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EFFECTS OF THE RHODAMINE 6G AND RHODAMINE B DYES ON PHOTO- AND THERMOSTABILITY OF POLYURETHANES AS LASER ACTIVE MEDIA

Pure and doped with xanthene dyes Rhodamine 6G and Rhodamine B polyurethane materials were exposed to intense radiation and tested by means of IR spectroscopy. The sensitizing effect of the dyes on photodestructive processes in the polymers was shown, and it was more pronounced in the case of Rhodamine 6G. According to the results of thermogravimetric analysis, the incorporated dyes caused insignificant changes in the thermal stability of polyurethane. The triplex construction of samples was proved to be an effective protection of the polymer films from the action of atmospheric oxygen.

Keywords: IR spectroscopy, thermogravimetric analysis, polyurethane, Rhodamine 6G, Rhodamine B.

1. Introduction

The polyurethanes (PU) are known as polymers used in different fields due to a wide range of their properties, which can be purposefully modified by varying their chemical nature, structure and composition [1]. One of the PU application areas is laser technology, in particular, their use as matrices of solid-state active elements in organic dye lasers and passive Q-switches [2–5].

Polyurethane matrices have advantages such as easy dye introduction and material homogeneity, which is especially important for obtaining high-quality generation with a narrow spectral width. The long operation lifetime and generation efficiency of the mentioned laser elements are largely determined by the physicochemical properties of the chosen polymer matrices, in particular, their resistance to irradiation.

A number of works discuss photooxidative and photochemical processes in polyurethanes, which determine stability of these materials (for example, [6–13]). When the molecules of the introduced dyes are excited under irradiation, part of this energy is not radiated and converted into heat, and this effect contributes to the degradation of the dye. However, the presence of the dye could be an additional decomposition source for the polymer, as evidenced by the monograph [12], which presents data on the sensitization of polymer photooxidative destruction by various dyes. It should be noted that in the area of local heating in laser materials, photodestruction of both dyes and polymers is also accompanied by the thermodestructive and thermooxidative processes. Therefore, we considered it important to investigate the effect of introduced dyes on the irradiation resistance and thermodestructive processes of PU doped with the xanthene dyes Rhodamine 6G (R6G) and Rhodamine B (RB), which are widely used in laser technology.

2. Experimental

2.1. Materials and objects of the study

- Polyoxypropylene glycol with a molecular weight of 1000 (PPG) (Rakita, Poland), dried under vacuum at

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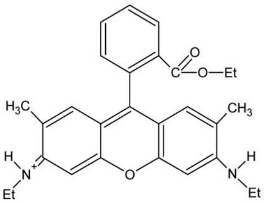
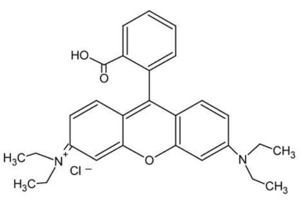
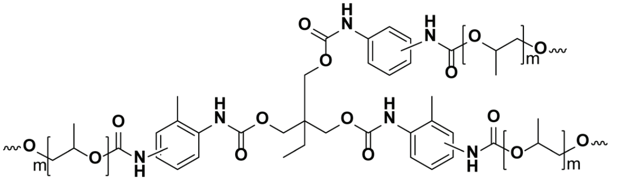
$t = 70$–80 °C and $P = 5$–6 mm Hg for 5–6 hours and stored in an atmosphere of dry nitrogen;

- 2,4-toluene diisocyanate (TDI) (81.4%, Merck), used without additional purification;
- trimethylolpropane (TMP) (99.9%, Merck), dried under vacuum at $f = 48$ °C for 60 hours;
- two xanthene dyes: Rhodamine 6G (R6G) and Rhodamine B (RB), (>99.5%, Exciton, Inc.), used without additional purification:


R6G


RB

The objects of the study were pure and dye-doped (R6G and RB) polyurethanes (PU), based on PPG, TDI, TMP:



Synthesis of the initial (PU_0) and doped (PU_{R6G} and PU_{RB}, respectively) PU was carried out according to [14]. The concentration of dyes was 1×10^{-4} mol/L.

2.2. Methods

Photochemical experiments were performed using the integrated ultraviolet and visible radiation of an ultrahigh-pressure mercury arc lamp DRK-120. Studies on the photostability of the PU films were carried out by irradiating the sample area with the light of the specified lamp at an irradiation dose of 1.5 kJ/cm^2 (the films were exposed to air). The choice of the irradiation dose was determined by the known degree of degradation of R6G in the polyurethane acrylate (PUA) matrix studied in detail earlier [15].

