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THERMAL STABILITY OF THE STRUCTURE OF FULLERENE-CONTAINING COMPOSITES BASED ON EPOXY RESIN

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The study investigated the influence of fullerenes on the thermal decomposition of epoxy resin using thermoprogrammed desorption mass spectrometry (TPD-MS) and Raman spectroscopy. Special attention was paid to changes in the polymer matrix degradation mechanism upon addition of fullerenes at low concentrations (0.01–0.1 wt. %). It has been found that a fullerene content of 0.05 wt. % ensures the greatest thermal stability of the composite, with a pronounced suppression of heavy decomposition fragments and a predominance of light volatile products. This indicates an alteration of the kinetics and pathways of thermodegradative processes in the presence of the nanofiller.

The effect of fullerene content on the activation energy of thermal decomposition was analyzed, revealing that under certain conditions fullerenes can both retard and accelerate polymer degradation. This dual behavior is likely related to the introduction of structural defects in the polymer matrix, which affect the stability of chemical bonds. The physical mechanisms of fullerene action are discussed in terms of phonon relaxation, changes in electronic transport, and molecular mobility within the polymer/nanofiller interphase.

Raman spectra of the epoxy-fullerene composites show significant differences compared to those of the neat resin, confirming the impact of fullerenes on its structural properties. The low intensity of the characteristic fullerene bands in the composite spectra is attributed to the filler's low concentration, while observed band shifts point to local stresses in the polymer matrix arising during curing.

These results provide deeper insight into the stabilization mechanisms of epoxy-based polymer composites by fullerenes and help to define the optimal conditions for their use as functional nanoadditives. The observed enhancement of thermal resistance underscores the effectiveness of fullerenes in suppressing thermodegradative processes and highlights their promise for high-temperature polymer applications.

Keywords: epoxy resin, fullerenes, thermal stability, thermal decomposition, activation energy of degradation

INTRODUCTION

Epoxy resin is one of the most popular thermosetting polymers, regularly used in coatings, adhesives, sports equipment, leisure equipment, automobiles, and aerospace [1–4]. This wide range of applications for cured epoxies is due to their outstanding mechanical properties, high corrosion resistance, and excellent heat resistance. In addition to these advantages, they are lightweight, easy to mold in liquid form, and have low shrinkage during curing [5, 6]. However, highly cross-linked epoxy

matrices become brittle due to limited plastic deformation [7, 8], which leads to spontaneous failure of the epoxy resin due to localized stress concentration that initiates microcrack formation in the system [9, 10]. One of the ways to combat such negative effects is the use of nanoscale modifying additives to form nanocomposites with improved performance characteristics.

One of such nanoscale additives for modifying the properties of polymers (thermomechanical, optical, electrical, etc.) in order to create new functional polymeric materials is fullerene C₆₀

[11, 12]. Fullerene-containing polymers have characteristic features that can be considered as advantages or disadvantages depending on the plans for use. Having a more ordered and stable structure, polymers with covalently attached C_{60} are synthesized by more complex methods, and fullerene-polymer bonds significantly change the electronic structure of macromolecules and thus the properties of polymers [13].

It is known that composites with fullerenes have unique microstructure, mechanical, thermal, and other physical characteristics [14]. Thus, in [15], the effect was studied of the introduction of two different forms of functionalized fullerene C_{60} (FC_{60}) into the polymer matrix on the tribological and anticorrosive characteristics of the epoxy coating. It was found that the composite coatings showed a lower coefficient of friction, a smaller area of wear marks, and higher corrosion resistance compared to pure epoxy resin due to the balance of reinforcing, lubricating, and barrier properties of nanofillers and the cracks they create, and the optimal concentration of FC_{60} additive was 0.5 wt. %.

A comparative study of samples of epoxy nanocomposites reinforced with fullerene (C_{60}) [16, 17] showed that the addition of 0.5 wt. % C_{60} to the epoxy system increased the tensile strength, Young's modulus, and fracture toughness of the resulting epoxy nanocomposite by 13, 16.5, and 16 %, respectively, compared to the pure epoxy system.

Thus, fullerene is an important nanocarbon nanofiller used with epoxy resins. The combination of fullerene and epoxy resin leads to the improvement of many useful characteristics, which allows the use of epoxy/fullerene nanocomposites as anticorrosive, fire-resistant, organic photovoltaic materials, coatings, and adhesives [18].

