

Study of structural parameters of epoxy composites using deep neural networks*

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Abstract

The mechanisms of structural transformations in the epoxy matrix in terms of the mobility of the paramagnetic probe and the change in the areas of exothermic solidification peaks upon the introduction of aluminium oxide (Al_2O_3), zinc oxide (ZnO) and polytetrafluoroethylene (PTFE) were investigated. It has been found that Al_2O_3 and ZnO contribute to a significant decrease in the relative mobility of the probe from $t_0/t_f=0.95$ to $t_0/t_f=0.2$ in the material, respectively. It is proved that these processes are associated with the formation of physical nodes. In turn, PTFE provides an increase in wear resistance due to the formation of transfer films on friction surfaces. The introduction of ZnO and Al_2O_3 into the epoxy composite provides the most significant reduction in the peak area to $S_n/S_0=0.1$ at a concentration of 90 wt% and $S_n/S_0=0.2$ at 80 wt%, respectively, and PTFE - $S_n/S_0=0.45$ at 100 wt%. The use of neural networks and the Akim method for mathematical processing confirmed a high correlation between the predicted and experimental results ($R^2 > 0.98$). The histograms of residual values indicate the minimum deviations of the predicted data from the experimental values. The adequacy of the selected modelling methods for processing the experimental results has been proved. An improvement in wear resistance was found due to an increase in strength when filling with Al_2O_3 and ZnO (40-60 wt% and 30-50 wt% per 100 wt% of ED-20 binder, respectively). The use of PTFE (50-70 wt%) improves the antifriction characteristics of epoxy composites due to the formation of transfer films on friction surfaces. The expediency of an integrated approach, including experimental methods, approximation algorithms and neural network analysis for optimising the composition of epoxy composites has been proved.

Keywords

interpolation, transfer films, epoxy composite, relative mobility, Akim method, polymer matrix, neural networks.

1. Introduction

Composite coatings play an important role in ensuring the reliability and durability of structures, including by improving their physical and mechanical [1] and tribotechnical [2] characteristics, in various industries. Polymer composite materials (CM) are widely used due to their high wear resistance, chemical resistance, and a set of properties under specific operating conditions as coatings [3]. Such materials are widely used in mechanical engineering, aviation

*AdvAIT-2025: 2nd International Workshop on Advanced Applied Information Technologies: AI & DSS, December 05, 2025, Khmelnytskyi, Ukraine, Zilina, Slovakia

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and automotive industries, etc. The use of polymer composites helps reduce maintenance costs and extends the service life of equipment. The study of the tribotechnical characteristics of polymeric materials is an important area for improving the performance of equipment under various operating conditions [4-5].

Modern research in the field of CM development is aimed at identifying the regularities of the influence of the polymer matrix structure on its properties that can withstand extreme operating conditions [6-7], elevated temperatures [8-9], high mechanical loads [10-11], and aggressive environments [12]. The analysis of structural processes occurring during the formation of composites is important in the study of properties [13]. The study of these phenomena makes it possible to determine the mechanisms for improving the performance of the material.

The research of the relative change in the areas of exothermic peaks as structural parameters of a CM is an important aspect of assessing the thermal stability of polymer composites. This structural parameter correlates with the degree of crosslinking of the polymer matrix. The study of thermal effects associated with phase transformations and chemical transformations in the material allows us to identify regularities between the composition of CM and their characteristics.

The research of the relative mobility of the paramagnetic probe is an important area of analysis of the structural properties of the polymer CM. This parameter reflects changes in the process of molecular mobility in the material. The use of the electron paramagnetic resonance method allows obtaining data on the mobility of macromolecular segments and predicting their response to mechanical loads. A decrease in the mobility of the paramagnetic probe in the composite with the introduction of fillers indicates an increase in the degree of crosslinking and, as a result, an increase in the strength of the material. Changing the deformation characteristics of antifriction materials reduces the coefficient of friction and wear rate. The study of these parameters makes it possible to optimise the composition of composites taking into account their operational requirements and expands the possibilities for developing materials with increased wear resistance [14-16].