IR spectra of the original and irradiated films were obtained using a Tensor-37 Fourier-transform spectrometer from Bruker (Germany) in the mid-IR range with a resolution of 4 cm^{-1}. The data were recorded from the film surface with a multiple disrupted total internal reflection attachment.

To study polymer degradation processes with a simultaneous monitoring of the polymer mass and the rate of mass change, thermogravimetric analysis (TGA) was used. The experiments were performed on a Derivatograph Q–1500D of F. Paulik, J. Paulik, L. Erdey system in the temperature range of 20–700 °C in air atmosphere with continuous removal of gaseous degradation products. Heating rate was 10 degrees/min. The sample weight was 50 mg. The materials were pre-grinded under conditions that limit mechanical degradation. Temperature intervals of the decomposition stages were estimated from the differential mass loss curves (DTG) taking into account the proportional correlation between the peak area of the DTG curve and the mass loss at the corresponding stage (TG), and the quality of stage separation on the DTG curves significantly exceeds the capabilities of integral weight loss curves (TG).

3. Results and Discussion

The polyurethane chosen for the study is an effective laser medium [15–18]. Due to the presence of a carboxyl group in the molecule, the xanthene dye RB used in this study can be covalently bound to the polymer chain, as shown in [19]. This was proved by the IR spectral investigations of the interaction product between the RB and TDI [19].

3.1. Effects of R6G and RB dyes on the PU photo- and photooxidative degradation

Despite numerous data on the PU photooxidative degradation, interpreting the results turned out to be quite difficult. Fig. 1 presents the IR spectra of initial and irradiated, pure and RB, R6G dye-doped PU. The spectra shown are typical for PU.

To study the changes observed during irradiation of the samples, some fragments of their IR spectra were analyzed. Fig. 2 shows PU spectral regions with a broad structureless absorption band with a maximum at 3295 cm^{-1}, corresponding to the stretching vibrations of NH groups, ν(NH) (3500–3100 cm^{-1}). The introduction of dyes into the original PU_0 does not affect the position of the absorption peak, but causes a slight narrowing of the contour (by ~7–9 cm^{-1}). After irradiation, the half-width of the absorption band increases (by ~12–18 cm^{-1}), and as a result, the spectral contour becomes broader compared to the initial samples. These changes are more pronounced in the

Table 1. **Graphical decomposition of the –C=O stretching vibration band in the pure and RB dye-doped polyurethanes before and after irradiation**

Samples	ν_1 peak position, cm^{-1}		ν_1 relative intensity, %		ν_2 peak position, cm^{-1}		ν_2 relative intensity, %		$I_{\nu1/\nu2}$ ratio	
	before	after	before	after	before	after	before	after	before	after
PU_0	1705	1709	44	72	1728	1731	56	28	0.79	2.57
PU_{RB}	1711	1711	83	78	1731	1731	17	22	4.88	3.55

Table 2. **Graphical decomposition of the –C=O stretching vibration band in the R6G dye-doped polyurethane before and after irradiation**

ν_1 and ν_2 peak positions, cm^{-1}		ν_1 and ν_2 relative integral intensity, %		$I_{\nu1/\nu2}$ ratio	
before	after	before	after	before	after
1709	1674	82	15	4.56	
1731	1700	18	31		0.62
	1725		50		
	1774		4		

irradiated R6G dye-doped PU material. Some spectral broadening was also observed earlier upon irradiation of the original PU_0 [20]. The mentioned effects are attributed to the possible redistribution of the hydrogen bond network, accumulation of the additional hydroxyl groups during the oxidation process, and the formation of new hydrogen-bonded associates with both lower and higher interaction energies.

The analysis of changes in IR spectral fragments with the absorption contours, characteristic of the carbonyl group stretching vibrations, ν(C=O) (~1700 cm^{-1}) provides the possibility of explaining photooxidative processes in the PU during irradiation. Fig. 3 shows the C=O vibration band of the irradiated pure and dye-doped PU with a graphical decomposition into the components (Gaussian distribution) and its comparison with the spectra of initial unirradiated samples. Table 1 lists the values of the relative integral intensity for the low- (ν_1) and high-frequency (ν_2) separated peaks and their ratios for the initial and irradiated, pure and RB-colored PU. Table 2 presents the same data for the corresponding PU samples in the case of the R6G dye.

