESEARCH RESULTS

Previously, for the correct identification of elements of different atomic masses of organic biogenic lignocellulosic filler (OBLF), two X-ray fluorescence spectra of the sample were obtained, which were analyzed in two energy ranges, in particular: a wide range of 0…55 keV (Fig. 1) and a narrow range of 0…15 keV (Fig. 2). This is due to the fact that X-ray fluorescence analysis (XFA) cannot optimally reflect the entire energy range, since the instrument detector has different sensitivity to low- and high-energy photons, which affects the identification of heavy (with atomic number $Z > 30$), light (with atomic number $Z \leq 12$) and medium (with atomic number $Z = 13$-30) elements of the OBLF. Based on the presented spectra in a wide energy range, the presence of impurity heavy elements was detected, while the detailed spectrum in the low-energy region provided the identification and quantitative assessment of light and medium elements characteristic of organic biogenic lignocellulosic filler.

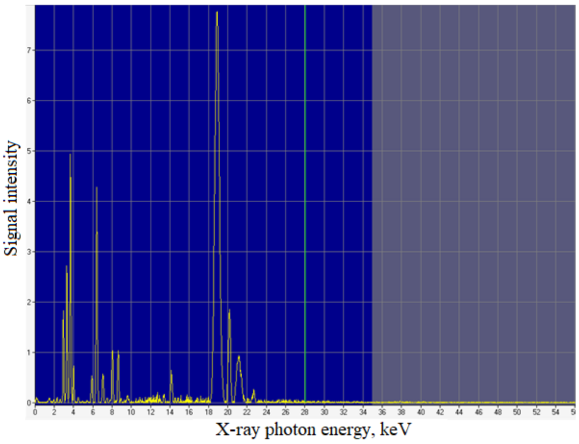

Fig. 1 – Results of X-ray fluorescence analysis in a wide range (0…55 keV)

At the same time, the position of the peaks in the X-ray fluorescence spectra allowed us to determine the elemental composition of the eperimental sample, and their intensity allows us to determine the quantitative content of the corresponding elements (Table 1). The presence of medium Ca, K, P, and light Mg elements was established, which are typical elements for organic biogenic lignocellulosic materials and are due to the presence in the structure of the mineral phase of plant raw materials (calcium, potassium salts, phosphate complexes). The presence of these elements can affect the segmental mobility of the epoxy chain.

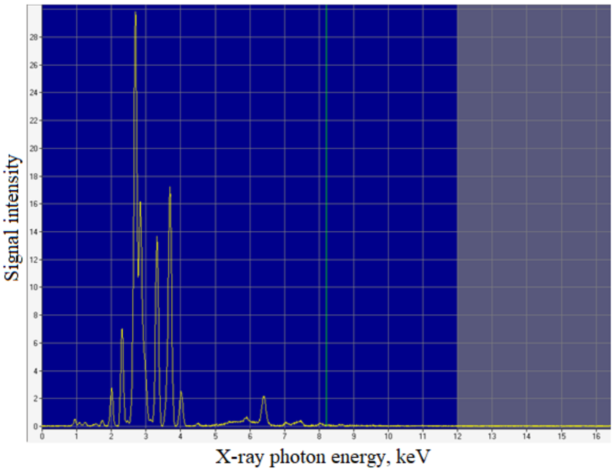

Fig. 2 – Results of X-ray fluorescence analysis in a narrow range (0…15 keV)

Table 1 – Results of the study of organic biogenic lignocellulosic filler by X-ray fluorescence analysis

Atomic number of the element	Name of the element	Intensity	Concentration, %
17	Cl	350528	43.127
20	Ca	25643	18.865
19	K	14111	16.598
16	S	146559	2.561
15	P	54987	1.839
26	Fe	27545	1.363
13	Al	1219	0.311
12	Mg	582	0.302
25	Mn	3491	0.272
29	Cu	7548	0.129
30	Zn	7107	0.092
24	Cr	240	0.032