The Akeem method and neural network algorithms are widely used to analyse complex physicochemical and tribotechnical processes [17-21]. The Akeem method is used to process experimental data and approximate nonlinear dependencies, which reduces the influence of noise and allows obtaining accurate functional relationships between the parameters under study. This method is particularly effective in cases where traditional interpolation or regression approaches do not provide the required accuracy [22-23]. Neural network algorithms are actively used to predict the physical and chemical properties of materials based on major experimental data sets [24-25]. They allow detecting hidden patterns that are difficult for classical analysis methods [26-28]. The use of machine learning in the analysis of tribotechnical studies allows not only to identify the relationships between structural parameters and performance characteristics, but also to optimise the material composition for scientific prediction of CM properties. Recent advances in the application of neural networks and regression models for predictive analysis in materials science and biosensor technology have demonstrated high accuracy and robustness, especially when combined with experimental data processing methods and differential equations on lattices.

Unfortunately, modern scientists have not paid enough attention to these areas of research. The study of the structural characteristics of materials using neural networks will reveal

patterns that affect the characteristics of materials, as well as develop scientifically sound approaches to optimising the composition of polymer composites in order to increase their operational reliability.

The aim of this work is a comprehensive analysis of the influence of structural characteristics, based on changes in the areas of exothermic solidification peaks and relative mobility of the paramagnetic probe, on the physical and mechanical characteristics using electronic paramagnetic resonance and differential thermal analysis of composites with the analysis of research results by neural networks.

2. Materials and investigation procedure

The binder for creating composites was chosen based on the operating conditions of the components of mechanisms and machines. First of all, alternating loads were taken into account, which determines the mechanism of destruction of the surface layer of the material. Epoxy composites as a coating have sufficiently high strength of adhesive bonds to the working surface. An important characteristic of these materials is low residual stresses during moulding in the product. In connection with the above, we chose the epoxy-diane resin ED-20 (GOST 10587-76) and the polyethylene polyamine hardener (TU 6-05-241-202-78). The amine hardener (PEPA) allows the material to be formed at room temperatures on long-dimensional surfaces of complex profiles. The following fillers were used for studies: polytetrafluoroethylene - PTFE (GOST 10007-78), aluminium oxide Al₂O₃ (TU 6-09-426-75), zinc oxide ZnO (GOST 10262-62). The composites were prepared by hydrodynamic mixing of the components to obtain a homogeneous mixture. Depending on the tasks set in the experiment, some of the samples were vacuumed before curing and used as control samples.

Structural parameters were studied by differential thermal analysis (DTA). This research method was used to determine the interaction of ingredients in the CM. The activation energy during the formation of the CM was estimated from the DTA curves:

$$\ln \Delta t = C' - \epsilon / RT \quad (1)$$

$$\ln -2 \ln = A_0 - \epsilon / RT \quad (2)$$

$$\ln V_0 = B - \epsilon / RT \quad (3)$$

where Δt is the temperature change corresponding to the depth of the DTA peak at a given temperature; ϵ is the activation energy; R is the universal gas constant; T is the temperature; V_0 is the rate of decrease in the mass of a substance determined by the curves TG, C' , A_0 , B are constants.

The heating rate was 5 K/min in air.

Structural parameters in the material were determined using the method of electron paramagnetic resonance (EPR method) on a radiospectrometer of the RE-1306 brand. The use of EPR to study the kinetics of changes in the relative number of radicals during material formation is the most reliable method for studying the structural parameters of CM.

The resonance condition in the case of EPR is presented in the form:

$$pv = g \mu_B H_0 / 2\pi \quad (4)$$

where $\mu_B = 9,2741024$ Am is the Bohr magneton; g is a dimensionless factor or spectroscopic splitting factor (g-factor). The mobility of macromolecules in the binder at temperatures above

and below the glass transition temperature of the matrix (T_g) was determined by the mobility of the introduced paramagnetic probe. The value between the outer maxima on the resonance curve was also taken into account. The relative number of paramagnetic centres (free radicals) in the CM was estimated by the amplitude of the resonance curve.

