sciendo

Management Systems in Production Engineering

2025, Volume 33, Issue 4, pp. 576-585

Date of submission of the article to the Editor: 04/2025
Date of acceptance of the article by the Editor: 10/2025

DOI 10.2478/mspe-2025-0060

IMPROVEMENT OF THERMOPHYSICAL CHARACTERISTICS OF POLYMER COATINGS FOR PROTECTING WATER TRANSPORT BY USING A MIXTURE OF DISCRETE ORGANIC FIBERS

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Abstract:
This work presents the technological aspects of the reinforcement of the thermosetting binder to improve the thermophysical characteristics of polymeric materials and protective coatings based on them. Epoxy binder ED-20 was used to form polymer materials, which were polymerized with polyethylene polyamine (PEPA) in the ratio: 100 parts by weight of ED-20 epoxy oligomer and 10 parts by weight of PEPA hardener. A mixture of discrete organic fibers with the content of q = 0.25-2.00 wt.% was used to improve the thermophysical properties of polymer materials. The key parameters for determining the temperature range for the future operation of the designed composite materials without changing their properties were defined by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). The maximum temperature of the mass loss beginning is T0 = 624.0°K; the relative mass loss is εm = 65.7%; the initial temperature of the exoeffect is Tn = 475.5°K; and the maximum temperature of the exoeffect peak is Tmax = 545.5°K. The mathematical calculation of the activation energy of thermal destruction was performed to determine the resistance to the degradation of chemical bonds under the temperature influence. It has been proven that composites filled with a discrete organic fiber mixture with the content of q = 0.75 wt.% are characterized by the maximum activation energy (Ea = 167.2 kJ/mol), which indicates the thermal stability of reinforced composites.

Key words: epoxy binder, hardener, a mixture of discrete organic fibers, polymer material, heat resistance, destruction, relative mass loss, activation energy

INTRODUCTION

With the development of the polymer materials manufacturing industry and the expansion of their application areas, increased requirements are being put forward for the properties of polymer materials and coatings based on them, especially those used in the transport industry [1, 2, 3, 4, 5, 6, 7, 8]. The expansion of the functionality of polymer materials is possible through using a range of additives (modifiers, fillers, plasticizers), which allow the obtaining of composite materials for functional purposes. At the same time, their large-scale production and use in many industries is associated with one of the advantages – the possibility of creating structural elements and protective coatings with specified properties [9, 10]. Improving the properties of epoxy composite materials (CM) is possible by introducing fillers of different physicochemical nature and dispersion into the volume of the polymer matrix. The use of fibrous additives as reinforcing materials is due to improving the complex properties under the condition of a rational combination of components. For example, the reinforcement of the polymer matrix with high-strength carbon, boron, organic, and glass fibers increases specific strength and stiffness, which is several times higher than traditional metals and alloys, and

improves operational reliability [11, 12, 13, 14]. The technological line for the production of polymer materials is characterized by approximately threefold energy savings (compared to metals and alloys) per kilogram of KM. However, despite the achievements in the field of reinforced composite materials, there is a need to develop new composites and coatings based on them, which are not inferior in properties and cost to domestic and foreign analogues.

Considering the above, this work aimed to determine the influence of the content of a mixture of discrete organic fibers based on cotton (52%) and polyester (48%) on the parameters of the thermophysical properties of polymer CMs.

Simultaneously, to achieve this goal, the authors provide:

- based on the results of thermogravimetric analysis (TGA), determine the initial temperature of mass loss of advanced polymer CMs filled with a mixture of discrete fibers, as well as the temperatures at which mass loss of composites occurs (by 5%, 10%, 20%);
- perform a mathematical calculation of the activation energy of thermal destruction to determine the stability of chemical bonds in polymer CMs to conclude their resistance to thermal effects;
- based on differential thermal analysis (DTA) data, determine the initial temperature and the maximum exoeffect of the designed polymer CMs filled with a mixture of discrete fibers to establish their critical operating temperature.