Additionally, the highest (among the listed elements) percentage content of chlorine (Cl) was established, which is – 43.1 %, which belongs to the group of medium elements. Chlorine does not belong to the structural element of lignocellulose in the composition of OBLC. It belongs to the mineral component of biogenic raw materials and reflects the presence of ionic salts of plant origin, present mainly in the form of KCl, $CaCl_2$, $MgCl_2$. These compounds do not directly participate in the processes of chemical crosslinking of the epoxy matrix, but can indirectly affect the interfacial interaction of the polymer with the filler due to a change in the ionic state and physicochemical properties of the surface layer of the polymer. While the presence in lower concentrations of iron (Fe), aluminum (Al), magnesium (Mg), which can act as centers of coordination interaction with -OH and C=O groups can provide additional rigidity of the epoxy chain. No less important is the absence of toxic elements (Pb, Cd, Hg), which further indicates the environmental safety of the OBLF. Additionally, gas chromatography analysis with mass spectrometric detection of organic biogenic lignocellulosic filler was performed. The composition of low-molecular organic components present in its structure was identified, and their relative content was determined using software (Fig. 3, Table 2).

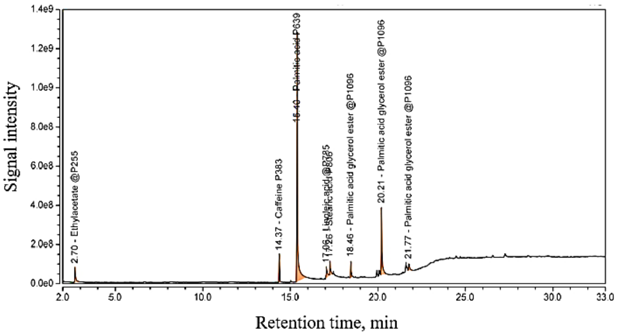

Fig. 3 – Chromatogram of organic biogenic lignocellulosic filler obtained by gas chromatography with mass spectrometric detection

Chromatogram analysis revealed a spectrum of peaks in the retention time range of 2.5…22 min, which indicates a complex multicomponent organic composition of the filler. According to the results of a library search using the NIST and MPW5e databases, the following main compounds were identified. Ethyl acetate was detected in the area of low retention times (Ret. time ≈ 2.70 min). It was believed that the presence of ethyl acetate could be a product of the technological processing of the raw materials. In the range of 14-15 min, caffeine (3.88 % of the relative peak area) was recorded, which is a characteristic marker of the coffee origin of biogenic raw materials, i.e. coffee production waste. The dominant component of the organic phase of the biogenic lignocellulosic filler is palmitic acid (59.74 %), which together with linoleic (4.15 %) and stearic acids (3.96 %) forms a fraction of saturated and unsaturated fatty acids. In addition, glycerol esters of palmitic acid (over 23 % in total) were found in the sample, which indicates the presence of lipid and wax components of biogenic origin.

Based on the analysis of the chromatogram (Fig. 3, Table 2), it can be stated that fatty acids and their esters are characterized by long aliphatic chains containing polar functional groups (–COOH, –COO–), which are able to participate in the physicochemical interaction with the epoxy matrix. From a materials science point of view, these components can perform a dual function. First of all, they can perform the function of structure-forming elements of the polymer network, as well as influence the relaxation processes and redistribution of residual stresses in the polymer material. Further, the optimal content of organic biogenic lignocellulosic filler in the epoxy binder was determined, which affects the values of physical and mechanical properties. It was experimentally established (Fig. 4, curve 1) that increasing the filler content from $q = 0.5$ wt. % to $q = 1.0$ wt. % provides an increase in the value of the ultimate bending stresses from $\sigma_b = 63.8$ MPa to $\sigma_b = 102.8$ MPa. A further increase in the filler content (over 1.0 wt. %) leads to a gradual decrease in the ultimate bending stresses. At the same time, the comparison of the results of the study of X-ray fluorescence analysis, GC-MS and variable dynamics of destructive stresses during bending allows us to state the combined effect of the mineral and organic components of the biogenic lignocellulosic filler. In particular, the identified cations Ca^{2+}, K^+, Mg^{2+} and Fe^{3+} (Table 1) can act as physicochemical centers of interphase interaction, contributing to the limitation of the mobility of the main chain and segments of the epoxy composite.