The Akeem method was used to process the experimental data. This approach will ensure accurate detection of nonlinear dependencies with minimal error in the analysis of research results. Cubic splines were constructed to describe the change in parameters as a function of the concentration of fillers in the material. The basic equation of the spline:

$$S_i = a_i + b_i(x - x_i) + c_i(x - x_i)^2 + d_i(x - x_i)^3 \quad (5)$$

where $S_i(x)$ is the value of the function on the interval $[x_i, x_{i+1}]$, and a_i, b_i, c_i, d_i are the coefficients determined from the smoothness conditions.

The Akeem method provides a more complete interpolation of the experimental results.

The slope angles between the closest points were calculated using the following formulas:

$$m_i = \frac{y_i - y_{i-1}}{x_i - x_{i-1}} \quad m_{i+1} = \frac{y_{i+1} - y_i}{x_{i+1} - x_i} \quad (6)$$

At each point, the derivative was defined as a weighted average:

$$S'(x_i) = \frac{|x_{i+1} - x_i| \cdot m_{i+1} + |x_i - x_{i-1}| \cdot m_i}{|x_{i+1} - x_i| + |x_i - x_{i-1}|} \quad (7)$$

Next, interpolation curves were constructed. Deep artificial networks were used to determine the tribotechnical characteristics of the CM. The experimental data were normalised to the range (0.1) to improve the neural network training process.

The structure of the neural network:

Here is an example of a bulleted list:

- an input layer with neurons according to the number of input parameters;
- two hidden layers with 32 and 16 neurons respectively;
- an output layer with 2 neurons.

The training is based on the back-propagation of error. The method of the MSE (mean squared error) loss function was used. The Adam algorithm was used for optimisation. The initial learning rate was 0.001. The data was divided into training (80%) and test (20%) samples. The accuracy of the selected model was tested on the test sample using the coefficient of determination.

3. Results and discussion

Determination of structural processes in polymer CM is an important area of research into the mechanisms of interaction between material components. The study of structural changes makes it possible to predict the properties of CM under real operating conditions. Improving the physical and mechanical characteristics of epoxy composites contributes to increased wear resistance. This is achieved both by improving the structural parameters and by introducing

reinforcing fillers such as aluminium and zinc oxides. The use of additives capable of forming transfer films is also promising. In this case, the positive effect of mechanical characteristics in friction contact is realised. All deformation processes take place in the surface layer, i.e. in the material of the transfer films. As a rule, two such approaches are used in the development of antifriction characteristics. To determine the mechanism for improving the characteristics of epoxy CMs, exothermic effects were studied (DTA method). The exothermic curing peaks make it possible to estimate the energy released during the formation of intermolecular bonds in the polymer, which is a criterion for determining its degree of crosslinking. Determining the optimal level of structural stability and adaptability of the material helps to ensure its resistance to frictional loads, which increases the efficiency of friction units.

The dependence of the relative change in the area of the exothermic peak on the mass fraction of aluminium oxide (Al_2O_3) was investigated (Fig. 1.a). A tendency to decrease the peak value with increasing filler concentration was observed, indicating a gradual decrease in the thermosetting activity of the polymer matrix. When filling up to 20 wt% per 100 wt% of the binder, the change in the area of the exothermic S_n/S_0 peak decreases from $S_n/S_0=0.95$ to $S_n/S_0=0.65$. It was found that this is due to the primary formation of bonds between Al_2O_3 particles and the polymer, which stabilises the macromolecular structure. In the range of 20-50 wt.% per 100 wt% of binder (hereinafter the concentration of fillers was set in wt% per 100 wt% of binder), the peak area decreases to $S_n/S_0=0.4$, which characterises an increase in the cohesive interaction between the surface of the oxide fillers. An increase in the mass fraction of Al_2O_3 to 80 wt% reduces the peak area to $S_n/S_0=0.2$, which indicates almost complete filling of the intermolecular space with filler particles, which reduces the thermal effect of solidification.