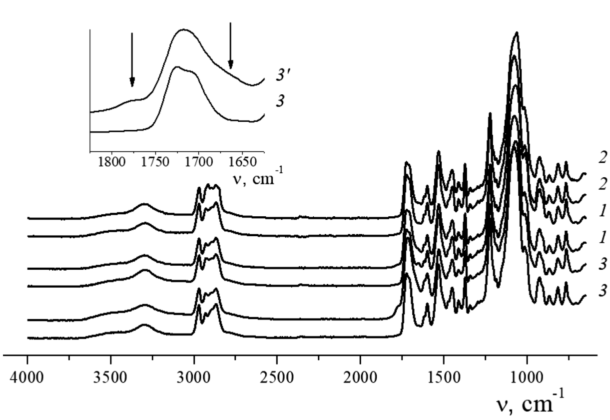


Fig. 1. The IR spectra of initial and irradiated PU_0 (*1, 1'*), doped with the dyes PU_{RB} (*2, 2'*) and PU_{R6G} (*3, 3'*), respectively. The inset shows a spectral fragment of the COC vibrations for the initial (*3*) and irradiated (*3'*) PU_{R6G} dye-doped polymer

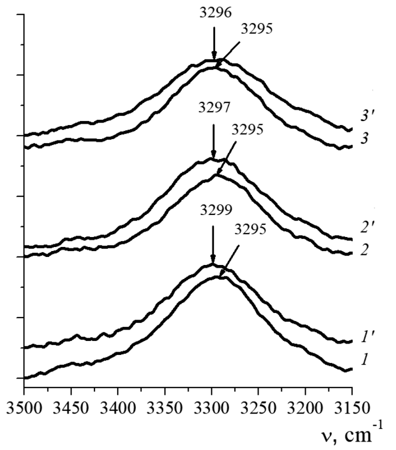


Fig. 2. Fragments of the IR spectra in the region of NH group stretching vibrations for initial and irradiated pure PU_0 (*1, 1'*), and dye-doped PU_{RB} (*2, 2'*) and PU_{R6G} (*3, 3'*)

The structure of the PU, synthesized from the PPG and TDI is known to include flexible and rigid segments, which causes their thermodynamic incompatibility and microphase separation with the formation of a domain alignment [21]. The IR spectroscopic experiments allow a detailed description of intermolecu-