However, most of the works on epoxy/fullerene nanocomposites mainly consider the effect of fullerene impurities on the mechanical properties of the resulting composites, while, for example, such a characteristic as thermal stability is not given much attention, although it is known [19] that fillers can significantly change the thermal properties of polymers. Therefore, the study on the dependence of the thermal stability of epoxy nanocomposites on the fullerene content is an urgent problem.

Studies [20] on the effects of the addition of fullerene C_{60} on the mechanical and dielectric properties of epoxy resin and using the

thermogravimetric analysis (TGA) method showed that the introduction of fullerene C_{60} lead to an extension of the last stage of thermal degradation above 700 °C, indicating its stabilizing effect. The thermal degradation of epoxy resin containing modified carbon nanotubes was also investigated [21, 22]. The study used thermogravimetric analysis and kinetic models to estimate the activation energy of the degradation process. Since the thermal degradation of epoxy resin filled with fullerenes under vacuum conditions using TPD-MS has hardly been studied, according to the literature, it can be attested that there is a scientific niche for further research in this area.

It should be noted that epoxies are widely used in the aerospace, automotive, and electronics industries due to their excellent mechanical properties and chemical resistance. However, under high temperatures in a vacuum, they may undergo thermal decomposition.

The study of thermal degradation in vacuum using TPD-MS allows for a deeper understanding of the degradation mechanisms and the identification of volatile products formed during heating. This, in turn, contributes to the development of more heat-resistant materials and improves existing systems for high-temperature applications. The study on the thermal degradation of epoxy resin modified with fullerenes in vacuum is extremely relevant, especially in the context of materials used in the aerospace and electronics industries, where materials are exposed to extreme temperature and vacuum conditions. Vacuum conditions avoid oxidative reactions, which makes it possible to study more accurately the mechanisms of thermal degradation and identify volatile decomposition products. This, in turn, contributes to the development of new heat-resistant materials with improved performance.

The aim of the work is to investigate the influence of fullerenes on the mechanism of thermal decomposition of epoxy resin and to determine the optimal content of nanofiller for improving the thermal stability of the composite using thermally programmed desorption mass spectrometry and Raman spectroscopy.

MATERIALS AND METHODS

In this work, composites based on an epoxy matrix filled with a mixture of fullerenes were prepared and studied. For this purpose, the epoxy

resin “Epoxy 520” (Czech production) was used, which was cured with polyethylene polyamine (PEPA) in a ratio of 7 : 1. A fullerene solution in toluene was added to the liquid resin and thoroughly mixed. To determine the optimal ratio of components in the binder-modifier system, different contents of the fullerene mixture (0.01, 0.05, and 0.1 wt. %) were used.

Due to their chemical reactivity, fullerenes must chemically bond with the atoms of the resin and hardener.

It has been found that the maximum filling of the resin with fullerenes is 0.1 %. At higher fillings, the intensity of chemical reactions between the components of the composite, which occur with the release of heat and intense gas formation, does not allow to obtain a composite.

The composition of volatile products of thermal degradation of composites was determined by TPD-MS using an MX 7304A mass spectrometer (Sumy, Ukraine) with temperature control in the range from 25 to 800 °C under vacuum conditions. The mass spectra of the destruction products of the samples were recorded and analyzed using an automated computer system. The spectra were recorded in the range of m/z 15–200 a.m.u. Desorption curves characterizing the decomposition process of resin and lignin-based composites were obtained from the mass spectrometric analysis data.

To record Raman signals, a MDR-23 spectrometer equipped with a cooled CCD detector iDus 420 Andor (UK) was used. Raman signals were excited using a diode-pumped solid-state laser line ($\lambda_{exc} = 671$ nm). The laser radiation power density on the sample surface did not exceed 10^3 W/cm², which made it impossible to thermally modify the samples. The spectral resolution for all wavelengths of the excitation radiation did not exceed 4 cm⁻¹ and was determined by the width of the peak of silicon phonons in a single crystal Si. The position of the Si phonon peak at 520.5 cm⁻¹ was used as a reference for determining the peak positions in the Raman spectra.