Table 2 – Results of the study of organic biogenic lignocellulosic filler by gas chromatography with mass spectrometric detection

No.	Return time, min	Name of the identified compound	Compound code	Relative area, %	Degree of library compliance, %
1	2,70	Ethyl acetate @P255	141-78-6	4,64	26,92
2	14,37	Caffeine P383	58-08-2	3,88	96,91
3	15,40	Palmitic acid P639	57-10-3	59,74	89,38
4	17,06	Linoleic acid @P785	60-33-3	4,15	92,38
5	17,26	Stearic acid P806	57-11-4	3,96	85,11
6	18,46	Glyceride (glycerol ester) of palmitic acid @P1096	23470-00-0	2,52	44,73
7	20,20	Palmitic acid glyceride @P1096	23470-00-0	18,64	98,18
8	21,77	Glycerol palmitic acid ester @P1096	23470-00-0	2,46	48,70

While fatty acids and their glycerol esters (Fig. 3, Table 2), due to the presence of polar groups –COOH– and –COO–, are able to form hydrogen and dipole-dipole bonds with hydroxyl and epoxy groups of the matrix, which ensures a uniform distribution of stresses in the polymer volume.

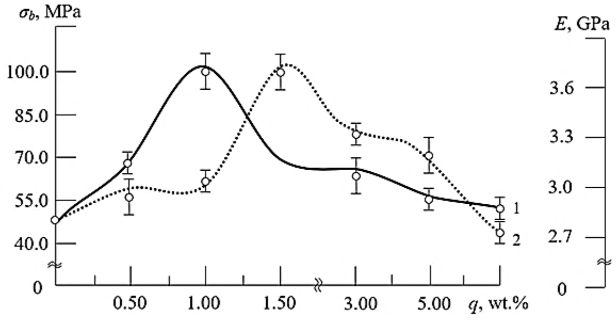

Fig. 4 – Influence of the content of organic biogenic lignocellulosic filler (OBLF) on the physical and mechanical properties of epoxy composites: 1 – bending failure stress (σ_b); 2 – bending modulus of elasticity (*E*)

Analysis of the study of the influence of the content of organic biogenic lignocellulosic filler on the value of the elastic modulus (Fig. 4, curve 2). Unlike the destructive stresses during bending, the elastic modulus (*E*) is the maximum value at a slightly higher content of OBLC. In particular, the maximum value of the elastic modulus, which is $E = 3.7$ GPa, was observed when the additive was introduced at a content of $q = 1.5$ wt. %. Similar results of the study indicate a different mechanism of the filler's influence on the stiffness and strength of the composite. It was believed that the increase in the elastic modulus is associated with the formation of a more rigid polymer structure due to the physical reinforcement of the epoxy matrix with OBLF particles, while the maximum strength values are realized under the condition of an optimal balance between the stiffness and the ability of the material to relax stresses, which is consistent with the work [19].

Additionally, it was found that the impact strength of the composites (Fig. 5, curve 1) increases from $W = 7.0$ kJ/m^2 (unfilled matrix) to $W = 16.4$ kJ/m^2 with the introduction of OBLF at a content of $q = 1.00$ wt. %. It was believed that the increase in impact strength is explained by the activation of mechanisms of microplastic deformation and dissipation of impact energy, due to the presence of lipid components in the filler structure – palmitic, linoleic, stearic acids and their esters (Table 2). Long aliphatic chains of these compounds play the role of plasticizing fragments, which reduce the degree of crack formation in the polymer volume by deviating cracks from the basic trajectory of their propagation.