When polytetrafluoroethylene (PTFE) was introduced into the CM, a smooth decrease in values in the range from $S_n/S_0=0.95$ to $S_n/S_0=0.45$ was observed with an increase in the filler concentration from 30 wt% to 100 wt%. In this case, PTFE is a modifier that changes the hardening mechanism. In the range of 30-60 wt.%, a decrease in the exothermic peak area from $S_n/S_0=0.75$ to $S_n/S_0=0.6$ was observed, which indicates changes in the thermal characteristics of the CM. At concentrations above 80 wt.%, the exothermic effects slow down, which may indicate a change in the mechanism of thermal interaction in the polymer due to the influence of the PTFE surface.

When zinc oxide was introduced into the binder (Fig. 1.c), a sharp decrease in the exothermic peak area from $S_n/S_0=0.9$ to $S_n/S_0=0.55$ was observed at a ZnO concentration of up to 10 wt.%. It was found that ZnO significantly affects the curing kinetics of the polymer matrix. In the range of 20-50 wt.%, the value of $S_n/S_0=0.3$, which indicates the achievement of stabilisation of exothermic effects. In the range of 60-80 wt% of ZnO , $S_n/S_0=0.2$, which may be caused by the interaction at the interface. A change in the thermophysical properties of the composite was found. At concentrations above 90 wt%, the value of $S_n/S_0=0.1$, which indicates the maximum stiffness of the structure material and complete stabilisation of the solidification processes. A material with high mechanical properties is formed.

It has been established that the introduction of Al_2O_3 and ZnO significantly changes the thermal effects of polymer composites when they are moulded into products. The use of PTFE provides a more gradual decrease in the relative area of exothermic peaks, which indicates a gradual change in the curing mechanism caused by the thermal effect of the filler. The obtained results of CM research make it possible to determine the optimal concentrations of fillers in the composite to achieve a balance between their mechanical and structural characteristics.