LITERATURE REVIEW

The demand for the use of polymer composite materials (CMs) has increased significantly in many industries. Considering the multifunctionality of polymers and the complex of improved properties [15], they can be used as individual products [16, 17, 18], as coatings for various functional purposes [19, 20, 21, 22, 23, 24], and as adhesive materials for transport [25, 26, 27, 28, 29], in particular, for water transport. Thus, in [30], it was shown that the increase in impact toughness by 55%-60% and the increase in the time of the CM crack propagation to the moment of failure by 15%-20% (compared with unreinforced polymer material) is associated with the self-regulation of the structure of composites filled with discrete fibers. It makes it possible to use such materials to form coatings resistant to impact loads for vehicles. The authors of works [31, 32] showed that increasing the content of one of the components in the volume of the polymer matrix provides a change in the kinetic parameters of the polymer system, which in turn provides a change in both the structure and the physical and mechanical properties of filled epoxy composite materials. It has been shown [33, 34] that the modification of carbon nanotubes (CNTs) ensures the formation of polar functional groups. The additional combination of them with fibrous additives improves their mechanical properties (relative to unreinforced polymer material); in particular, the modulus of elasticity increases by 6%-8% [35, 36], and the bending strength limit is 40%-42% [37]. The authors of the work [38] obtained polymer CMs with natural-reinforcing materials (fabrics made of linen, cotton, hemp, cattail leaves, and wheat straw) for protecting aluminium panels operating under the conditions of impact loads, tensile loads, and peel adhesion.

Simultaneously, the exploitation of vehicles under different climatic conditions requires the development of composites with not only a high mechanical strength [35]. New CMs with possible usage at variable temperatures (low and elevated ones) are needed [34].

It was shown [34, 35] that the combination of CNTs with fibrous fillers provides high parameters of mechanical characteristics in the temperature range of -40-80°C. This fact is associated with additional radial compression by the matrix material of the nano-disperse component due to different coefficients of linear expansion and increased interphase interaction [39]. At elevated temperatures, the main disadvantage of polymer materials is the ignition of products based on them [40]. The authors of studies [41, 42] showed that using fibrous additives and reactive plastic binders (poly-ester, epoxy) in forming heat-insulating sandwich panels for water transport is quite effective and provides a significant reduction in the flammability of such materials.

At the same time, thermosetting polymers filled with fibrous additives have certain disadvantages: the delamination of coatings due to water absorption of reinforcing fibers and a narrow range of operating temperatures. The existing shortcomings of such polymer materials make it necessary to find ways to regulate the thermophysical properties of CMs. In this direction, it is advisable to apply the principle of poly-reinforcement, connecting fibers of different physicochemical natures and geometric parameters in a thermosetting binder. This, in turn, will make it possible to change the structure and, therefore, improve the thermophysical properties of the polymer material.

METHODS, RESULTS AND DISCUSSION

The main component for the binder in the formation of the CM was selected epoxy diane oligomer brand ED-20 (ISO 18280:2010, Technobudresurs, Kyiv, Ukraine), which is characterized by a complex of improved properties compared to other known reactive plastics [43, 44].

A polyethylene polyamine (PEPA) hardener (TU 6-05-241-202-78, Techno-budresurs, Kyiv, Ukraine) was used to cross-link epoxy compositions, which allows materials to be cross-linked at room temperature [45, 46].

A mixture of discrete organic fibers (Nortex, Republic of Belarus, Minsk district) based on cotton and polyester (MDOFCP) was used as a filler, where the content of the natural component (cotton) prevails. The choice of a fibrous filler is related to its cost. Since the fibrous filler is a waste of sewing production, its cost is insignificant, which is reflected in the cost of a protective coating as the final product.

The composition and parameters of MDOFCP are as follows: 52% cotton (natural component), 48% polyester (synthetic component), l = 15-30 mm, d = 20-25 μm.

Note that cotton fibers are characterized by low density and ability to biological degradation. They can provide resistance to the action of alkalis and solutions of inorganic salts. They also have low thermal conductivity and high resistance to temperature effects. At the same time, polyester is characterized by elasticity and increased strength.

To improve the degree of wetting of the mixture of organic fibers and, therefore, the interphase interaction of the "polymer-fiber" system, epoxy composites were formed according to the following technology:

- preliminary dosing of ED-20 epoxy diene resin, heating the resin to a temperature of T = 353±2°K and keeping it at this temperature for τ = 20±0.1 min;
- dosing a mixture of discrete fibers;
- adding a mixture of discrete fibers into the composition in the following ratio: 50% of the additive to the epoxy binder, 50% of the additive to the PEPA hardener;
- a mechanical combination of oligomer ED-20 and a mixture of discrete fibers during time τ = 1±0.1 min;
- ultrasonic treatment (UT) of the composition during the time τ_3 = 1,5±0,1 min;
- cooling the composition to room temperature during time τ = 60±5 min;
- mechanical combination of the PEPA hardener and a mixture of discrete fibers during time τ = 1±0.1 min;
- ultrasonic treatment (UT) of the composition during the time τ_3 = 1,5±0,1 min;
- mixing of two compositions (ED-20 with a mixture of discrete fibers and PEPA with a mixture of discrete fibers) during the time τ = 5±0.1 min.