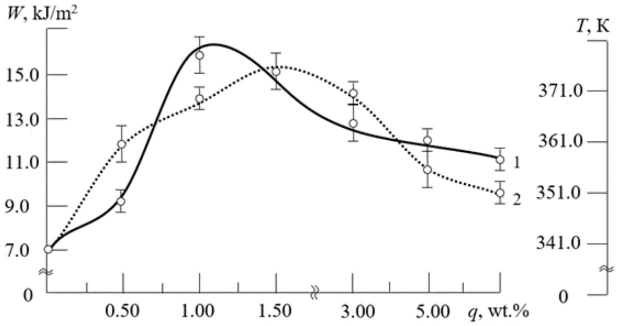

Fig. 5 – Effect of organic biogenic lignocellulosic filler (OBLF) content on the impact strength and Martens heat resistance of epoxy composites: 1 – impact strength (*W*); 2 – Martens heat resistance (*T*)

Further, the influence of the content of organic biogenic lignocellulosic filler on the values of thermophysical properties was analyzed, in particular: Martens heat resistance, thermal coefficient of linear expansion, glass transition temperature, and shrinkage.

Analysis of the curve of the dependence of the Martens heat resistance on the content of OBLF (Fig. 5, curve 2) allowed us to establish the maximum of the studied property $T = 372…368$ K at a close, but somewhat higher filler content, which is $q = 1.5…3.0$ wt.%. This indicates that at the optimal filler content, relaxation processes in the epoxy matrix slow down, and the temperature of the transition to a highly elastic state shifts to the region of higher values. While the obtained value of heat resistance ($T = 354…351$ K) at an excess content of OBLF

(q = 5.0…7.0 wt.%) can be explained by the aggregation of filler particles and an increase in the proportion of weakly bound organic components, which leads to the formation of a defective structure.

For an in-depth analysis of the influence of organic biogenic lignocellulosic filler on the deformation behavior of epoxy composites with increasing temperature, dilatometric studies were conducted (Fig. 6, Table 3), the results of which yielded temperature dependences of relative deformation and determined the thermal coefficient of linear expansion (TCLE), glass transition temperature, and shrinkage of composites (Tables 3, 4). Analysis of dilatometric curves (Fig. 6) allows us to state that the introduction of OBLF significantly changes the nature of thermal expansion of the epoxy matrix in the temperature range ΔT = 303…473 K. For the unfilled matrix, a typical character of deformation growth for amorphous polymers was observed with a sharp increase in the slope of the curve in the region of the glass transition temperature (T_c). According to the obtained research results (Table 3), in the temperature range ΔT = 303…323 K it is shown that the introduction of OBLF at a content of q = 1.0…1.5 wt. % leads to a significant decrease in the value of the TCLE from $\alpha = 2{,}50 \cdot 10^{-5}$ to α = 1.71…1,66 10^{-5} К$^{-1}$.

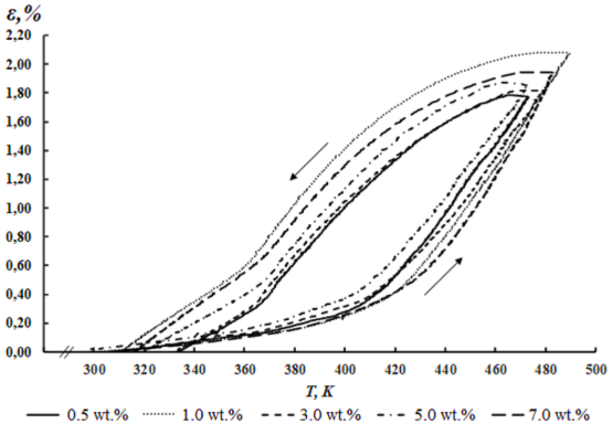

Fig. 5 – Indicators of dilatometric curves of epoxy composites depending on the change in the content of biogenic lignocellulosic filler

