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INVESTIGATION OF THE BIODEGRADATION OF POLYURETHANE-UREAS CONTAINING FRAGMENTS OF GRAFT COPOLYMER POLYVINYL ALCOHOL—POLYETHYLENE GLYCOL IN THE STRUCTURE UNDER MODEL CONDITIONS OF THE INFLAMMATORY PROCESS

The aim of this study was to investigate the biodegradation capacity of hydrophilic polyurethane-ureas (PUUs) containing fragments of 4,4'-diaminodiphenylmethane (DADPh) and a grafted copolymer polyvinyl alcohol—polyethylene glycol (PVA—PEG) in their structure under model conditions of the inflammatory process when in contact with blood. The biodegradation capability was evaluated by infrared (IR) spectroscopy, physical-mechanical testing, and differential scanning calorimetry (DSC) by monitoring changes in the structure, physical-mechanical, and thermophysical properties of the PUUs under the influence of the Fenton reagent over incubation periods of 1, 3, and 6 months. According to IR spectroscopy data, exposure to the Fenton reagent initiates an oxidative degradation process of the investigated PUUs, accompanied by the cleavage of specific chemical bonds, alterations in hydrogen bonding, and structural transformations of the polymer matrix. The physical-mechanical properties depend on the incubation duration in the Fenton reagent and change nonlinearly. After 6 months of incubation, compared to the control, an increase in strength and relative elongation at break was observed, which can be explained by the formation of new hydrogen bonds under the influence of the model medium. DSC results

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revealed that after incubation in the Fenton reagent, the PUU samples exhibited an increase in the glass transition temperature (T_g) and a jump in heat capacity (ΔC_p) at the glass transition, indicating structural changes during the oxidative degradation process. Thus, the investigated materials demonstrate the process of oxidative biodegradation under the influence of a model medium. Therefore, PUUs containing fragments of the grafted copolymer PVA—PEG in their structure represent a promising polymer matrix for temporary medical applications.

Keywords: *polyurethane-urea, grafted copolymer polyvinyl alcohol—polyethylene glycol, oxidative biodegradation, Fenton reagent.*

Introduction

Hydrophilic polyurethane-ureas (PUUs) containing fragments of the grafted copolymer polyvinyl alcohol—polyethylene glycol (PVA—PEG) and the chain extender 4,4′-diaminodiphenylmethane (DADPh) in their structure [1] are a promising polymer matrix for medical applications and require further biomedical evaluations, particularly concerning their biodegradation capacity.

The ability to biodegrade under conditions mimicking the physiological environment is one of the most important characteristics of polymeric materials intended for medical use. Factors such as the chemical composition of the polymer and the hydrophilicity of the polymer matrix influence the biodegradation processes [2, 3]. In particular, an increase in the hydrophilic component within the polymer matrix leads to an enhanced rate of biodegradation [4, 5]. It is also important to consider the impact of the soft and hard segments within the structure of the PUUs on the biodegradation processes. Variations in the component ratios can significantly affect the stability and degradation rate of PUUs *in vitro* [6, 7]. The length of the soft segments also influences biodegradation in alkaline solutions; segmented PUUs with longer soft segments, which result in increased hydrophobicity, exhibit a lower degradation rate. Moreover, a narrow molecular weight distribution may be another factor contributing to the slower degradation of PUUs [8]. The degree of biodegradation may also depend on the pH of the medium and the type of hard segment (i.e., the amide chain extender). Studies have shown that amide bonds are resistant to degradation in an alkaline medium, whereas at neutral pH there is a sharp decrease in the viscosity of the multiblock copolymer poly(urethane—amide) [9].

Biodegradation processes in the polymer matrix are accompanied by changes in its structure and, consequently, alterations in the properties of the studied material. Since the chemical composition of the polymer influences the course of these biodegradation processes, it is necessary to investigate the biodegradation capacity of PUUs synthesized with different molar ratios of components.

We conducted studies [10] on the biodegradation capacity of polyurethane-ureas that contain a grafted copolymer PVA—PEG as the hydrophilic component and DADPh as the chain extender, using biological medium 199, which mimics blood plasma and consists of a complex mixture of proteins, amino acids, hydrocarbons, lipids, salts, hormones, enzymes, antibodies, and dissolved gases.