Then, hardening of the CM was performed to the experimentally established regime [47, 48]: the formation of samples and their holding for a time of τ = 12.0±0.1 h at a temperature of T = 293±2°K, heating at a rate of υ = 3°K/min to the temperature T = 393±2°K, holding the CM for a time of τ = 2.0±0.05 h, slow cooling to a temperature of T = 293±2°K. In order to stabilize the structural processes in the matrix, the samples were kept for a time of τ = 24 h in the air at a temperature of T = 293±2°K followed by experimental tests.

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of the CM samples, with the usage of the derivatograph "Thermoscan-2," were performed for the study of the filler nature influence on thermal transformations in composites. The research was conducted in the temperature range ΔT = 298-873°K using quartz crucibles for samples (V = 0.5 cm^3). Al_2O_3 (m = 0.5 g) was used as the reference substance. The mass of the test sample was m = 0.3 g, and the accuracy of determining the change in the sample mass was Δm = 0.02 g. The rate of temperature increase during the study was υ = 5°K/min with the error in temperature determination ΔT = ±1°K. The accuracy of the determining thermal effects was 3 J/g.

The activation energy calculation was carried out by mathematical processing of the TGA-curve according to the Broido method [49, 50, 51]. The condition for using this method is the first-order decomposition reaction, which applies to both thermosetting and thermoplastic polymers. The mass loss of a substance is a first-order process (n = 1) if the linear dependence of ln(100/(100 – Δm)) on the inverse temperature 103/T, °K^{-1} is maintained. According to this method, it is necessary to determine the mass loss (Δm) of the composite at a given temperature T and graphically construct a line in which E must be expressed by the tangent of the slope of the logarithmic dependence Δm on the inverse temperature T. Then, the value of the activation energy of thermal destruction (kJ/mol) can be found by the formula:

$$\mathrm{E}_a = -R \cdot \mathrm{tg}(\varphi). \quad (1)$$

To graphically determine the activation energy of thermal destruction, the graph should have a straight line, the tangent of the angle of inclination ϕ of which it is possible to calculate the activation energy E_a (Fig. 1).

Then,

$$-\mathrm{tg}(\varphi) = \frac{y_i}{x_i}, \quad (2)$$

$$E = R \cdot \frac{y_i}{x_i}, \quad (3)$$

where:

$x_i = x_{\text{init}} - x_{\text{fin}}$ – the length of the line along the abscissa;

$y_i = y_{\text{init}} - y_{\text{fin}}$ – the length of the line along the ordinate;

$[x_{\text{init}}; y_{\text{init}}]$ and $[x_{\text{fin}}; y_{\text{fin}}]$ – coordinates of the beginning and end of the line, respectively.

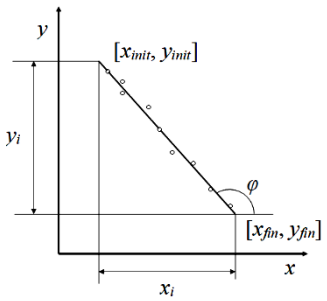

Fig. 1 To graphically determination of the activation energy

Thermogravimetric analysis (TGA) of composites filled with a mixture of discrete organic fibers. The ability of a polymer material or a protective coating based on a thermoset matrix to work at different temperature ranges allows expanding the areas of their application in transport significantly. Therefore, we studied the behavior of composite materials filled with a mixture of discrete fibers in the temperature range T = 293-873°K by TGA (Fig. 2, a-e, curve 1). To study the complete process of bond decomposition in the polymer, a temperature rise rate of 5°K/min was chosen. The content of the MDOFCP was varied within q = 0.25-2.00 wt.%.

It was shown [33] that the unfilled epoxy matrix is characterized by the following thermophysical parameters determined based on the analysis of the TGA curves: T_0 = 587.9°K, T_5 = 616.0°K, T_{10} = 625.0°K, T_{20} = 641.0°K, T_K = 723.0°K, ε_m = 80.7%. The analysis of the TGA curves (Figure 2, curve 1, Table 1) made it possible determination the initial temperature at which the destruction of CM occurs.