The activation energy E_d of the thermal degradation of composites was calculated using the Wigner-Polanyi equation [23]:

$$E_d = \frac{\ln\left(\frac{\theta_1}{\theta_2}\right) \cdot R \cdot (T_1 \cdot T_2)}{(T_2 - T_1)} \quad (1)$$

where T_1 and T_2 are the values of the peak temperatures of the maximum intensity of the desorption spectrum (according to TPD-MS), R is the universal gas constant, $R = 8.314$ J·mol⁻¹·K⁻¹, θ_1 and θ_2 are the areas under the curve corresponding to the relative amount of volatile products at T_1 and T_2 , respectively.

RESULTS AND DISCUSSION

One of the key criteria for the reliability of polymeric materials is their thermal structural stability, which ensures long-term operation under high-temperature conditions. The mass spectra of unfilled epoxy resin (0) and its composites with fullerenes at 0.01, 0.05, and 0.1 wt. % at temperatures in the range of 324–329 °C were obtained by TPD-MS (Fig. 1). The peaks in the range of 15–140 m/z correspond to fragments of the polymer matrix formed during the thermal degradation of the epoxy resin. The absence of heavy decomposition fragments larger than $m/z = 140$ indicates that the epoxy resin completely decomposes into molecular products without formation of stable macro radicals, including fullerenes that could stabilize the polymer matrix and prevent the formation of heavy thermal degradation fragments.

As can be seen from Fig. 1, the most intense peak is at $m/z = 18$, which may correspond to the H₂O fragment. Other significant peaks at $m/z = 28$ are thermal decomposition products such as CO, $m/z = 44$ is carbon dioxide CO₂ or organic fragment (C₂H₄O), $m/z = 94$ can correspond to (C₆H₄OH⁺) or (C₆H₅CH₃⁺). Peaks in the range of higher m/z can be fragments of the decomposition of aromatic structures of the polymer, for example, $m/z = 107$ C₆H₅CO and $m/z = 134$ C₈H₆O₂⁺. The peak with $m/z = 134$ indicates the formation of heavier decomposition products, which may be polymer residues.

Thus, the thermal decomposition of the epoxy matrix occurs completely before reaching 800 °C, and as can be seen from the mass spectra, without formation of heavy fragments, i.e., the destruction process proceeds through the formation of low molecular weight products. The destruction of composites with fullerenes is radical or depolymerization in nature, as the polymer breaks down into monomers and small aromatic structures. The result of adding fullerenes to epoxy resin is their influence on the mechanism of polymer structure destruction, which prevents the formation of larger decomposition fragments.