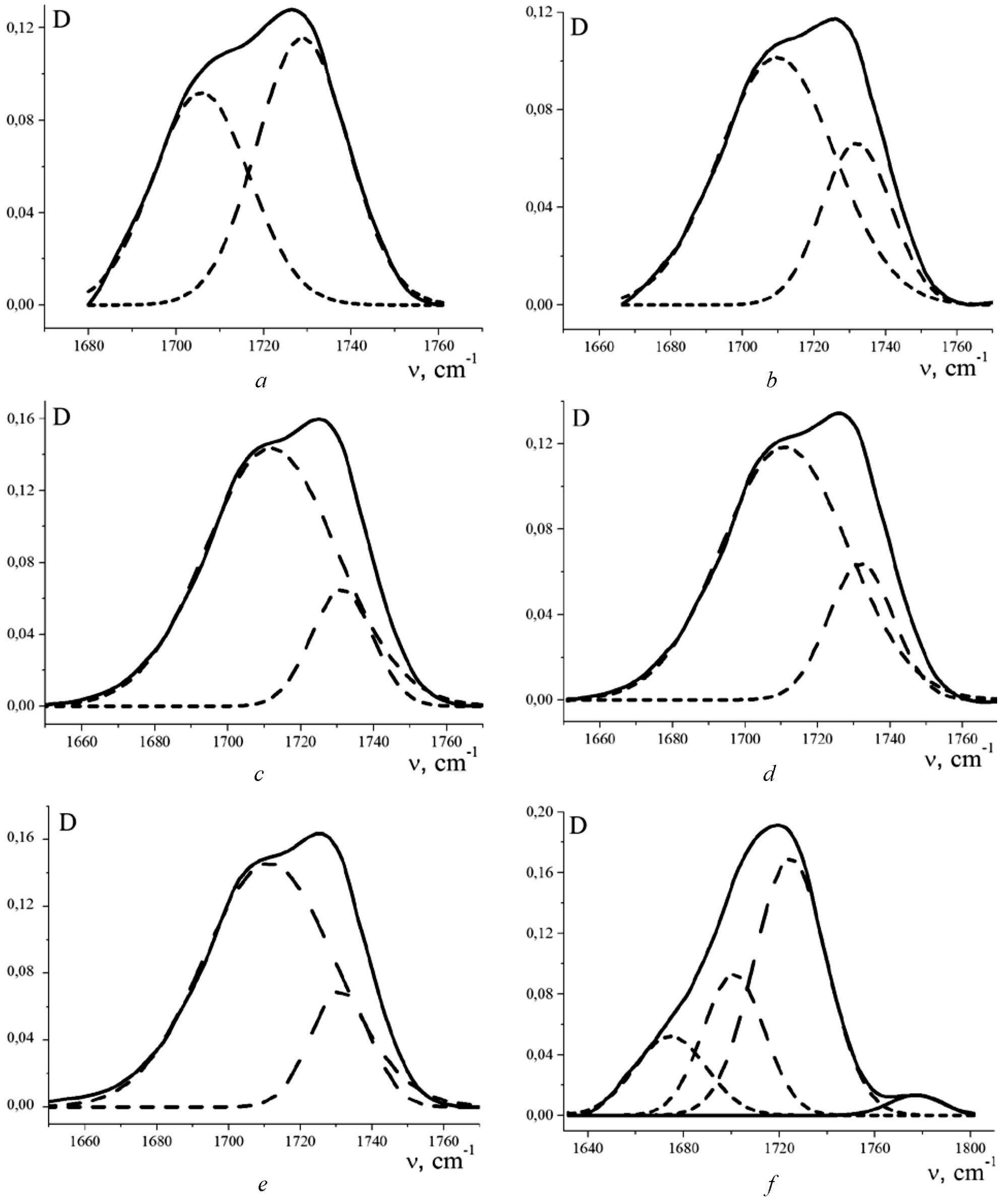


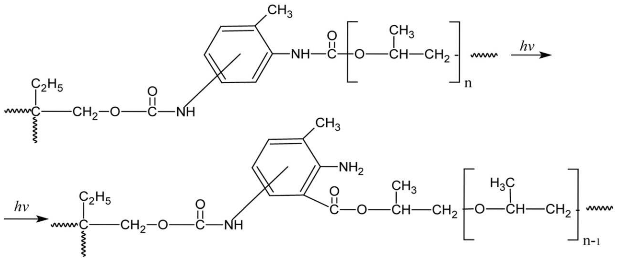
Fig. 3. Fragments of the IR spectra in the region of C=O group stretching vibrations for the initial (*a*) and irradiated (*b*) pure PU_0, the dye-doped initial (*c*) and irradiated (*d*) PU_{RB}, and the initial (*e*) and irradiated (*f*) PU_{R6G}, decomposed using Gaussian distribution

lar interactions between these components [21]. In the segregated PU, the stretching vibration band of the C=O bond in the urethane group – Amide I, ν(C=O) takes on a complex shape and splits into two spectral peaks, corresponding to different types of interactions [21]. The position of this band is sensitive to the environment and interactions of the urethane group [21,22]. The latter can be located either inside a rigid block, where urethane-urethane hydrogen bonds are mainly realized (a low-frequency component, ν_1), or in a polyether environment with the urethane-oxygen interactions of the ether group [22], or can belong to the free C=O groups (a high-frequency component, ν_2) [6, 8] (Fig. 3, *a*).

The analysis of these spectral fragments for the pure and dye-doped PU presented in [23] showed the redistribution of the relative intensities of the low- and high-frequency absorption peaks upon the introduction of dyes into the polymer. This indicates that in the presence of dyes, the self-association of urethane groups within the rigid segments prevails, whereas the interaction between urethane groups and oligoether flexible parts decreases. At the same time, greater steric restriction in the case of the PU with covalently bound RB results in the higher intensity ratio ($I_{\nu 1/\nu 2}$) of the low- to the high-frequency spectral components (Table 1, 2).

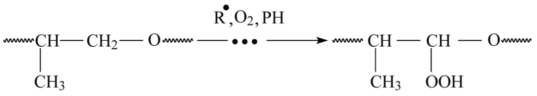
Upon irradiation of the pure PU_0 [24], the complex stretching vibration band of C=O groups (Amide I) retains its two-component structure (Fig. 3, *b*), but there is also some intensity redistribution, observed for the ν_1 and ν_2 peaks with a blue shift of the latter one (Table 1). The increased value of $I_{\nu 1/\nu 2}$ after irradiation indicates that a significant part of the associated C=O groups is located inside the rigid domains, and the possible photooxidative processes occur in the oligoether blocks [24].