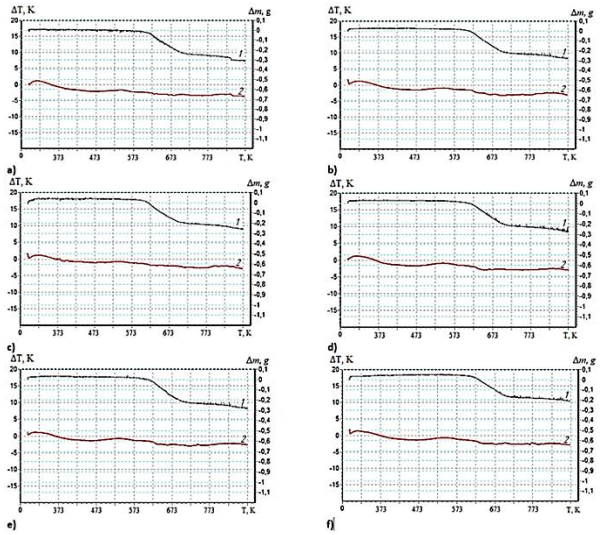

Fig. 2 Results of thermogravimetric (1) and differential-thermal (2) of composites materials filled with a mixture of discrete organic fibers: a) 0.25 wt.%; b) 0.50 wt.%; c) 0.75 wt.%; d) 1.00 wt.%; e) 1.50 wt.%; f) 2.00 wt.%

Table 1

Heat resistance of composites with different content of the mixture of discrete fibers

Content of a Mixture of Discrete Fibres (MDOFCP), *q*, wt.%	T_0, °K	T_5, °K	T_{10}, °K	T_{20}, °K	T_f, °K	ε_m, %
0.25	605.7	617.0	627.3	646.6	732.2	76.7
0.50	616.5	617.1	629.2	641.7	749,1	63.7
0.75	624.0	628.5	634.6	650.1	754.5	63.7
1.00	621.1	620.3	630.9	642.8	752.7	66.3
1.50	621.6	620.4	631.1	642.9	752.7	69.7
2.00	613.0	621.7	634.1	640.6	743.3	67.7

Note: *T_0 – is the initial temperature of the mass loss (the beginning of destruction); T_5, T_{10}, T_{20} – are the mass loss temperatures (5%, 10%, 20%); T_f – is the final mass loss temperature (completion of destruction); ε_m – is the relative mass loss*

The lowest temperature at which the destruction begins is characterized for the CM filled with MDOFCP with the content of q = 0.25 wt.%, which is higher by ΔT_0 = 17.8°K compared to the unfilled epoxy matrix [33]. The obtained T_0 indicates the active effect of the filler. Its influence is determined by the resistance of physico-chemical bonds to the influence of temperature and, therefore, the ability to inhibit the process of thermal destruction of the fibrous component. Simultaneously, the highest temperature (among the investigated CMs) at which the destruction of polymers begins is characterized for CMs filled by MDOFCP with the content of q = 0.75 wt.% (Table 1).

The temperature of the beginning of mass loss for such materials is T_0 = 624.0°K, which is higher by ΔT_0 = 36.1°K compared to the unfilled epoxy matrix. It was believed that a significant shift in the temperature of the beginning of mass loss to the region of higher temperatures is associated with the limitation of mobility and deformation of the kinetic elements in the CM structure (segments of the polymer network and the main chain) under the temperature influence. This, in turn, creates conditions for the redistribution of internal energy due to the partial restriction of mobility and, therefore, the flexibility of macromolecules.

At the same time, the lower the flexibility and mobility of the main chain and its components, the greater the potential barrier to its rotation. As a result, the higher the temperature at which structural changes occur. Moreover, intense destruction (T = 5...20%°K) with breaking of the maximum number of bonds in the CM structure was observed in the temperature range ΔT = 617-650°K. Simultaneously, the most stable temperature increase step in the temperature range with observed mass loss (5%, 10%, 20%), was defined for the CM filled by MDOFCP with the content of q = 0.75 wt.% (Table 1). This result indicates the polymer's thermodynamic balancing due to the material's structural homogeneity and, therefore, a high degree of cross-linking. Additionally, the authors of the works [52, 53] showed that fibers based on a natural component (cotton) have high surface energy and can also form ether and hydrogen bonds during polymer cross-linking. Obviously, this can explain the increased resistance to temperature effects of composites with optimal content of a discrete fiber mixture. The obtained relative mass loss confirms these positions. The smallest relative mass loss, which is ε_m = 63.7%, is characterized for the CM with MDOFCP with the content of q = 0.75 wt.% (Table 1). Note that although the final temperature of mass loss does not significantly affect the operational performance of the developed materials, the maximum temperature TK = 754°K for the end of the destruction process is characterised for the CM filled by MDOFCP with the content of q = 0.75 wt.%.