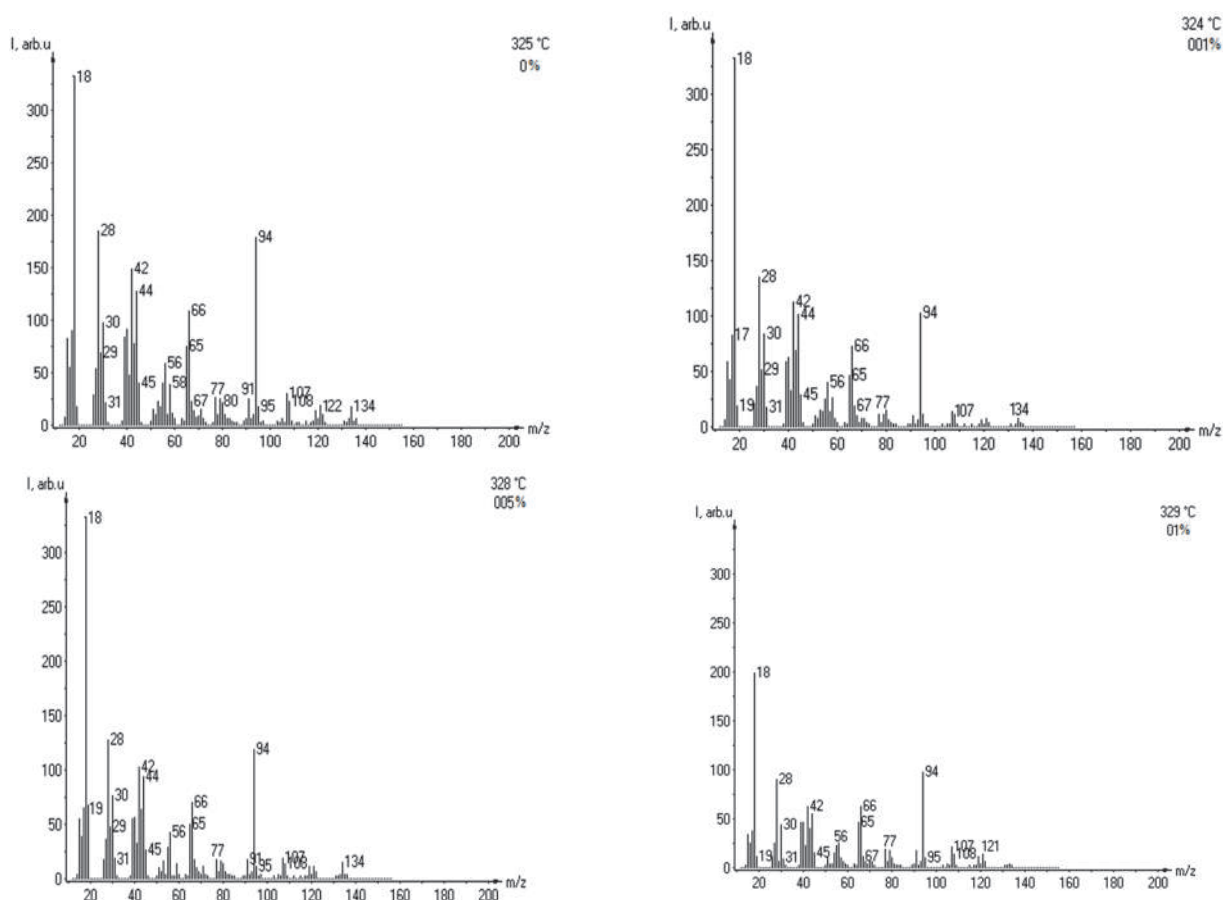


Fig. 1. Mass spectra of unfilled epoxy resin (0 %) and its composites with fullerenes at 0.01, 0.05, and 0.1 wt. % at temperatures in the range of 324–329 °C

Fig. 2 shows thermograms that demonstrate the thermal decomposition of epoxy resin composites with a hardener modified with fullerenes at different concentrations (0, 0.05, 0.01, and 0.1 %). All the graphs show the main decomposition peaks corresponding to the emission of various decomposition products (m/z 18 – H_2O , m/z 28 – CO , m/z 42 – $\text{C}_2\text{H}_2\text{O}$, m/z 77 – C_6H_5 , m/z 107 – $\text{C}_6\text{H}_5\text{CO}$ and m/z 134 – $\text{C}_8\text{H}_6\text{O}_2$). As can be seen from Fig. 2, for the unfilled epoxy, a high peak intensity is observed at ~ 300 °C for all decomposition products. When considering $m/z = 18$ (H_2O , water), it was found that the addition of fullerenes reduced the intensity of degradation, indicating a decrease in polymer dehydration. The peak of benzene fragments emission ($m/z = 77$ (C_6H_5)) is in the range of 350–400 °C. With the addition of fullerenes, its intensity decreases, indicating improved resistance to thermal decomposition. The largest changes are observed in the fragment

$m/z = 134$ (complex organic molecules), where the decomposition temperature rises from 350–400 to 450–500 °C, and at 0.1 % of fullerenes the peak almost completely disappears. This indicates a significant improvement in heat resistance and a reduction in the amount of volatile decomposition products. Thus, the filling of epoxy resin with fullerenes increases the thermal stability of the composite, especially in the high temperature range. That is, fullerene is most effective in the high temperature range, improving the polymer's resistance to decomposition. Reduced emission of volatile thermal decomposition products at temperatures > 400 °C means that the polymer structure becomes more heat-resistant. Probably, fullerenes effectively inhibit the decomposition of complex organic fragments, which confirms its role as a thermal stabilizer.

When considering the temperatures of the maximum desorption of thermal decomposition

products of unfilled epoxy resin and its composites with fullerenes at 0.01, 0.05, and 0.1 wt. % (Fig. 3), no correlation between the

content of fullerenes and the m/z of decomposition products is observed.