However, considerable interest lies in investigating the biodegradation capacity of polymeric materials in a model medium (the Fenton reagent), which simulates the conditions of oxidative degradation of the polymer during the inflammatory process. The Fenton reagent generates hydroxyl radicals (OH·) and other reactive oxygen species (ROS) [11] that induce the oxidative degradation of biomolecules [12]. During the inflammatory process, a release of ROS also occurs, especially during the oxidative burst of neutrophils and macrophages. These radicals can attack biomaterials, leading to their structural breakdown [13].

Under the influence of the Fenton reagent and ROS in the body, polyurethanes undergo hydrolysis, oxidative degradation, and macromolecular breakdown, which alters their mechanical properties and biocompatibility.

Considering that oxidative processes similar to those occurring during inflammation in the body can lead to hydrolysis, degradation, and breakdown of polyurethane macromolecules, thereby affecting their mechanical stability and biocompatibility, the aim of this work is to investigate the time-dependent biodegradation capacity of polyurethane-ureas containing a grafted copolymer PVA—PEG in their structure under the influence of the Fenton reagent.

Experimental part

Materials. The objects of study were PUUs based on diisocyanate prepolymer (DPP), chain extender DADPh, and grafted copolymer PVA-PEG, synthe-

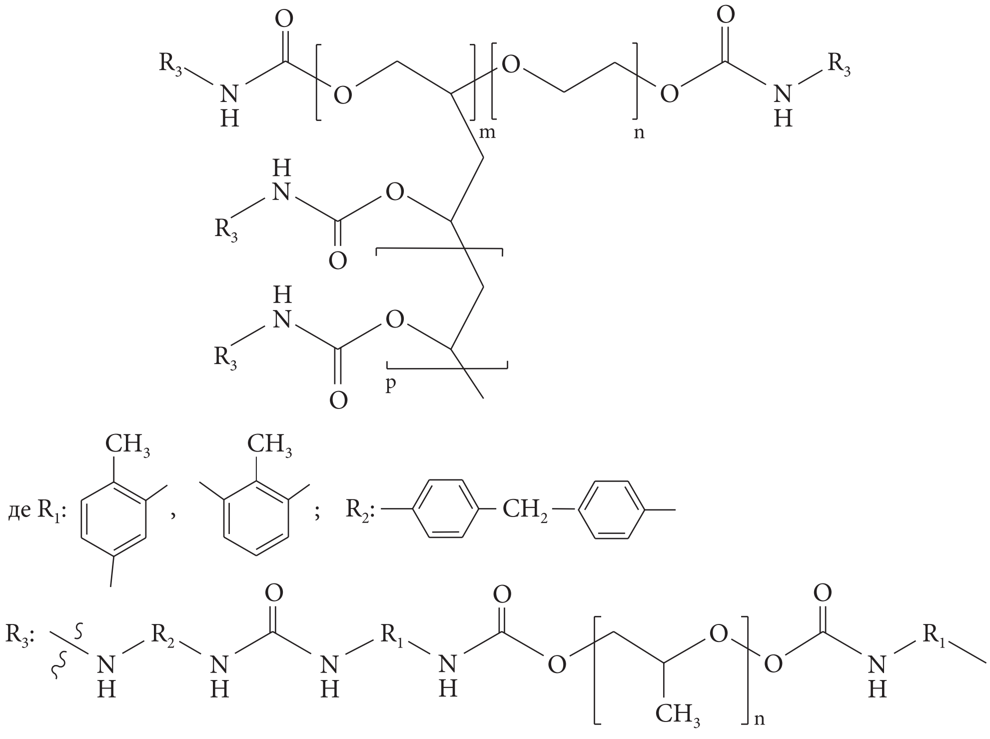


Fig. 1. Structural formula of branching nodes of the PUU

sized at molar ratios of DPP:DADPh:PVA-PEG as 1.0:0.7:0.3 (PUU1), 1.0:0.8:0.2 (PUU2), 1.0:0.9:0.1 (PUU3) (Fig. 1).

Fenton's reagent, a solution of highly diluted hydrogen peroxide (H_2O_2) and divalent iron salt ($FeSO_4 \times 7H_2O$) simulating the conditions of the inflammatory process when the material comes into contact with blood was used as a model medium to study the ability to biodegradability.

Incubation in the Fenton reagent. Samples were placed in sterile containers, filled with 25 ml of model medium, and kept in a thermostat at a temperature of (37 ± 1) °C for periods of 1, 3, and 6 months. The medium solutions were changed daily. After the specified periods in the Fenton reagent, the samples were removed, washed with distilled water, and dried to constant weight at room temperature.