So, the initial temperature of mass loss of the designed polymer CMs was determined based on thermogravimetric analysis (TGA). The highest temperature at which the beginning of destruction was observed is defined for the CM filled with a mixture of discrete fibers with the content of q = 0.75 wt.%. The high degree of cross-linking of such composites provides inhibition of thermal destruction. It is additionally confirmed by the obtained temperature values at which mass loss occurs by 5%, 10%, and 20%, namely: T_5 = 628.5°K, T_{10} = 634.6 K, T_{20} = 650.1°K.

Calculation of the activation energy of thermal destruction of composite materials filled with discrete organic fibers.

To confirm the results of TGA, namely, the effect of MDOFCP on the heat resistance of CM, the activation energy of thermal destruction was additionally calculated. The obtained results of mathematical calculations will make it possible to estimate the degree of cross-linking of composite materials filled with MDOFCP. It is because the activation energy can be described as the excess of thermal energy necessary for destructing chemical bonds under thermal conditions in the presence of oxygen. The activation energy of thermal destruction was determined using the TGA curves (Fig. 2, curve 1) according to the Broido method [49, 50, 51]. Note that the most intensive course of the thermal destruction reaction for filled CMs was marked in the temperature range ΔT = 605-754°K (Fig. 2, curve 1, Table 1). It corresponds to a mass loss of

T_5...90%°K. Considering above, the temperature range ΔT = 573-713°K was selected for the study of the TGA curves, which is necessary for determining the activation energy of the studied composites. Previously, the mass loss of materials was determined in the specified temperature range with an interval of ΔT = 10°K (Fig. 3) for the CM filled by MDOFCP with the content of q = 0.25 wt.%.

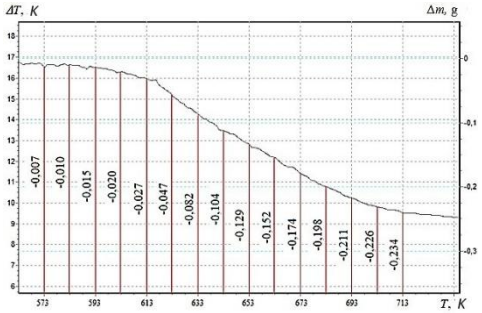

Fig. 3 The mass loss of the composite material filled by MDOFCP (at the content of q = 0.25 wt.%), with the interval of ΔT = 10°K in the temperature range ΔT = 573-713°K

Similarly, the measurements were conducted for CMs filled by MDOFCP with the content of q = 0.50-2.00 wt.% (Table 2, 3, 4, 5).

The mass value of the studied composite material was calculated as a percentage using formula 4:

$$(100 - \Delta m)\% = \left(100 - \left(\frac{m_{in}+\Delta m}{\Delta m} \cdot 100\right)\right)\%, \qquad (4)$$

where:

min – initial sample mass at initial tested temperature T_1 = 573°K (min = const), g;

Δm – sample mass loss, g.

The mass of the composite material at the initial temperature was taken as 100%. Tables 2 and 3 show the results of processing the TGA curve and the parameters necessary to calculate the activation energy of composites filled by MDOFCP.

Table 2

The results of processing the composites TGA curves

T, °K	Sample mass, g (according to TGA curves)					
	Content of a Mixture of Discrete Organic Fibers, q, wt.%					
	0.25	**0.50**	**0.75**	**0.10**	**1.50**	**2.00**
573	0.29	0.33	0.32	0.32	0.32	0.35
583	0.29	0.33	0.32	0.32	0.31	0.34
593	0.29	0.32	0.31	0.31	0.31	0.34
603	0.28	0.32	0.30	0.31	0.31	0.34
613	0.27	0.30	0.29	0.30	0.30	0.33
623	0.25	0.28	0.27	0.28	0.28	0.32
633	0.22	0.25	0.25	0.25	0.26	0.29
643	0.20	0.23	0.22	0.22	0.23	0.27
653	0.17	0.20	0.20	0.20	0.21	0.25
663	0.15	0.19	0.15	0.17	0.18	0.22
673	0.13	0.16	0.12	0.15	0.16	0.20
683	0.10	0.14	0.11	0.12	0.14	0.18
693	0.09	0.12	0,10	0.10	0.11	0.15
703	0.07	0.11	0.09	0.09	0.10	0.14
713	0.07	0.10	0.09	0.08	0.09	0.13

The calculation of the activation energy is based on the mathematical processing of the TGA curve using this dependence [53, 54]:

$$\ln\left(\ln\frac{100}{100-\Delta m}\right) = -\frac{E}{R}\cdot\frac{1}{T} + const, \qquad (5)$$

where:

Δm – is the loss of sample mass, g;

E – is the activation energy, kJ/mol;

R – is the universal gas constant, R = 8.31 J/mol °K;

T – is the temperature, °K.