In the irradiated PU_{RB} sample, the contour of the stretching vibration band remains practically the same compared to the initial sample, but there is a decrease in the $I_{\nu 1/\nu 2}$ ratio (Fig. 3, *c, d*, Table 1). This indicates that a significant part of the associated C=O groups is located inside the rigid domains. However, such changes in relative intensity can also be caused by the realization of numerous photooxidative radical reactions during irradiation with the formation of various oxidation products [11]. The ultrahigh-pressure mercury lamp used to irradiate the samples is characterized by a wide-range spectrum (polychromatic light), so during irradiation, the probability of Fries transformation (rearrangement) is not excluded, which usually occurs at $\lambda < 300$ nm:



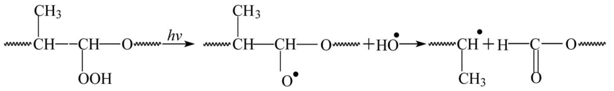
Because of such rearrangement, amino and ester groups appear, which can also affect the IR spectra of the irradiated films.

According to [8], light absorption by aromatic chromophores can cause oxidation of polyether fragments, and the PU oligoether fragments have a sufficiently high reactivity towards oxidation:



$$\sim\underset{CH_3}{CH}-CH_2-O\sim-\bullet\bullet\bullet\xrightarrow{\dot{R},O_2,PH}\sim\underset{CH_3}{CH}-\underset{OOH}{CH}-O\sim$$

The authors of Ref. [11] showed that the main decomposition pathway of secondary peroxides is the formation of formates:

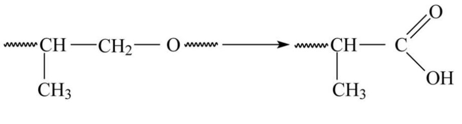


$$\sim\overset{CH_3}{CH}-\underset{OOH}{CH}-O\sim\xrightarrow{h\nu}\sim\overset{CH_3}{CH}-\underset{O^{\bullet}}{CH}-O\sim+H\dot{O}\longrightarrow\sim\underset{CH_3}{\dot{CH}}+H-\underset{\overset{\|}{O}}{C}-O\sim$$

The formate group formed in this reaction can also affect the IR spectral region of the complex carbonyl band.

In the irradiated PU_{R6G}, the C=O stretching vibration absorption is seen as a more extended contour with the appearance of a weak band on the left and a pronounced shoulder on the right (Fig. 1, inset). Its graphical decomposition is presented in Fig. 3, *f*. Here, four spectral components can be distinguished at 1674, 1700, 1725, and 1774 cm^{-1}.

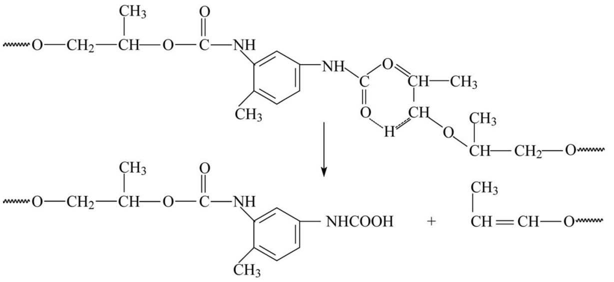
The relative intensity of these peaks is given in Table 2. According to [8], the appearance of the vibration maximum at 1774 cm^{-1} is due to the formation of saturated carboxylic acids during the oxidation of the PU oligoether fragments:



$$\sim\underset{CH_3}{CH}-CH_2-O\sim\longrightarrow\sim\underset{CH_3}{CH}-C\begin{array}{l}/\!\!/O\\ \backslash OH\end{array}$$

There, the presence of such carboxylic acids was confirmed by the IR spectral analysis of the SF_4-treated, irradiated PU film based on diphenylmethane diisocyanate and polytetramethylene glycol.

The vibration component at 1674 cm^{-1} can be ascribed to the formation of compounds with unsaturated C=C double bonds (Norrish reaction of type II) [6]:



Significant spectral changes in the irradiated PU_{R6G} sample are reflected in the relative intensity ratio of the absorption bands at ∼1700 and 1725 cm^{-1}. The ν_1 and ν_2 peak positions are shifted towards low frequencies relative to the unirradiated initial PU_{R6G} and almost coincides with the maxima of the corresponding bands in the unirradiated pure PU_0 (Tables 1, 2). Here, the ν_1 and ν_2 relative intensity redistribution is also observed (Fig. 3, *e, f*, Table 2), which evidences a significant decrease in the part of the associated C=O groups located inside the rigid domains. The higher intensity of the ν_2 spectral peak, which is a characteristic of the interactions between the urethane and oxygen of the ether or the C=O groups of various compounds, formed during oxidation processes, most likely, photooxidative ones (Fries, Norrish reactions, photooxidation reactions in the polyether block) [8].