Methods of Investigation. The structure was examined using an FTIR spectrometer with Fourier transformation "Tensor-37" (Bruker) by the method of disturbed total internal reflection (DTIR) in the range of 500—4000 cm^{-1}, employing a diamond crystal trapezoidal prism (number of reflections N = 1, angle of incidence $\varphi = 39°$).

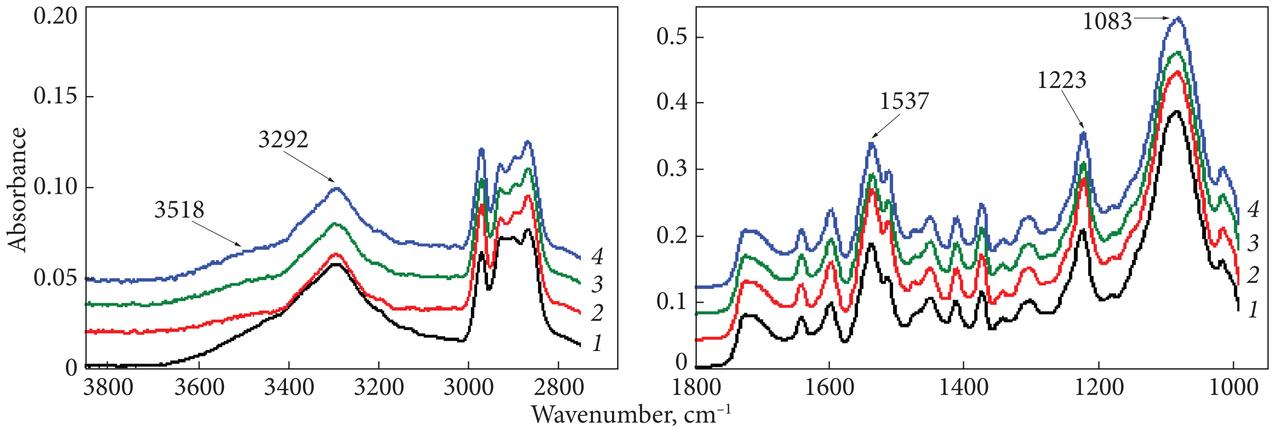
Physical-mechanical properties, such as tensile strength (σ, MPa) and relative elongation at break (ε, %), were measured using a P5 tensile testing machine in accordance with ISO 527-3.

Thermophysical properties (glass transition temperature (T_g) and change in heat capacity at the glass transition (ΔC_p) were studied using differential scanning calorimetry (DSC). The experiments were conducted over a temperature range from −90 °C to 200 °C (using the TA Instruments Q2000 device) at a heating rate of 20 °C/min under a nitrogen atmosphere. To eliminate the influence of the material's thermal and mechanical prehistory, two heating cycles were performed.

Results and Discussion

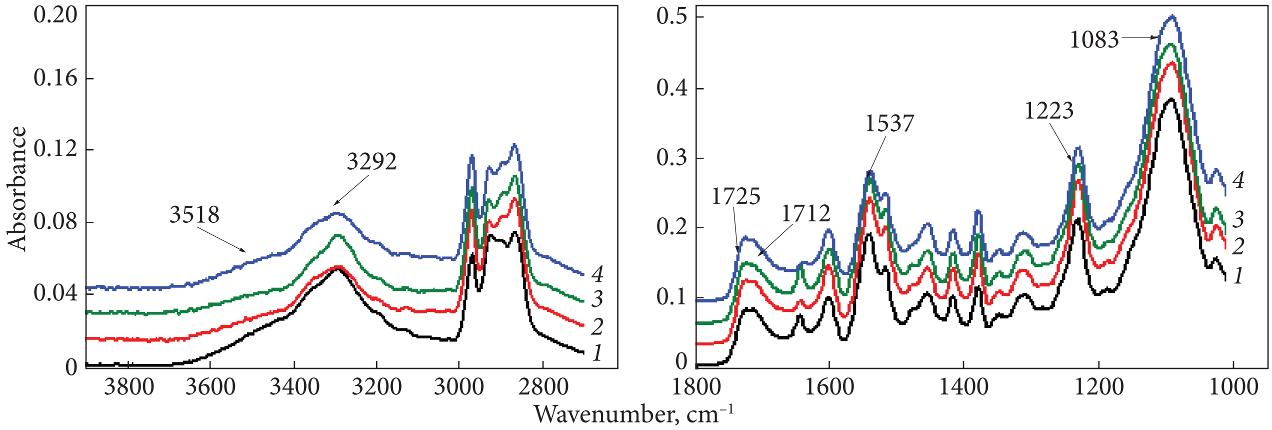
To investigate the biodegradation capability of polyurethane-ureas (PUUs) containing fragments of the grafted copolymer PVA—PEG, synthesized with different molar ratios of DPP: DADPh:PVA—PEG (1.0:0.7:0.3, 1.0:0.8:0.2, 1.0:0.9:0.1), the samples were incubated in the Fenton reagent for 1, 3, and 6 months. The influence of the model medium was assessed by comparing changes in the structure, physical-mechanical, and thermophysical properties of the samples before (control) and after incubation.