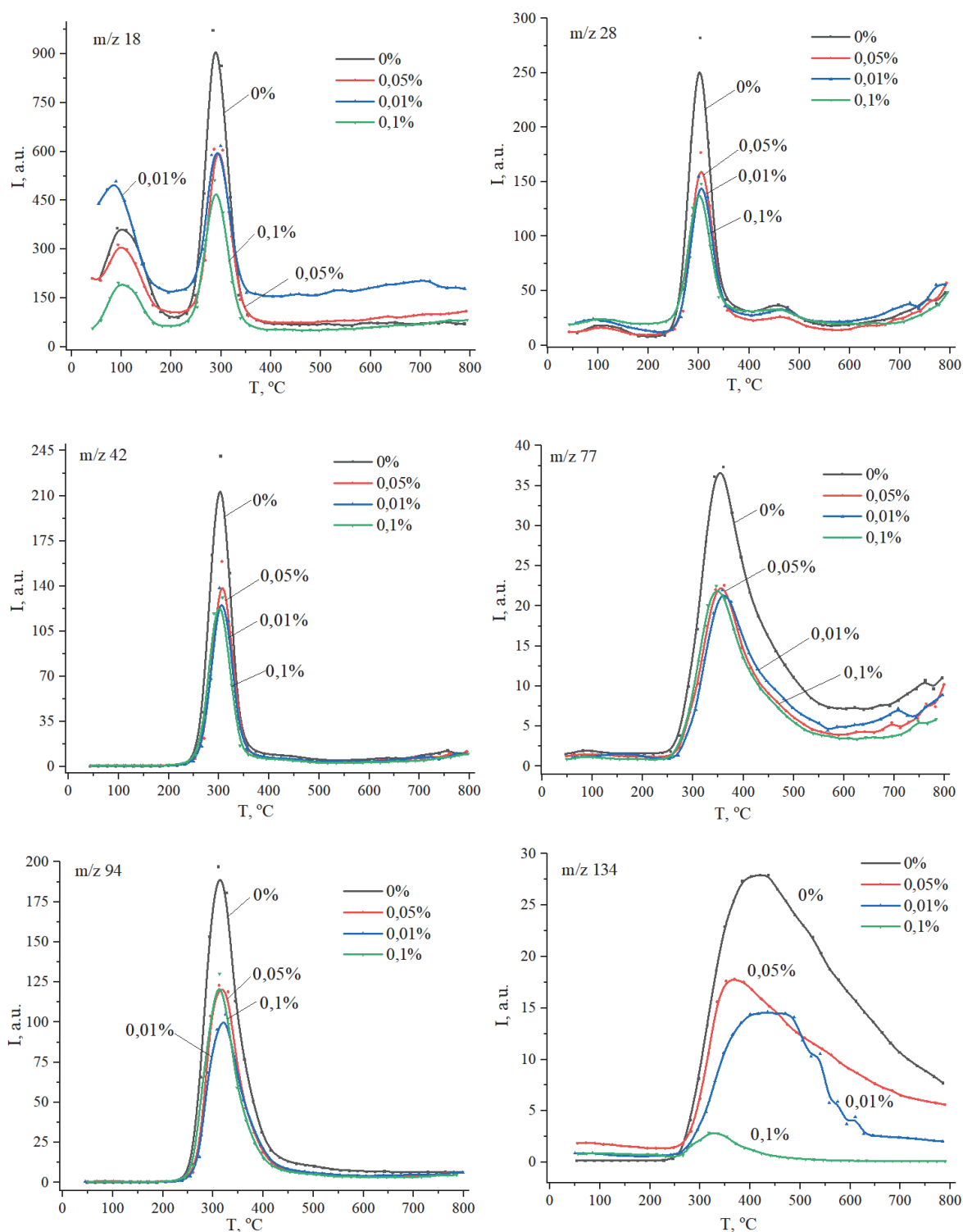


Fig. 2. Thermograms of decomposition products of unfilled epoxy resin (0 %) and its composites with fullerenes at 0.01, 0.05 and 0.1 wt. %

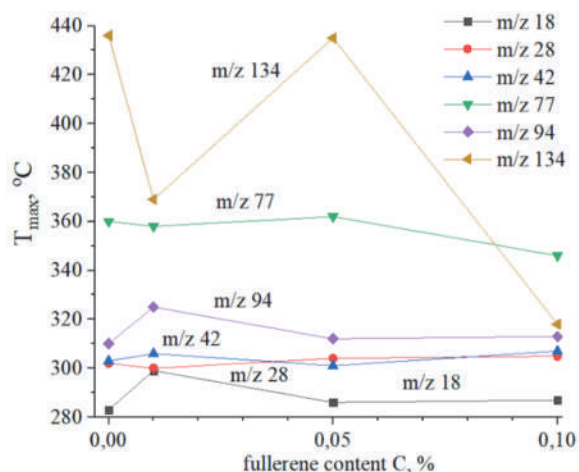


Fig. 3. Temperatures of the maximum desorption of thermal decomposition products of unfilled epoxy resin and its composites with fullerenes at 0.01, 0.05, and 0.1 wt. %