Table 3

The results of processing the TGA curve

T, °K	Change of the sample mass (100-Δm),%					
	Content of a Mixture of Discrete Organic Fibers, q, wt.%					
	0.25	**0.50**	**0.75**	**0.10**	**1.50**	**2.00**
573	2.33	-10.34	-6.21	-6.21	-5.94	-14.06
583	3.33	-10.34	-6.21	-5.17	-4.38	-13.44
593	5.00	-7.93	-1.72	-3.45	-3.13	-13.13
603	6.67	-6.21	0.34	-3.10	-1.88	-11.56
613	9.00	-1.03	2.41	0.34	0.31	-9.38
623	15.67	7.59	9.31	6.90	5.63	-5.00
633	27.33	17.93	18.62	16.55	13.13	2.19
643	34.67	25.52	26.90	26.55	21.25	9.69
653	43.00	34.14	35.86	35.86	28.44	17.19
663	50.67	39.31	50.69	44.48	36.88	24.38
673	58.00	47.93	62.07	52.76	44.69	31.25
683	66.00	54.83	65.86	61.38	51.56	38.75
693	70.33	60.69	68.62	69.31	58.75	45.63
703	75.33	65.17	72.76	73.79	62.50	51.56
713	78.00	67.59	72.76	76.90	65.63	53.13

The results of the double logarithm value calculations of the mass change in the samples are given in the Table. 4.

Table 4

The logarithmic dependence of Δm on the reciprocal temperature $10^3/T$, °K^{-1} at a thermal breakdown of composites materials filled with a mixture of discrete fibers

T, °K	ln{ln[100/(100-Δm)]}					
	Content of a Mixture of Discrete Organic Fibers, q, wt.%					
	0.25	**0.50**	**0.75**	**0.10**	**1.50**	**2.00**
573	-3.746	–	–	–	–	–
583	-3.384	–	–	–	–	–
593	-2.970	–	–	–	–	–
603	-2.674	–	–	–	–	–
613	-2.361	–	-3.712	-5.668	-5.767	–
623	-1.770	-2.540	-2.326	-2.639	-2.849	–
633	-1.142	-1.621	-1.580	-1.710	-1.961	-3.811
643	-0.854	-1.222	-1.161	-1.176	-1.432	-2.284
653	-0.576	-0.873	-0.812	-0.812	-1.095	-1.668
663	-0.347	-0.694	-0.347	-0.530	-0.776	-1.275
673	-0.142	-0.427	-0.031	-0.288	-0.524	-0.982
683	0.076	-0.230	0.072	-0.050	-0.322	-0.713
693	0.195	-0.069	0.148	0.167	-0.122	-0.496
703	0.336	0.053	0.263	0.292	-0.019	-0.322
713	0.415	0.119	0.263	0.382	0.066	-0.277

Using the data about the mass loss (Δm) of composites filled with MDOFCP at temperature T, a straight line was graphically constructed, in which E was determined by the tangent of the slope angle of the logarithmic dependence

Δ*m* on the inverse temperature *T*. The destruction activation energy in kJ/mol was found by the formula (1), which is indicated above. Figure 4 shows the graphical dependence of the destruction rate on the inverse temperature. The analytical results of the graphical determination of the activation energy for the designed CMs are shown in Table 5.