Table 3 – Values of the thermal coefficient of linear expansion of composite materials filled with organic biogenic lignocellulosic filler

Content of organic biogenic lignocellulosic filler (OBLF), q, wt. %.	Thermal coefficient of linear expansion, $\alpha \times 10^{-5}$, К$^{-1}$			
	Test temperature ranges, ΔT, К			
	303…323	303…373	303…423	303…473
Matrix	2.50	2.74	4.96	9.78
0.5	1.71	2.22	4.94	9.81
1.0	1.66	2.00	3.86	9.87
1.5	1.66	2.00	3.86	9.87
3.0	1.90	2.56	4.96	9.92
5.0	3.39	3.00	5.89	9.99
7.0	1.39	1.87	3.70	9.23

It was believed that the decrease in the TCLE value is due to the limitation of the segmental mobility of epoxy chains in the polymer volume, due to the presence of polar organic components (fatty acids and their esters) and mineral ions (Ca^{2+}, Mg^{2+}, Fe^{3+}), which provide physicochemical interaction between the polymer ingredients. When analyzing the TCLE value in wider temperature ranges (ΔT = 303…373 K and ΔT = 303…423 K), a similar pattern of change in linear dimensions was observed. The minimum values of TCLE (Table 3) are also characteristic of composites with a content of q = 1.0…1.5 wt. %.

While a further increase in the content of OBLF (q = 3.0…5.0 wt. %) provides an increase in the value of TKLE, which is associated with a violation of the homogeneity of the structure and the appearance of zones with increased local mobility of polymer chains. It should be noted that the results of the study correlate with mechanical tests, where an excess filler content leads to a decrease in strength characteristics.

It was also established that the glass transition temperature (T_c) of epoxy composites depends on the content of OBLC (Table 4). For the epoxy matrix, T_c is 320 K. The introduction of a filler into the epoxy binder at a content of q = 1.0…1.5 wt. % provides an increase in the glass transition temperature to 342.7 K, which indicates an increase in the effective crosslinking density and a decrease in the free volume of the polymer matrix.

Table 4 – Thermophysical properties of composite materials

No.	Characteristics	Organic biogenic lignocellulosic filler (OBLF), q, wt.%						
		Matrix	0.5	1.0	1.5	3.0	5.0	7.0
1	Glass transition temperature, T_c, К	320.0	320.5	342.7	342.7	338.5	331.7	328.1
2	Shrinking, ΔL, %	0.35	0.017	0.017	0.017	0.018	0.018	0.020

The obtained results are consistent with the hypothesis of the structure-forming role of organic components of OBLF [20]. Polar functional groups of fatty acids and glycerol esters of the filler are characterized by the ability to intermolecular interaction with active groups of the epoxy oligomer ED-20, forming additional physical nodes during polymer crosslinking. With an increase in the filler content q = 3.0…7.0 wt. %. the glass transition temperature decreases (to T_c = 338.5…328.1 K), which indicates the predominance of the plasticizing effect of the organic phase over the structure-forming one, as well as the possible aggregation of filler particles.

Additionally, it is shown (Table 4) that a significant reduction in shrinkage of epoxy composites filled with OBLF is due to the implementation of a compensation-structural mechanism, in which dispersed filler particles are integrated into the polymer matrix and limit the development of volumetric deformations during polymerization. While the presence of lipid components in the filler composition ensures relaxation of residual stresses at the stage of polymer network formation.

4. CONCLUSIONS

Based on comprehensive research, the following results were obtained:

1. The complex mineral-organic composition of biogenic lignocellulosic filler (BLCF) was established by X-ray fluorescence analysis and gas chromatography with mass spectrometric detection. It was shown that BLCF contains cations Ca^{2+}, K^+, Mg^{2+}, Fe^{3+} and organic components of lipid nature (fatty acids and their glycerol esters), which determine its functional activity in the composition of epoxy composites.