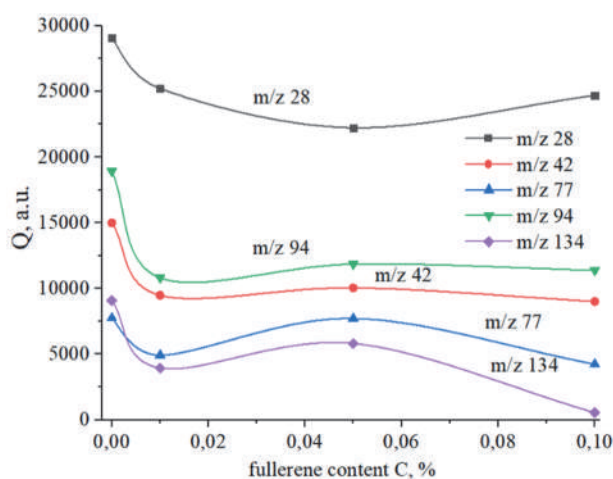


Fig. 4. The total amount of desorption of thermal decomposition products of unfilled epoxy resin and its composites with fullerenes at 0.01, 0.05, and 0.1 wt. %

Fig. 4 shows a graph showing the dependence of the total amount of desorbed decay products (Q) on the fullerene content (C , wt. %) in epoxy resin-curing agent composites. As can be seen from Fig. 4, at a low fullerene content (up to ~ 0.05 %), the intensity of many ions (e.g., m/z 42, 94) gradually increases, which may indicate the uniform incorporation of fullerenes into the polymer matrix without significant changes in the structure. At a content of ~ 0.05 – 0.1 wt. % of fullerene, insignificant changes are observed, which may be due to the critical concentration of fullerenes, after which the nature of the interaction between the polymer, hardener, and filler changes, for example, due to the possible formation of fullerene aggregates or their effect on the curing process. At a high fullerene content

(0.1 %), the Q value decreases or stabilizes. This may mean that fullerenes begin to adversely affect the curing process or cause phase separation of the composite. It is also possible to form a porous structure or gas emission, which makes it difficult to obtain high-quality material.

As can be seen from Fig. 5, at a content of 0.01 wt. % of fullerenes, the activation energy E_d decreases for all decomposition products, including carbon fragments.

At a content of 0.05 wt. % of fullerenes, the activation energy E_d increases, which indicates the formation of products with more difficult desorption, i.e., the polymer matrix becomes more thermally stable. Probably, if the decomposition occurs due to the rupture of C-O bonds in the matrix, then stabilization of the

polymer with fullerenes can increase the activation energy E_d for fragments that include oxygen-containing groups.

It is known that the change in the structure of filled composites is due to the presence of an interface and the interaction in the polymer-filler

interfacial region [24, 25]. Therefore, the interaction of fullerenes with polymer chains in the zone of local interaction between fullerenes and the polymer matrix is a key factor influencing the degradation of fullerenes.

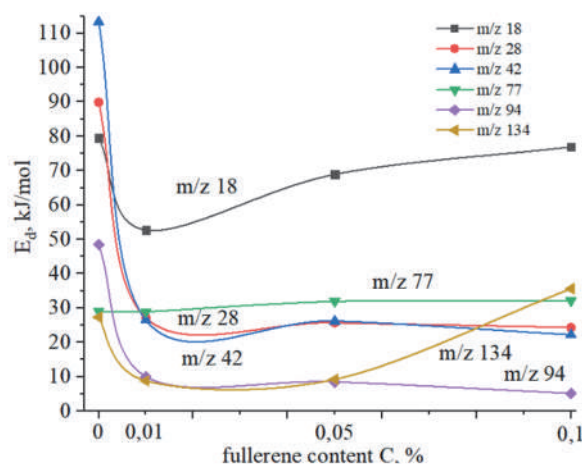


Fig. 5. Dependence of the activation energy of the degradation of epoxy resin decomposition products E_d on the fullerene content, calculated using the Wigner-Polanyi formula

We assume that fullerenes can be distributed within the polymer, enhancing its thermal stability, while weak intermolecular interactions, such as π - π stacking between the fullerene molecules and aromatic fragments of the polymer chains, contribute to the stabilization of the polymer matrix. The difference in heat resistance is likely due to the interaction with the active centers of the polymer, changing the degradation mechanism. With an increase in the content of fullerenes, fullerene aggregates are formed, resulting in defects that accelerate the local destruction of the structure. According to the analysis of Fig. 5, it can be assumed that if fullerenes bind to polymer functional groups, they can change the degradation mechanism by affecting the activation energy for certain degradation products.