Infrared spectroscopic studies of the PUU samples before and after exposure to the model medium revealed no significant changes in the overall chemi-



a *b*

Fig. 2. Fragments of the IR spectra of PUU1 before (*1*) and after incubation in the Fenton reagent for 1 month (*2*), 3 months (*3*), and 6 months (*4*) in the ranges of 3800—2800 cm^{-1} (*a*) and 1800—1000 cm^{-1} (*b*)



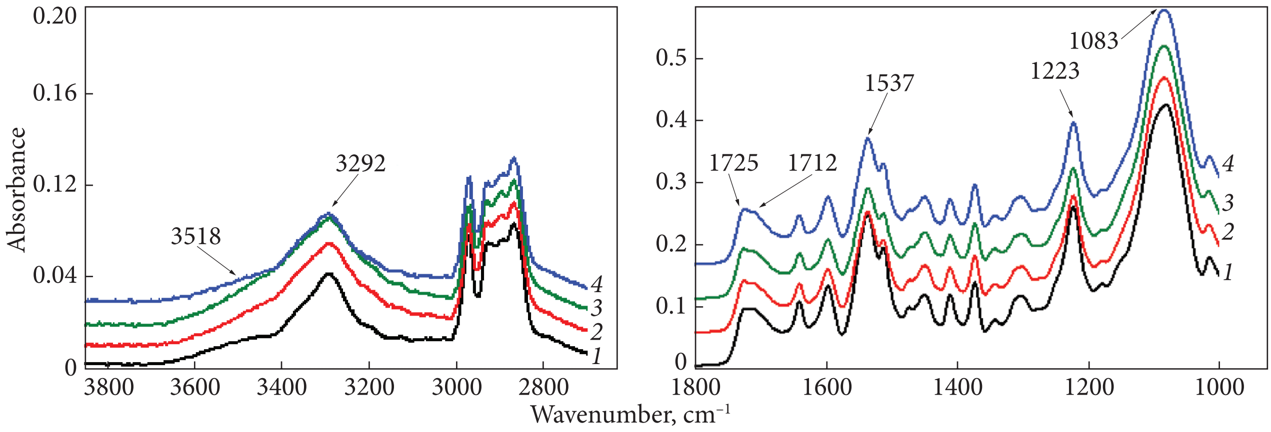
a *b*

Fig. 3. Fragments of the IR spectra of PUU2 before (*1*) and after incubation in the Fenton reagent for 1 month (*2*), 3 months (*3*), and 6 months (*4*) in the ranges of 3800—2800 cm^{-1} (*a*) and 1800—1000 cm^{-1} (*b*)

cal structure. However, variations were observed in the stretching vibration regions sensitive to hydrogen bonding. The absorption bands were assigned as described in [14—16].

For PUU1, in the frequency range of 3800—2800 cm^{-1} (Fig. 2*a*), the action of the Fenton reagent during 6 months of incubation resulted in a decrease in the intensity of the absorption bands corresponding to bound NH groups ($\nu_{\text{NH-bound}}$ with a maximum at 3292 cm^{-1}) and free NH groups ($\nu_{\text{NH-free}}$ with an approximate maximum at 3518 cm^{-1}). This decrease is associated with a reduction in the number of free and hydrogen-bonded NH groups on the surface layer of the samples (as the IR spectra were recorded from the surface of the materials). The reduction in the intensity of the NH-group bands may indicate a change in the configuration of hydrogen bonds or degradation of the polymer structure under the oxidative influence of the model medium. In the spectral interval of 1800—1000 cm^{-1} (Fig. 2*b*), after 6 months of incubation, an increase in the intensity of the δ_{NH} absorption band (1537 cm^{-1}) was observed. This may signify enhanced hydrogen bonding or the formation of new interactions between the NH groups of the polymer matrix and the OH groups of the components of the model medium. Additionally, an increase in the intensity of the $\nu_{\text{C—O}}$ absorption bands, with maxima at 1223 cm^{-1} and 1083 cm^{-1}, was noted. This likely reflects structural changes on the surface layerin the polymer, possibly related to oxidative processes or interactions with the medium components.