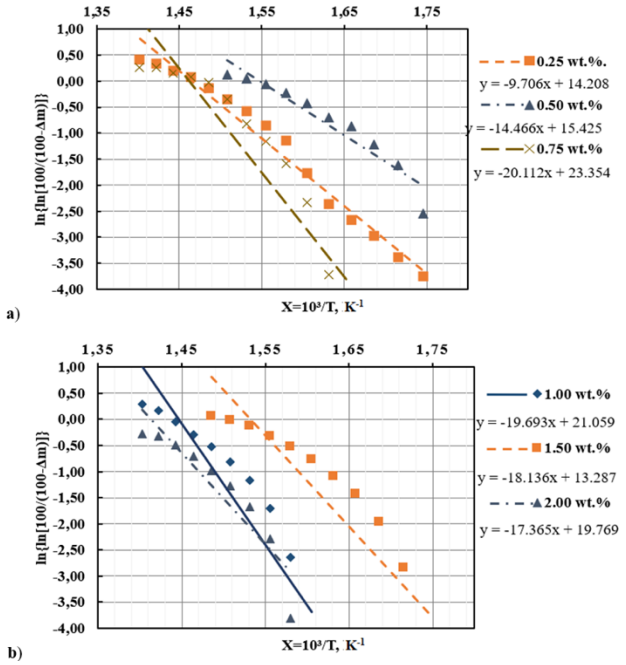

Fig. 4 Graphical dependence of the epoxy composites materials filled with a mixture of discrete organic fiber destruction rate on the inverse temperature

Table 5

Calculated activation energy value in thermal destruction of composites materials filled with a mixture of discrete organic fibers

Content of a Mixture of Discrete Organic Fibers, *q*, wt.%	X_{HD}	X_k	X_i	Y_H	Y_k	Y_i	$tq_{(\phi)}$	Activation energy E_a, kJ/mol
0.25	1.745	1.403	0.342	0.591	-2.729	3.319	9.706	80.694
0.50	1.605	1.403	0.202	0.591	-7.790	2.922	14.464	120.254
0.75	1.658	1.403	0.255	-4.863	-9.992	5.129	20.112	167.211
1.00	1.631	1.403	0.228	-6.5703	-11.060	4.490	19.693	163.728
1.50	1.631	1.403	0.228	-12.158	-16.293	4.135	18.136	150.785
2.00	1.580	1.403	0.177	-4.5941	-7.6677	3.074	17.365	144.373

Therefore, according to the Broido method, the activation energy of thermal destruction of composites filled with MDOFCP was calculated. It was considered that increasing the activation energy indicates a decrease in the velocity of the thermal destruction process of CMs.

It has been experimentally proved that the highest activation energy (E_a = 167.2 kJ/mol) for the studied materials is required for the thermal destruction of the designed composite filled by MDOFCP with the content of *q* = 0.75 wt.% (Table 5). This testifies to the resistance of physico-chemical bonds to the influence of raised temperature due to the reduction of the segmental mobility of structural elements of the polymer. Simultaneously, it is necessary to indicate the existing correlation of the mathematical calculations with the obtained results of the TGA experiments on the effect of a discrete fiber mixture on the thermal destruction process. This testifies to the results' reliability and confirmation of the given provisions.

Differential thermal analysis (DTA) of composites filled with a discrete fiber mixture. Based on previous research results [33, 54], it was determined that structural processes that occur before the moment T_0 (before the temperature of the beginning of mass loss) are significant during the exploitation of the polymeric material in conditions under the influence of increased temperatures. Therefore, we analyzed the DTA curve (Fig. 2, curve 2) in the temperature range ΔT = 303-605°K. It was defined that the temperature interval of the initial temperature of the exoeffects for CMs filled with MDOFCP is T_n = 472.2-475.5°K, which by ΔT_n = 12-15°K was more than the similar interval for the unfilled epoxy matrix [33, 54, 55, 56]. That is, it was shown that the beginning of the thermal, structural processes, such as the mobility and deformation of macromolecules and polymer segments, starts in the analogous temperature range (Table 6).

Table 6

Temperature intervals of exoeffects of CMs filled with a MDOFCP obtained by DTA

Content of a Mixture of Discrete Organic Fibres, *q*, wt.%	Temperature intervals of exoeffects				Maximal value of exoeffects, T_{max}, °K
	T_{init}, °K	T_f', °K	ΔT_1, °K	ΔT_2, °K	
0.25	472.7	652.7	180.0	1.23	542.6
0.50	472.2	641.5	169.3	1.22	528.7
0.75	475.5	669.4	193.9	2.10	545.5
1.00	472.6	666.2	193.6	2.09	542.2
1.50	472.9	656.8	183.9	2.05	543.2
2.00	462.8	665.7	202.9	2.02	543.0

Note: *T_{init} – is the initial temperature of exoeffects; T_f – is the final temperature of exoeffects; ΔT_1 – is the temperature interval of exoeffect; ΔT_2 – is the temperature difference between the sample in which the transformations take place and the standard in which there are no transformations.*

The differential thermal analysis was adequate for determining the initial and maximum temperature of the exoeffect, which allowed substantiating the possibility of using the designed composite materials in the range up to critical operating temperatures, i.e., up to temperature *T* = 545.5°K.