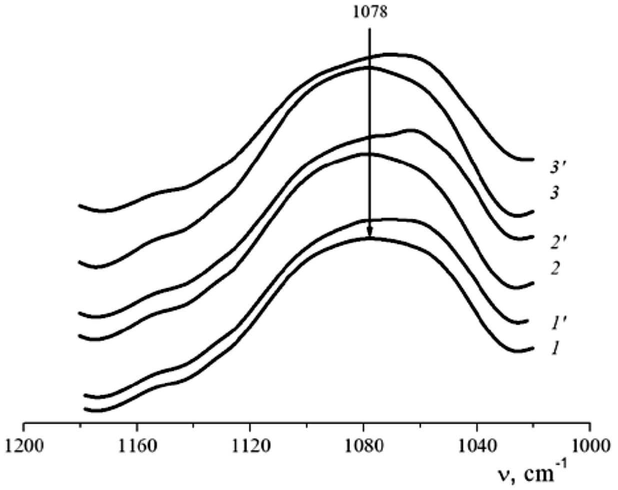


Fig. 4. Fragments of the IR spectra in the region of C–O–C asymmetric stretching vibrations for initial and irradiated pure PU_0 (*1, 1′*), and dye-doped PU_{RB} (*2, 2′*) and PU_{R6G} (*3, 3′*)

The above IR spectral analysis shows that more intense destructive processes occur in the R6G-doped PU. This can be due to the presence of covalent bonds between the RB dye and PU chains. Therefore, upon irradiation, when the dye molecules become excited, part of the non-radiative energy is transferred to the polymer through such chemical bonds. It may be assumed that this energy is distributed more or less evenly along the polymer chains and it is partially scattered. The R6G dye molecules are not fixed to the PU and upon irradiation, can be located in its more mobile parts (polyether segments), accidentally transferring their non-radiative energy and causing various photooxidative and photoradical processes there.

The absorption band of ν(C–O–C) symmetric and asymmetric stretching vibrations, which also characterizes photooxidative processes, is quite narrow and intense in all the studied samples (Fig. 1). Its detailed analysis in the spectral range of 1020–1180 cm^{-1} has been performed (Fig. 4).

A significant intensity redistribution is observed in the spectral range of 1030–1100 cm^{-1} (Fig. 4). The intense band at 1078 cm^{-1} in the unirradiated pure and dye-doped PU is ascribed to the asymmetric stretching vibrations of the C–O–C ether bonds. In the irradiated samples, it acquires a more extended contour and its maximum is shifted towards lower frequencies. Such spectral changes can be associated with the appearance of hydroperoxide (COOH) or alcohol (COH) groups during irradiation, the C–O vibrations of which absorb in the range of 1030–1050 cm^{-1} [21, 22]. Peroxide and hydroperoxide fragments are quite easily decomposed into free radicals and activate the polymer oxidation.

Thus, the IR spectral analysis of the irradiated pure and dye-doped polymer films indicates the occurrence of deeper destructive processes in the dye-doped PU materials, which is in agreement with [12], where the data on the dye sensitization of the polymer photooxidative destruction is presented.

3.2. The R6G and RB dye effects on the PU thermal and thermo-oxidative degradation

Thermal properties of the pure and dye-doped PU matrices were investigated by the thermogravimetric analysis (TGA). Fig. 5 shows the TGA curves for