For PUU2, in the IR spectral range of 3800—2800 cm^{-1} (Fig. 3*a*), after 6 months of incubation



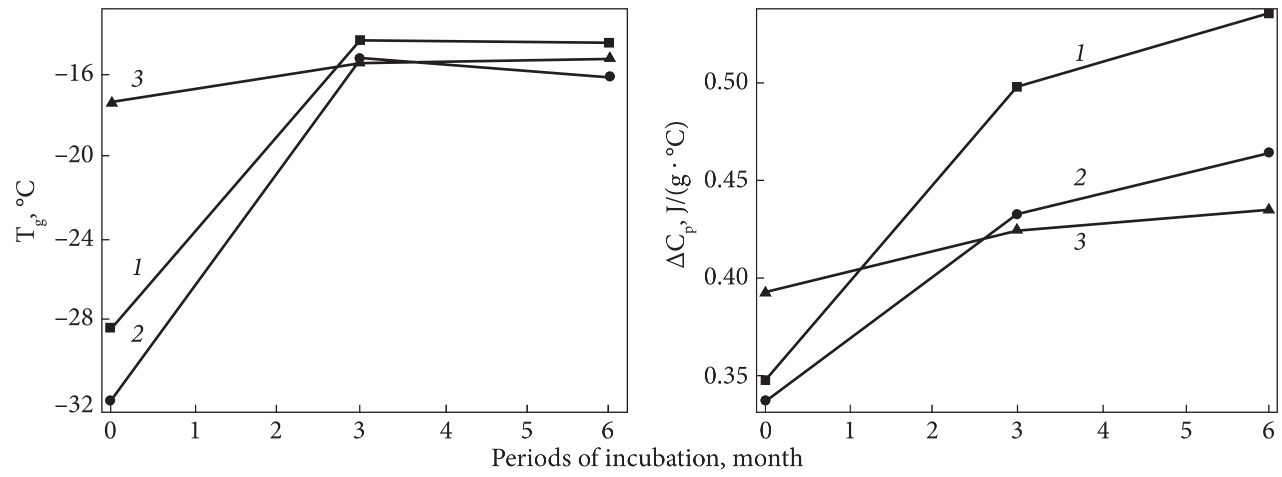
a *b*

Fig. 4. Fragments of the IR spectra of PUU3 before (*1*) and after incubation in the Fenton reagent for 1 month (*2*), 3 months (*3*), and 6 months (*4*) in the ranges of 3800—2800 cm^{-1} (*a*) and 1800—1000 cm^{-1} (*b*)

in the Fenton reagent, similar to PUU1, a decrease in the intensity of the $\nu_{\text{NH-bound}}$ (3292 cm^{-1}) and $\nu_{\text{NH-free}}$ (3518 cm^{-1}) absorption bands was observed. In the 1800—1000 cm^{-1} range (Fig. 3*b*), a redistribution of the intensity of the absorption bands of the urethane fragment's carbonyl groups was observed: non-associated C=O groups with a maximum at 1725 cm^{-1} and associated C=O groups with a maximum at 1712 cm^{-1}. After incubation in the Fenton reagent, an increase in the intensity of the $\nu_{C=O}$ non-associated band at 1725 cm^{-1} and a decrease in the intensity of the $\nu_{C=O}$ associated band at 1712 cm^{-1} were observed, indicating an increase in the number of non-associated carbonyl groups on the sample surface due to the disruption of hydrogen bonds. Moreover, similarly to PUU1, under the influence of the model medium, an increase in the intensity of the δ_{NH} (1537 cm^{-1}) and $\nu_{C—O}$ (1223 and 1083 cm^{-1}) absorption bands was observed, which may be the result of structural changes in the polymer matrix.