2. The optimal content of OBLF in the epoxy binder was experimentally established, which is $q = 1.0\ldots1.5$ wt. %, at which an increase in the value of physical and mechanical characteristics was observed. In particular, the destructive stresses during bending increase from $\sigma_b = 48.0$ MPa to $\sigma_b = 102.8$ MPa, and the elastic modulus from $E = 2.7$ GPa to $E = 3.7$ GPa, which indicates effective physical reinforcement of the polymer matrix and interfacial interaction of the polymer ingredients.

3. It is shown that the increase in the impact strength of epoxy composites from $W = 7.0$ kJ/m^2 to $W = 16.4$ kJ/m^2 is due to the activation of the mechanisms of microplastic deformation and dissipation of impact energy, which are realized due to the presence of lipid components with long aliphatic chains in the composition of the organic biogenic lignocellulosic filler, capable of reducing the intensity of crack formation in the polymer volume.

4. Based on the analysis of dilatometric curves, it was found that the introduction of biogenic lignocellulosic filler at a content of $q = 1.0\ldots1.5$ wt. % is accompanied by a decrease in the derivative $\Delta\varepsilon/\Delta T$, which corresponds to a decrease in the thermal coefficient of linear expansion of the epoxy matrix. Additionally, a shift of dilatometric curves to the region of higher temperatures was established, which ensures an increase in the glass transition temperature from $T_c = 320.0$ K to $T_c = 342.7$ K. The resulting set of effects indicates a restructuring of the supramolecular organization of the epoxy matrix with a transition to a structurally ordered three-dimensional polymer network, in which an increase in the effective crosslinking density and a decrease in the free volume are due to the formation of a developed near-surface interphase region with limited segmental mobility of polymer chains.

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Структурно-хімічні особливості епоксидних композитів, наповнених біогенним лігноцелюлозним наповнювачем, отриманим з агропромислових відходів

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У роботі досліджено вплив органічного біогенного мікродисперсного лігноцелюлозного наповнювача на фізико-механічні та теплофізичні властивості епоксидних композитів. Приділено увагу аналізу структурних і міжфазових ефектів, що реалізуються на мікро- та нанорівнях і визначають експлуатаційні характеристики матеріалів. Елементний склад наповнювача досліджено методом рентгенофлуоресцентного аналізу, що дозволило ідентифікувати мінеральні компоненти, здатні формувати активні центри які забезпечують міжфазову взаємодію з епоксидною матрицею. Органічну складову наповнювача досліджено методом газової хроматографії з мас-спектрометричним детектуванням. При цьому виявлено наявність жирних кислот та їх гліцеринових естерів із полярними функціональними групами, здатними брати участь у міжмолекулярній взаємодії. Доведено, що введення наповнювача за вмісту $q = 1,0…1,5$ мас.ч. забезпечує підвищення руйнівних напружень при згинанні, модуля пружності, ударної в'язкості епоксидних композитів та комплексу теплофізичних властивостей. Це пов'язано із дією різних механізмів, зокрема фізичного армування та релаксації напружень в об'ємі полімеру. Отримані результати комплексних досліджень підтверджують, що біогенний лігноцелюлозний наповнювач виконує не лише роль дисперсного армувального компонента, а й функцію структуроутворюючого елемента, який забезпечує спрямоване керування властивостями епоксидних композитів за рахунок нанорівневої міжфазової взаємодії. Це відкриває перспективи створення функціональних полімерних матеріалів із прогнозованими фізико-механічними та теплофізичними характеристиками.

Ключові слова: Епоксидні нанокомпозити, Покриття, Біогенний наповнювач, Епоксидна смола, Газова хроматографія, Рентгенофлуоресцентний аналіз, Наноструктури, Експлуатаційні характеристики.