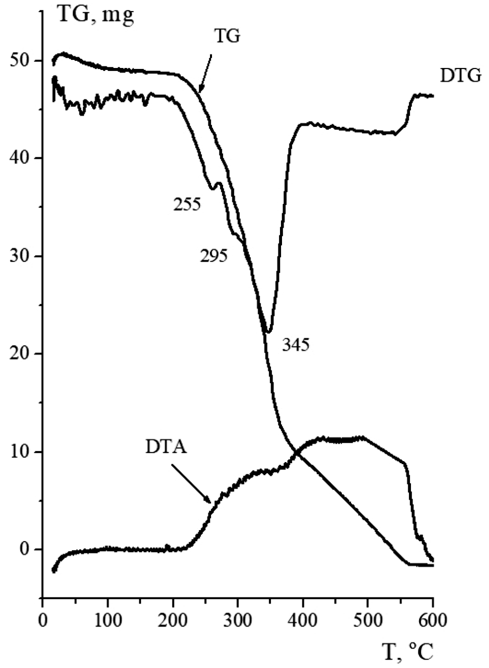
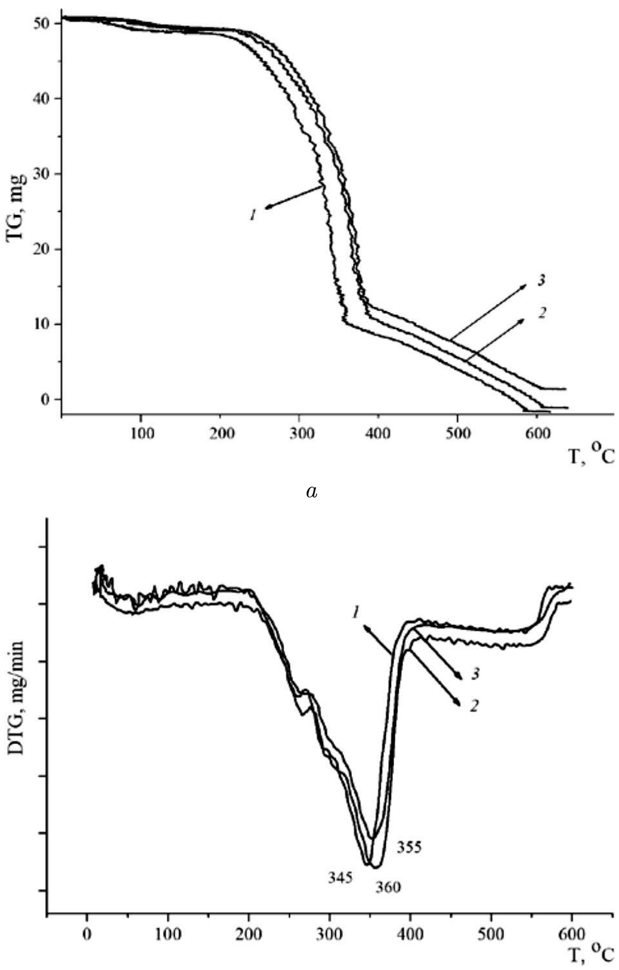


Fig. 5. TGA curves of the initial PU_0 polymer

Table 3. **Parameters of pure and dye-doped PU thermal oxidative destruction**

Samples	$T_{\max}$ of the weight loss rate, °C	Weight loss at $T_{\max}$, %	Weight loss at selected temperatures, %		
			250	400	500
PU_0	345	56	10	82	95
PU_{R6G}	355	50	8	76	88
PU_{RB}	360	42	6	78	92

the initial PU_0, namely, the TG and DTG (integral and differential weight loss, respectively), and the DTA (differential thermal analysis) curves. According to these data, the weight loss in the temperature range 100–220 °C is not accompanied by a noticeable heat release and corresponds to the thermal decay of the polymer. The primary products of such decomposition are the initial diisocyanates and diols, which upon further heating participate in oxidative processes and secondary reactions [11, 13, 25].



a

b

Fig. 6. Temperature dependences of pure PU_0 (*1*) and dye-doped PU_{RB} (*2*) and PU_{R6G} (*3*) weight loss: TG (*a*), DTG (*b*)

The stages of weight loss in the temperature range 250–400 °C occur with intense heat release and correspond to thermo-oxidative processes of the PU degradation. In particular, the rate of weight loss with a characteristic temperature of 255 °C is assigned to the burnout of secondary reaction products in the liquid state, and the maximum decomposition rate with a characteristic temperature of 345 °C corresponds to the burnout of gaseous products. The high temperatures above 500 °C are known as a region, where the carbon skeleton burns out.

The TG and DTG curves of the dye-doped polymer samples (Fig. 6) demonstrate the effect of dyes on the PU resistance to thermal oxidative decay. Three temperature points have been chosen for comparison: 250 °C (approximately the onset of thermal oxidative decomposition); 400 °C (the end of the rapid thermal oxidative destruction) and 500 °C (the region of carbon skeleton burnout). The corresponding weight loss

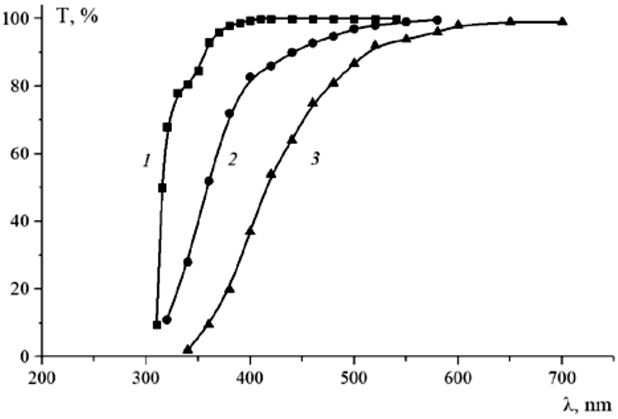


Fig. 7. Transmission spectra of pure PU layer between quartz substrates before (*1*) and after (*2*) irradiation and the irradiated unprotected PU film (*3*)

data, as well as the temperatures of the maximum weight loss rate and the weight loss at the moment of reaching this temperature are given in Table 3.