For PUU3, in the IR spectral range of 3800—2800 cm^{-1} (Fig. 4*a*), after 3 months of incubation in the Fenton reagent, an increase in the intensity of the absorption bands for $\nu_{\text{NH-bound}}$ (3292 cm^{-1}) and $\nu_{\text{NH-free}}$ (3518 cm^{-1}) was observed. However, after 6 months, their intensity decreased, which may be attributed to the degradation of NH groups or changes in their interactions within the polymer matrix. In the frequency range of 1800—1000 cm^{-1} (Fig. 4*b*), after incubation in the model medium, an increase in the intensity of the absorption band for non-associated $\nu_{C=O}$ (1725 cm^{-1}) and a decrease in the intensity of the absorption band for associated $\nu_{C=O}$ (1712 cm^{-1}) were observed, indicating an increase in the number of non-associated carbonyl groups, similar to the changes observed in PUU2. However, unlike the other samples, for PUU3 after 6 months of incubation, a decrease in the intensity of the δ_{NH} (1537 cm^{-1}) and $\nu_{C—O}$ (1223 and 1083 cm^{-1}) absorption bands was observed, which may indicate destructive processes occurring via a different mechanism or other structural changes in the polymer.

Thus, the analysis of the IR spectra demonstrates changes in the functional groups involved in hydrogen bonding, which may serve as an indicator of structural alterations in the surface layers of the polymer matrix. In particular, the decreases in the intensity of the absorption bands for free NH ($\nu_{\text{NH-free}}$) and hydrogen-bonded NH ($\nu_{\text{NH-bound}}$) suggests a reduction in the number of these groups or a change in their state due to the influence of the model medium. This may indicate disruption of internal hydrogen bonds and partial cleavage of chemical bonds within the polymer matrix. Conversely, an increase in the intensity of these bands could be associated with the formation of new hydrogen bonds between the medium molecules and the polymer matrix. Moreover, the redistribution of the intensities of the absorption bands of the carbonyl groups (associated and non-associated) in the urethane fragment, namely an increase in non-associated $\nu_{C=O}$ and a decrease in associated $\nu_{C=O}$ indicates a weakening of hydrogen bonds in the polymer matrix. Changes in the urea groups (δ_{NH}, $\nu_{C=O}$, $\nu_{C–O}$) observed in the IR spectra suggest a rearrangement of the polymer's surface structure



a *b*

Fig. 5. Changes in the glass transition temperature (T_g) (*a*) and the jump in heat capacity (ΔC_p) at the glass transition (*b*) of the second heating cycle after incubation in the Fenton reagent for PUU1 (*1*), PUU (*2*), and PUU (*3*)

Physical-mechanical properties of PUUs after incubation in the Fenton reagent

PUUs	Periods of incubation			
	control	1 month	3 month	6 month
	σ, MPa			
PUU1	0.60 ± 0.03	0.45 ± 0.01	0.44 ± 0.02	0.38 ± 0.02
PUU2	0.31 ± 0.01	0.41 ± 0.02	0.21 ± 0.01	0.36 ± 0.03
PUU3	0.42 ± 0.03	0.63 ± 0.02	0.50 ± 0.02	0.45 ± 0.02
	ε, %			
PUU1	68.0 ± 2.2	167.6 ± 3.3	235.4 ± 8.2	215.4 ± 8.2
PUU2	47.4 ± 3.0	144.2 ± 9 .9	47.7 ± 1.7	92.1 ± 8.0
PUU3	186.0 ± 9.0	238.7 ± 1 6.7	161.6 ± 9.9	239.9 ± 7.8

under the influence of the model medium. This may result from the degradation or formation of new hydrogen bonds between the molecules of the medium and the polymer matrix.

Thus, the results of the IR spectroscopic studies confirm that the oxidative degradation process of the investigated PUUs is accompanied by the cleavage of chemical bonds in the surface layers, alterations in hydrogen bonding, and a structural rearrangement of the polymer matrix.

The physical-mechanical properties of the samples after incubation in the Fenton reagent for 1, 3, and 6 months change depending on the duration of their exposure to the model medium.