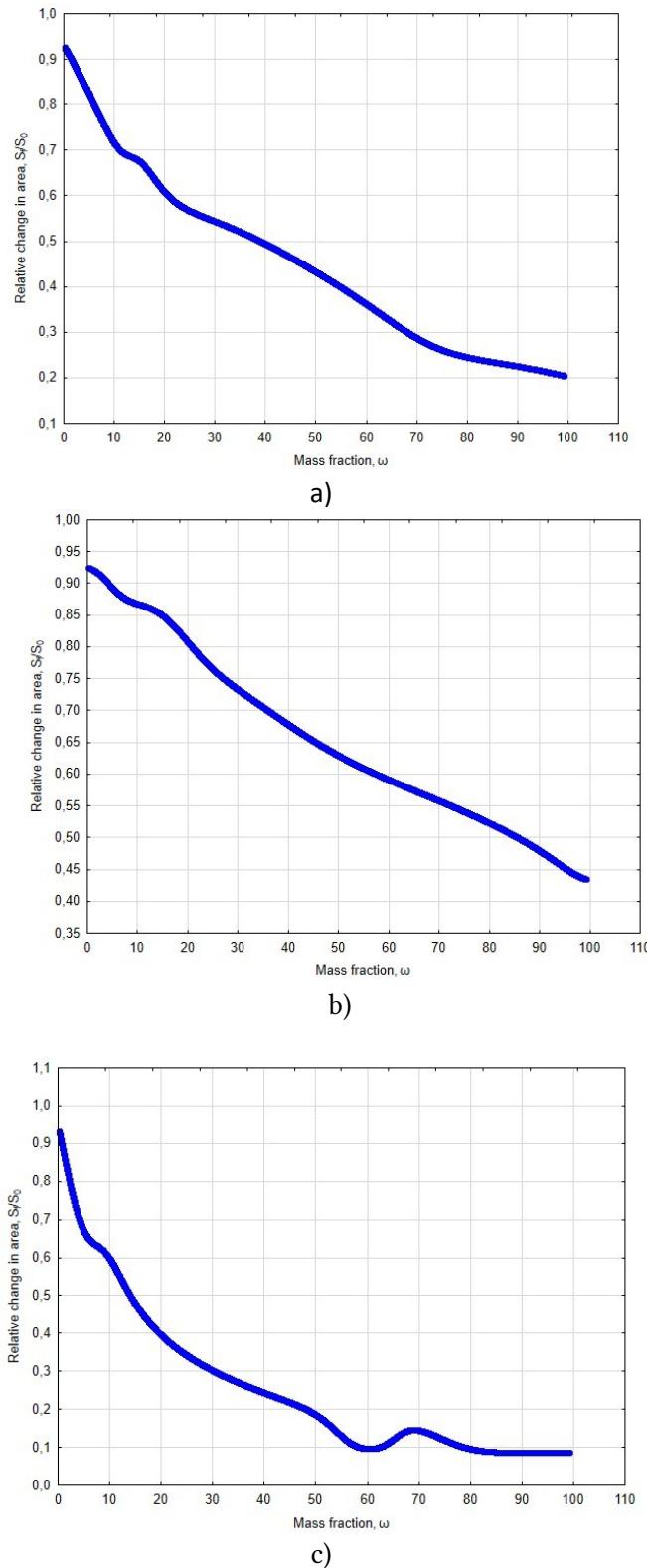


Figure 1: Results of studies of the relative change in the areas of exothermic hardening peaks with the concentration of fillers: a) Al₂O₃, b) PTFE c) ZnO.

The relative mobility of the paramagnetic probe (Fig. 2) is an indicator of the interaction at the interface in the system 'binder macromolecules - solid filler surface'. The mobility of the paramagnetic probe above the glass transition temperature (T_g) when Al_2O_3 is introduced was studied (Fig. 2.a). In this case, there are no physical nodes in the formation of the structural grid. A decrease in the value from $t_0^t/t_f^t=1.0$ to $t_0^t/t_f^t=0.91$ was observed with an increase in the filler concentration from 10 wt% to 40 wt%. At 20-30 wt% of Al_2O_3 in the CM, the relative mobility decreases to $t_0^t/t_f^t=0.95$, which can be explained by the increase in the interaction between filler and polymer particles. With a further increase in the mass fraction in the CM to 40 wt%, a decrease in mobility to $t_0^t/t_f^t=0.91$ was observed, which may indicate an increase in the physical interaction of the polymer phase and filler even at temperatures above T_c . This is confirmed by a decrease in molecular mobility in the CM, which is determined by the mobility of the paramagnetic probe.

The initial values of mobility (Fig. 2.b) are at the level of $t_0^t/t_f^t=1.0$, (5-10 wt%), a decrease in the mobility of the paramagnetic probe to $t_0^t/t_f^t=0.985$ was observed. A temporary decrease in intermolecular interaction at the interface between the polymer matrix and the surface of ZnO particles was observed. A further increase in the filler concentration leads to a gradual decrease in mobility to $t_0^t/t_f^t=0.93$ at 40 wt%. This indicates the formation of a material with a rigid structure in the polymer matrix, especially at the interface.