Thus, fullerenes can both increase the thermal stability of the polymer and catalyze its degradation, depending on their concentration, distribution in the matrix, and the nature of their interactions. For different decomposition products, this is manifested in a change in the activation energy of degradation, which allows us to draw conclusions about changes in the mechanism of polymer degradation during heating.

Fig. 6 shows the Raman spectra of pure fullerene (curve 1) and the spectra of epoxy resin with different proportions of fullerene: curve 2 – 0 wt. %, curve 3 – 0.01 wt. %, curve 4 – 0.05 wt. %, curve 5 – 0.10 wt. %. The Raman spectrum of pure fullerene shows characteristic bands with frequencies of 1425, 1469, and 1572 cm^{-1} , corresponding to its vibrational modes of Hg, Ag, and Hg symmetry, respectively. The Raman spectra of the epoxy resin with fullerene differ significantly from the spectrum of the original epoxy resin sample without fullerene. However, the main changes occurred in the Raman spectra of the epoxy resin itself, which is due to its heating and the addition of fullerene to it. The analysis of the experimental spectra shows that the bands of fullerene include low-intensity peaks with frequencies of 1424, 1463, and 1471 cm^{-1} . Their low intensity is due, on the one hand, to the low concentration of fullerene in the epoxy resin, and, on the other hand, to the more efficient scattering (scattering cross section) of the epoxy resin. A slight shift of the fullerene band with a frequency of 1469 cm^{-1} to the high-frequency side may be due to the compression stresses on the part of the matrix that occur during the curing of the epoxy resin.

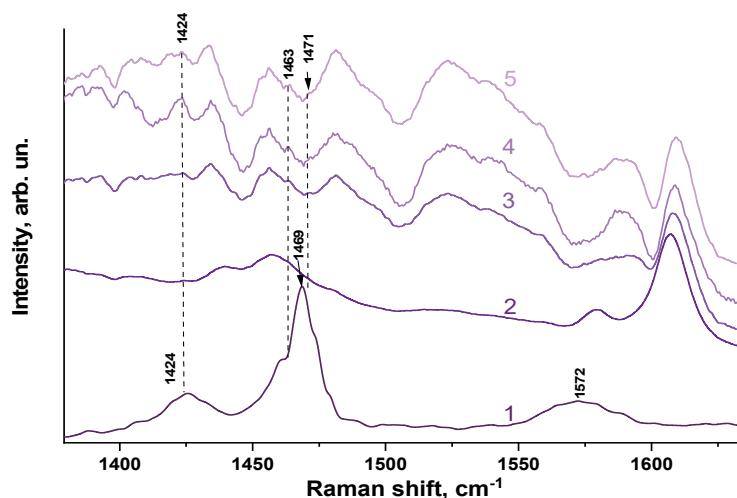


Fig. 6. Raman spectra of fullerene (I) and epoxy resin with different proportions of fullerene: 2 – 0 wt. %, 3 – 0.01 wt. %, 4 – 0.05 wt. %, 5 – 0.10 wt. %

It is known that fullerenes have high thermal conductivity due to their crystalline structure and delocalized π -bonds [26]. In our case, when the epoxy resin is heated, heat is absorbed, which excites vibration modes (phonons). Therefore, according to Figs. 3 and 4, it can be assumed that at optimal concentrations (0.05 wt. %), fullerenes act as effective phonon scatterers, improving thermal conductivity and evenly distributing heat in the composite. This prevents localized overheating and stabilizes the polymer. Thus, at the optimum fullerene content, heat is dissipated evenly, which prevents local overheating and increases the activation energy of destruction. With an excess of fullerenes, aggregates are formed that cause localized heat accumulation, which leads to local heating and initiates the thermal decomposition of the polymer.