The tensile strength of all PUUs prior to incubation in the Fenton reagent ranged from 0.31 to 0.60 MPa, while after 6 months of incubation it was 0.36—0.45 MPa (Table). For PUU1, a continuous decrease in tensile strength was observed throughout the entire incubation period in the Fenton reagent, which may be due to the oxidative degradation of the polymer matrix. For PUU2 and PUU3, the tensile strength changed nonlinearly. After 1 month of incubation, an increase in tensile strength was observed, which could result from structural rearrangements in the polymer matrix and a redistribution of hydrogen bonds. After 3 months, a decrease in tensile strength compared to the previous period was observed, possibly associated with the initial stages of oxidative degradation. After 6 months, PUU2 exhibited an increase in tensile strength, which might be attributed to the reorganization of the polymer network, whereas PUU3 showed a decrease in tensile strength compared to the 3-month period, indicating further cleavage of bonds within the polymer structure. Compared to the control, after 6 months of incubation, an increase in tensile strength of

7.14—16.13 % was observed for PUU2 and PUU3, which may be associated with changes in the ratio of the amorphous to crystalline phases of the polymer, while a decrease of 36.7 % was observed for PUU1, indicating a significant degree of material degradation.

The relative elongation at break for all PUUs prior to incubation was 47.4—186.0 %, and after 6 months of incubation it increased to 92.1—239.9 % (Table). For PUU1, an increase in relative elongation was observed during the first 3 months of incubation; however, after 6 months, a decrease was noted compared to the previous period, which is consistent with degradation processes. For PUU2 and PUU3, an increase in elongation was observed after 1 month, possibly due to a redistribution of hydrogen bonds within the polymer. After 3 months, a decrease compared to the previous period was noted, indicating the onset of polymer structure degradation. After 6 months, an increase is again observed, which may indicate an adaptation of the polymer structure to the new state. Compared to the control, after 6 months of incubation, the relative elongation at break for all PUUs increased by 28.98—216.76 %, which may be associated with a reorganization of the polymer network and a redistribution of intermolecular interactions.

The results of the physical-mechanical tests can be attributed to a structural rearrangement of the polymer matrix that occurs due to the redistribution of intra- and intermolecular hydrogen bonds. At the same time, oxidative degradation of the polymer under the influence of the model medium affects the mechanical properties of the material. The observed increase in tensile strength and relative elongation at break under the model conditions can be explained by a redistribution of internal hydrogen bonds within the polymer matrix, which temporarily enhances its strength.

Thus, the physical-mechanical properties depend on the duration of incubation in the Fenton reagent and change nonlinearly. After 6 months of incubation, compared to the control, PUU2 and PUU3 demonstrate an increase in both tensile strength and relative elongation at break, whereas PUU1 experiences a significant decrease in tensile strength, indicating substantial material degradation.

According to the DSC data, after incubation of PUU samples in the Fenton reagent, an increase in glass transition temperature (T_g) and a change in heat capacity (ΔC_p) at the second heating glass transition was observed (Fig. 5). To eliminate the influence of the thermal and mechanical history of the polymer material, two heating cycles were performed. The results of the second heating are the most reliable material characteristics.

In particular, after 6 months of incubation in the model medium, the glass transition temperature of PUU increases by 12.6—50.0 %, and the ΔC_p value increases by 10.8—53.9, %.

It should be noted that the changes for PUU3 are less intense compared to the changes observed in the other PUUs. Specifically, T_g for PUU3 increased by 12.6 %, and ΔC_p by 10.8 % after 6 months of incubation in the Fenton reagent. This indicates fewer structural changes in this sample compared to PUU1 and PUU2.

During oxidative degradation in the model medium, the breakdown of weaker, soft segments of the polyurethane-ureas could lead to an increase in the proportion of hard segments, a reduction in the segmental mobility of macromolecular chains, and a decrease in the free volume. Therefore, the increase in T_g and ΔC_p compared to the control is evidence of structural changes in the polymer matrix, particularly the densification of the structure, restriction of segmental mobility, and strengthening of intermolecular interactions.

The results of the *in vitro* studies confirm that materials based on PUUs containing segments of macrochain extender DADPh and the grafted copolymer PVA—PEG undergo the process of oxidative biodegradation under the influence of the model medium. This suggests their potential suitability for medical applications as temporary polymer matrices.