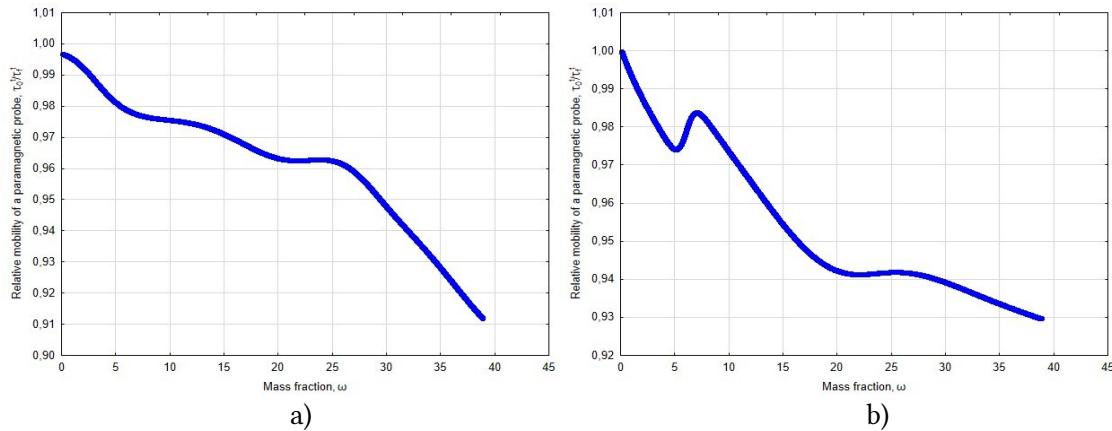


Figure 2: Results of studies of the relative mobility of the paramagnetic probe above the glass transition temperature relative to the filling: a) Al_2O_3 b) ZnO .

The macromolecular mobility of Al_2O_3 and ZnO is limited due to the increase in the number of bonds between the filler and the polymer due to the formation of additional bonds between the binder macromolecules and active centres on the filler surface (OH groups, exchange electrons, dislocations, etc.).

The mobility of a paramagnetic probe at a temperature above the glass transition temperature (T_g) when all physical nodes are destroyed is investigated. A decrease in mobility is observed (Fig. 3.a). With the introduction (up to 5 wt%), the mobility drops sharply from $t_0/t_f=1.0$ to about $t_0/t_f=0.55$, indicating the formation of stable bonds between the filler and the polymer, which sharply limits the confinement set of macromolecules. In the range of 5-30 wt% of aluminium oxide, the mobility value stabilises at 0.5-0.55, indicating that a balance has been achieved between the rigidity of the polymer matrix and the mobility of individual molecular segments. At a concentration of more than 30 wt%, a slight increase in mobility to $t_0/t_f=0.57$ was

observed, which may be due to an increase in the flexibility of individual macromolecular segments due to the effect of incomplete wetting of the filler.

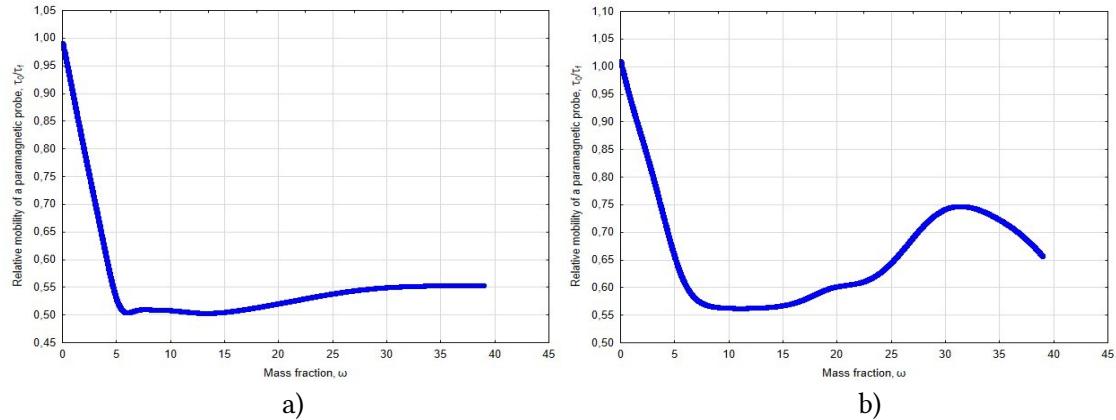


Figure 3: Results of studies of the relative mobility of the paramagnetic probe below the glass transition temperature relative to the filling: a) Al₂O₃ b) ZnO.

When ZnO (5 wt.%) was introduced into the material (Fig. 3.b), a drop in relative mobility from 1.0 to 0.5 was observed, indicating the formation of a dense polymer structure where macromolecular movement is sharply limited. At 5-25 wt%, the t_0/t_f value remains stable. In the range of 30-40 wt%, a characteristic increase in mobility to $t_0/t_f=0.7$ is observed, which is probably due to the formation of secondary interfacial interactions that can contribute to increased molecular flexibility.