Simultaneously, the initial temperature of the CM exoeffects determined by DTA did not coincide with the parameters of the initial temperature of mass loss T_0 (the TGA curve). Thus, the mass loss absence makes it possible to

use the designed composites filled with MDOFCP (regardless of the content) without changing their properties in the temperature range T_n = 303.0-475.5°K.

Additionally, it was shown [33] that the founded temperature range with the maximum of exothermic effects (T_{max}) for composites mainly characterizes the partial mobility and deformation of polymer segments (proved by IR spectroscopy) [57]. Namely, significant structural processes (the destruction of bonds) do not occur. The temperature maximum of the peak of the exoeffect (T_{max} = 545.5°K) was defined for the CM filled by MDOFCP with the content of q = 0.75 wt.%. This result indicates an increased degree of CMs cross-linking and, therefore, increased stability of physico-chemical bonds to the temperature influence. Thus, the temperature range of the composite (with MDOFCP) exploitation with the content of q = 0.75 wt.% can be raised. That is, CMs can be used in the temperature range ΔT = 475.5-545.5°K. It is essential to pay attention to the final temperature of the exoeffect (T_k', °K). The use of polymeric materials at this temperature interval is limited because of the destruction of -NH-, -CH- groups, primary amino groups -NH_2, CH_2-NH_2 [33, 54, 55, 56, 57]. Simultaneously, we note that the maximum final temperature of the exoeffect (T_k' = 669.4°K) is observed for the CM filled by MDOFCP with the content of q = 0.75 wt.%. The obtained research results confirm the above assumptions.

CONCLUSIONS

Bridge The mass change of the polymer composite materials upon heating was determined by thermogravimetric analysis (TGA), which made it possible to evaluate the effect of a discrete fiber mixture on the ability to inhibit thermal destruction. It has been proven that composites filled with a MDOFCP based on cotton and polyester with the content of q = 0.75 wt.% are characterized by the highest initial mass loss temperature T_0 = 624°K. The obtained research results indicate a correlation between the filler content, the structure, and the properties of the designed composites. The optimal content (q = 0.75 wt.%) of the discrete fiber mixture ensures the structural orderliness of the composite material and limits the mobility and deformation of the kinetic elements of the CM. This, in turn, is due to the thermal energy uptake by the fibrous component and its redistribution in the volume of the composite, which ensures a shift of the initial temperature of mass loss by ΔT_0 = 36.1°K (compared with the epoxy matrix) to the high temperature region. The obtained relative mass losses are an additional confirmation of the above provisions. It was established that when heated in the temperature range of ΔT = 624.0...754.5°K, composites filled with a MDOFCP with the content of q = 0.75 wt.% were characterised by the lowest relative mass loss ε_m = 65.7%.

Using the TGA curve, the composites' mass loss (Δm) at a given temperature was determined, and lines were graphically constructed along the tangent of the slope angles φ of the logarithmic dependence. Based on these data, the activation energy of thermal destruction of composite materials filled with a MDOFCP was calculated. It has been proven that the maximum excess of thermal energy required for destructing chemical bonds under the temperature influence is characterised for composites filled with the discrete organic fibre mixture with the content of q = 0.75 wt.%. This conclusion is made since their activation energy is the largest among the studied composites (E_a = 167.2 kJ/mol). The obtained activation energy results are consistent with the TGA results and confirm the assumption about the strength of the physico-chemical bonds and, therefore, their thermal stability.

By differential thermal analysis (DTA), it was defined that the key criteria for the long-term operation of composite materials under temperature influence were the initial temperature of the exoeffect and the maximum peak of the exoeffect. It was determined that there were no complex processes in the designed composite materials at the initial temperature of the exoeffect and the mobility and deformation of macromolecules and polymer segments at the maximum of the peak of the exoeffect. A comparison of the research results allow stating that composites filled with the discrete organic fibres mixture with the content of q = 0.75 wt.% are characterised by improved pointers of thermal stability due to the stability of chemical bonds (E = 167.2 kJ/mol) to the influence of temperature. Based on complex studies using differential thermal analysis, thermogravimetric analysis, and the results of mathematical calculations by the Broido method, it was proved that the operating temperature of the designed composites should not exceed T = 545.5°K.

ACKNOWLEDGMENTS

The authors would like to thank the KEGA grant agency for supporting research work and co-financing the project KEGA: 008TUKE-4/2025. Funded by the EU Next Generation EU through the Recovery and Resilience Plan for Slovakia under the project No. 09I05-03-V02-00042.

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