Conclusions

The study investigated the biodegradation capability of PUUs containing fragments of the macrochain extender DADPh and grafted copolymer PVA—PEG to undergo structural changes *in vitro* under simulated inflammatory process conditions. Changes in the structure, physical-mechanical, and thermophysical properties were assessed under the influence of the Fenton reagent over 1, 3, and 6 months of incubation. The experimental results show that oxidative degradation of the PUUs investigated, induced by the Fenton reagent, is accompanied by the cleavage of specific chemical bonds, changes in hydrogen bonding, and structural transformations of the polymer matrix, including densification and restricted segmental mobility. IR spectroscopy demonstrated that during oxidative degradation, the polyester components are the first to break down, as evidenced by the reduced intensity of C—O—C bands (1100—1000 cm^{-1}) and an increase in the glass transition temperature (T_g) of 12.6—50.0 %. Further degradation of urethane (νC=O) and urea (δNH, νC=O, νC—O) bonds progresses at later stages, explaining the nonlinear changes in the physical-mechanical characteristics. According to DSC data, after incubation in the Fenton reagent, an increase in T_g of 12.6—50.0 % and a jump in heat capacity (ΔC_p) of 10.8—53.9 % are observed, indicating the densification of the polymer structure and a decrease in the free volume of macromolecular chains. This confirms the reduction in segmental mobility and the strengthening of intermolecular interactions, which was also confirmed by the physical-mechanical tests. The polyester segment is found to be the first to break down, resulting in reduced polymer flexibility and an increase in T_g, while the subsequent breakdown of urethane and urea bonds leads to a reduction in mechanical strength and changes in thermophysical characteristics. At the same time, PUU3 exhibits fewer structural changes compared to PUU1 and PUU2, which is confirmed by the less pronounced increase in T_g (12.6 %) and ΔC_p (10.8 %).

Thus, it has been established that under the conditions of the model medium, the PUU samples undergo structural changes that are reflected in alterations in hydrogen bonding interactions, and partial degradation of the polymer matrix. The obtained in vitro results confirm that polymer materials based on PUUs containing fragments of DADPh and PVA—PEG demonstrate theprocess of oxidative biodegradation under the influence of the model medium, which may indicate their potential suitability as temporary polymer matrices for medical applications.

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ДОСЛІДЖЕННЯ БІОДЕСТРУКЦІЇ ПОЛІУРЕТАНСЕЧОВИН ІЗ ФРАГМЕНТАМИ ПРИЩЕПЛЕНОГО КОПОЛІМЕРУ ПОЛІВІНІЛОВИЙ СПИРТ-ПОЛІЕТИЛЕНГЛІКОЛЬ У СТРУКТУРІ В МОДЕЛЬНИХ УМОВАХ ЗАПАЛЬНОГО ПРОЦЕСУ

Досліджено здатність до біодеструкції гідрофільних поліуретансечовин (ПУС) із фрагментами 4,4'-діамінодифенілметану (ДАДФ) і прищепленого кополімеру полівініловий спирт—поліетиленгліколь (ПВС—ПЕГ) у структурі за модельних умов запального процесу при контакті матеріалу з кров'ю. Здатність до біодеструкції оцінювали методами ІЧ-спектроскопії, фізико-механічними випробуваннями та методом ДСК за зміною структури, фізико-механічних і теплофізичних властивостей ПУС під впливом реактива Фентона протягом 1, 3 і 6 місяців інкубації. За даними ІЧ-спектроскопії видно, що під впливом реактиву Фентона відбувається процес окиснювальної деструкції досліджуваних ПУС, який супроводжується розщепленням окремих хімічних зв'язків, зміною водневих взаємодій і структурними перетвореннями полімерної матриці. Фізико-механічні показники залежать від терміну інкубації у реактиві Фентона та змінюються нелінійно. Після 6 місяців інкубації ПУС порівняно з контролем спостерігали підвищення міцності і відносного подовження при розриві, що можна пояснити формуванням нових водневих зв'язків під впливом модельного середовища. За результатами ДСК, після інкубації зразків ПУС у реактиві Фентона спостерігали підвищення температури склування (T_c) і стрибка теплоємності (ΔC_p) при склуванні, що свідчить про структурні зміни у процесі окиснювальної деструкції. Отже, досліджувані матеріалидемонструютьпроцесокислювальноїдеструкціїпід впливом модельного середовища. Тому, ПУС із фрагментами прищепленого кополімеру ПВС—ПЕГ у структурі є перспективною полімерною матрицею медичного призначення тимчасового терміну дії.

Ключові слова: поліуретансечовина, прищеплений кополімер полівініловий спирт—поліетиленгліколь, окиснювальна біодеструкція, реактив Фентона.