Thus, the analysis of the obtained dependences shows that the behaviour of the relative mobility of the paramagnetic probe is largely determined by both the type of filler and temperature conditions. At temperatures above T_g , the material retains partial mobility even at high filler concentrations, while at temperatures below the glass transition, the CM structure material becomes much stiffer. A sharp drop in molecular mobility was observed at low filler concentrations. ZnO shows more complex processes in structural organisation. In this case, local peak values of mobility were observed, indicating phase transformations in the polymer system, while Al₂O₃ contributes to a gradual and uniform limitation of molecular mobility. This makes it possible to determine the optimal concentration of fillers depending on the specified performance characteristics of the material.

Further studies were carried out using the Akeem method to process the results of experimental studies. The exact dependencies of the main parameters were obtained. Additionally, the research results were analysed using neural network algorithms (Table 1), which made it possible to predict the tribotechnical characteristics of materials and assess their stability in operating conditions. Predicted and experimental dependencies are the result of testing the adequacy of the neural network. The efficiency of the used forecasting methodology was evaluated.

When studying the mobility of the paramagnetic probe, a linear correlation was observed, which confirms the correctness of the used prediction methodology. Minor deviations in some concentration ranges may be associated with local inhomogeneities in the structure of the polymer matrix material due to the peculiarities of the interaction of ingredients in the CM. We observed the prediction of changes in the areas of exothermic peaks of Sn/S₀ solidification,

where neural network algorithms provided high accuracy in calculating the structural parameters of the polymeric material.

Table 1
Characteristics of neural networks used in the CM study

Nº	Filler	Neural network	NN algorithm	Hidden activation	Output activation
Relative change in the area of exothermic solidification peaks					
1.	Al ₂ O ₃	MLP 1-9-2	BFGS 1657	Logistic	Identity
2.	PTFE	MLP 1-8-2	BFGS 10000	Logistic	Logistic
3.	ZnO	MLP 1-9-2	BFGS 9999	Logistic	Exponential
Relative mobility of the paramagnetic probe above the glass transition temperature					
4.	Al ₂ O ₃	MLP 1-9-2	BFGS 952	Tanh	Tanh
5.	ZnO	MLP 1-8-2	BFGS 831	Logistic	Identity
Relative mobility of the paramagnetic probe below the glass transition temperature					
6.	Al ₂ O ₃	MLP 1-8-2	BFGS 2015	Logistic	Identity
7.	ZnO	MLP 1-8-2	BFGS 1205	Tanh	Exponential

The obtained results confirm the correctness of the chosen prediction model and its ability to take into account complex intermolecular interactions in the material. This opens up prospects for the further use of neural networks in the process of developing and optimising the composition of polymer composites for operating conditions.

The results of the residual values (Table 2) establish the distribution of deviations between the experimental and predicted data obtained after processing by the Akeem method and neural networks. The residuals reflect the difference between the calculated and actual values, and the frequency of these deviations (Counts) indicates the number of corresponding values in the sample.

Table 2
Results of residual values in neural network modelling

u	als	0	0	0	0	0	0	0	01	02	03	04	05	06	07	08	09	01
		06	05	04	03	02	001											
Coun	ts	317	283	247	506	107	157	171	116	435	101	130	160	183	42	24	37	7
ZnO																		
Resid	u	-0.0	-0.00	-0.00	-0.00	-0.00	-0.00	-0.00	0.0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
		0	06	05	04	03	02	01		01	02	03		04	05	06	007	
Coun	ts	41	123	111	504	448	1022	1294	1817	1385	862	221		115	17	18	22	
Relative mobility of the paramagnetic probe below the glass transition temperature																		
Al ₂ O ₃																		
Resid	u	-0.00	-0.006	-0.005	-0.004	-0.003	-0.002	-0.001		0.0		0.001		0.002				
		7																
Coun	ts	7	21	13	24	51	134	3544		3837		282		87				
ZnO																		
Residua	ls	-0.0	-0.0	-0.0	-0.0	-0.0	-0.0	-0.0	0	0.0	0.	0.0	0.0	0.0	0.0	0.0	0.0	0
		07	06	05	04	03	02	01		01	0	03	04	.02				