When considering Figs. 3–5, it can be assumed that since fullerenes have a high electron affinity [27], they can accept electrons and act as electron traps. Thus, the thermal decomposition of the epoxy resin proceeds predominantly via homolytic cleavage of chemical bonds, leading to the formation of free radicals. At the optimum concentration (0.05 wt. %), fullerenes can act as effective electron acceptors, interacting with these radicals and thereby inhibiting further radical chain reactions, which stabilizes the polymer structure.

At another concentration (0.01, 0.1 wt. %), fullerenes can form conductive domains, which facilitates charge transfer and the radical

decomposition of the polymer. Thus, at low concentrations, fullerenes act as stabilizers, slowing down radical reactions, and at high concentrations, they promote charge transfer and degradation. The mobility of macromolecules in the polymer is limited at low concentrations of fullerenes due to intermolecular interactions (π - π stacking). This increases the activation energy of degradation. At high concentrations, fullerenes begin to act as defects, disrupting the polymer network, which reduces the activation energy. Thus, fullerenes change the dynamic properties of the polymer matrix, which affects the mechanism of its degradation: from stabilization (at low content) to acceleration of destruction (at high content).

CONCLUSIONS

It has been found that fullerenes change the thermal properties of the polymer matrix, which affects the mechanism of its degradation: from stabilization (at 0.05 wt. %) to acceleration of degradation (at 0.1 wt. %). Fullerenes most effectively improve the polymer's resistance to decomposition in the high temperature range. A decrease in the release of volatile thermal decomposition products at temperatures > 400 °C indicates that the polymer structure becomes more heat-resistant. Probably, fullerenes effectively inhibit the decomposition of complex organic fragments, which confirms their role as a thermal stabilizer. Thus, the optimal concentration of fullerenes can significantly

improve the heat resistance and durability of epoxy composites.

At the optimum content of fullerenes, heat is dissipated evenly, which prevents localized overheating and increases the activation energy of degradation. With an excess of fullerenes, aggregates are formed that cause localized heat

accumulation, which leads to local heating and initiates the thermal decomposition of the polymer. Thus, fullerenes increase the thermal structural stability of epoxy resin, contributing to the stabilization of the polymer network when heated.

Термічна стійкість структури фулеренвмісних композитів на основі епоксидної смоли

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У роботі досліджено вплив фулеренів на процес термічного розкладання епоксидної смоли за допомогою термопрограмованої десорбційної мас-спектрометрії (ТПД-МС) та Раман-спектроскопії. Особливу увагу приділено зміні механізму деструкції полімерної матриці при введенні фулеренів у низьких концентраціях (0.01–0.1 мас. %). Встановлено, що за вмісту фулеренів на рівні 0.05 мас. % досягається найкраща термостабільність композиту. При цьому утворення важких фрагментів терморозпаду значно пригнічується, а переважають легкі леткі продукти. Це свідчить про зміну кінетики та напрямку термодеструктивних процесів у присутності нанонаповнювачів.

Проаналізовано вплив вмісту фулеренів на енергію активації процесів термічного розкладання та встановлено, що за певних умов фулерени можуть як уповільнювати, так і пришвидшувати деструкцію полімера. Такий ефект пов'язаний, ймовірно, з утворенням структурних дефектів у полімерній матриці, що впливають на стабільність хімічних зв'язків. Обговорюються фізичні механізми впливу фулеренів, зокрема фононна релаксація, зміни електронного транспорту та молекулярної рухливості у міжфазній області полімер/нанонаповнювач.

Раманівські спектри епоксидної смоли з фулереном демонструють суттєві відмінності порівняно зі спектрами чистої смоли, що свідчить про вплив фулерену на її структурні властивості. Незначна інтенсивність характеристичних смуг фулерену у спектрах композиту пов'язана з низьким вмістом нанодобавки. Зсув смуг фулерену в спектрі вказує на наявність локальних напружень у полімерній матриці, що виникають під час її затвердіння.

Отримані результати дозволяють глибше зрозуміти природу стабілізації полімерних композитів на основі епоксидних смол і визначити оптимальні умови введення фулеренів як функціональних нанодобавок. Підвищення термостійкості структури композитів свідчить про ефективне пригнічення термодеструктивних процесів і перспективність подальшого використання фулеренів у високотемпературних полімерних матеріалах.

Ключові слова: епоксидна смола, фулерени, термостабільність, терморозпад, енергія активації деструкції

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