The results of experimental studies at temperatures above T_g in CMs indicate a symmetric distribution of residual values around zero. The high accuracy of the modelling for the relative mobility of the paramagnetic probe above T_g is confirmed. The results at temperatures below T_g in CM characterise the residual values for the system, where a shift towards negative residuals was observed, which may indicate a systematic underestimation of the predicted values in certain concentration ranges. The uniform distribution of residuals indicates a high correlation between the predicted and experimental results. Most of the values were observed in the range of -0.001 to 0.001, which confirms the minimal error of the predicted model. The analysis of the residual values confirms the effectiveness of the data processing methods used and the accuracy of the neural network model.

4. Conclusions

Based on the results of the research, the following can be stated:

1. The improvement of the characteristics was achieved by increasing the physical and mechanical properties due to the influence of the structural organization in the composite. It was found that the change in structural parameters when introducing fillers (Al₂O₃, ZnO, PTFE) was achieved by determining the mobility of the macromolecule when a paramagnetic probe was introduced into the polymeric material and the number of paramagnetic centres in the composite. Targeted control of structural parameters was achieved by taking into account the exothermic effects during the formation of the composite.
2. The introduction of Al₂O₃ into the polymer matrix contributes to a significant decrease in the relative mobility of the paramagnetic probe, which varies from Sn/S₀=0.95 to Sn/S₀=0.2 at a concentration of 80 wt.%. The use of ZnO in CMs leads to a decrease in the relative mobility of the paramagnetic probe. In the range of 50-70 wt.%, a local increase

of this parameter at $T < T_c$ to $t_0/t_f = 0.3$ is observed, which indicates a change in the material structure. At a concentration of 90 wt%, the relative mobility decreases to $t_0/t_f = 0.1$. The introduction of PTFE reduces the relative mobility of the paramagnetic probe from $t_0/t_f = 0.95$ to $t_0/t_f = 0.4$ at 90 wt% to 100 wt%, which makes it optimal for obtaining polymer composites with preserved mechanical characteristics.

3. The structure of formation in the composite was studied by changing the areas of exothermic hardening peaks. It was found that the greatest decrease in these characteristics was observed in the case of Al_2O_3 and ZnO , where the Sn/S_0 peak decreases from $\text{Sn}/\text{S}_0 = 0.95$ to $\text{Sn}/\text{S}_0 = 0.2$ at 80 wt% and $\text{Sn}/\text{S}_0 = 0.1$ at 90 wt%, respectively. The effectiveness in stabilising the polymer matrix has been proved.
4. The obtained histograms of the residual values showed minimal deviations between the predicted and experimental results, where the average deviation for Al_2O_3 was ± 0.0002 , and for ZnO - ± 0.0003 , which indicates the high accuracy of the selected model. The overall correlation level between the experimental and predicted values exceeds 0.98, which confirms the effectiveness of using neural network methods to analyse the tribotechnical characteristics of polymeric materials. The results obtained allow us to recommend Al_2O_3 at a concentration of 40-60 wt% as a filler to ensure high material stiffness. ZnO is more suitable for the creation of adaptive materials with balanced flexibility and stiffness in the concentration range of 30-50 wt%. For polymeric compositions, the use of PTFE with a content of 50-70 wt% provides improved tribotechnical characteristics due to the formation of transfer films.
5. Thus, the results of the study confirm the possibility of targeted control of the properties of polymer composites by the choice of fillers and their concentration. The use of Al_2O_3 allows the creation of rigid materials with increased wear resistance, ZnO provides variability in mechanical characteristics, and PTFE improves tribotechnical characteristics due to the formation of transfer films from PTFE.

Declaration on Generative AI

The authors have not employed any Generative AI tools.

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