The Fig. 6 and Table 3 show that the introduction of dyes does not significantly affect the polymer thermal decomposition stage. However, in the temperature region of thermo-oxidative destruction, a noticeable improvement in the thermal stability of dye-doped PU materials is observed. Although the PU thermo-oxidative destruction starts at nearly the same temperature for all three samples, in the intensive burnout region, the weight loss is less in the dye-doped PU materials (Table 3). The patterns of the TG curves in the temperature range 250–350 °C are almost similar for the $\mathrm{PU_{RB}}$ and $\mathrm{PU_{R6G}}$ (Fig. 6, *a*) with slightly smaller weight losses in the case of the RB dye. The presence of a chemical bond between the dye and polymer chains has a small effect on the thermo-oxidative destruction of the system, based on the studied PU.

It should also be noted that the temperature of the maximum decomposition rate at the intensive burnout stage in the dye-doped PU shifts to higher values (Table 3, Fig. 6, *b*). This indicates a slowdown of the polymer destruction in this temperature range in the presence of dyes. At temperatures from 400 and above 500 °C, when the carbon skeleton burns out, more intensive burnout is observed in the $\mathrm{PU_{RB}}$ sample, while the pure $\mathrm{PU_0}$ and $\mathrm{PU_{R6G}}$ possess nearly the same burnout rates. However, the weight loss in the dye-doped PU is slightly less even in this case.

Thus, based on the results of thermogravimetric analysis, it can be concluded that the presence of R6G and RB xanthene dyes in the PU, regardless of the covalent bond formation with the polymer chain, somewhat improves the polymer resistance to thermal oxidation, in contrast to the photooxidative properties.

To reduce the effect of atmospheric oxygen on the stability of the polymer, a triplex [26] or multiplex [27] design is used, where the dye-doped polymer layer is confined by glass substrates. In addition, in such designs of laser elements, heat removal from the active zone is improved due to the higher thermal conductivity of glass compared to the polymer. The use of such designs is one of the effective ways to protect the PU films. Fig. 7 shows the electronic transmission spectra of the protected and unprotected PU films. Minor changes after irradiation in the spectra of the protected PU film relative to the unprotected one indicate the effectiveness of polymer protection in the triplex design.

4. Conclusions

IR-studies of the irradiated pure and R6G and RB doped polyurethane materials show that the dye introduction into the polymer matrix sensitizes photooxidative and photodestructive processes relative to the initial PU. At the same time, these processes occur more intensively in the R6G dye-doped PU.

According to the TGA results, the presence of xanthene dyes slightly improves the PU thermal stability, and the formation or absence of a covalent bond between the dye and polymer chains has no effect on the PU thermal destruction.

An effective way demonstrated to protect the dye-doped polymer films as active laser media from the action of atmospheric oxygen and provide better heat dissipation in these materials is to use the triplex design.

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ВПЛИВ БАРВНИКІВ РОДАМІН 6Ж І РОДАМІН Б НА ФОТО- ТА ТЕРМОСТАБІЛЬНІСТЬ ПОЛІУРЕТАНІВ В ЯКОСТІ АКТИВНОГО СЕРЕДОВИЩА ДЛЯ ЛАЗЕРІВ

Поліуретанові матеріали, чисті та леговані ксантеновими барвниками родаміну 6Ж та родаміну Б, були піддані інтенсивному опроміненню та досліджені за допомогою ІЧ-спектроскопії. Було показано сенсибілізаційний вплив барвників на фотодеструктивні процеси в полімерах, який був більш вираженим у випадку родаміну 6Ж. Згідно з результатами термогравіметричного аналізу, введені барвники спричиняли незначні зміни термічної стабільності поліуретану. Було доведено, що триплексна конструкція зразків є ефективним захистом полімерних плівок від дії кисню повітря.

Ключові слова: ІЧ-спектроскопія, термогравіметричний аналіз, поліуретан, родамін 6Ж, родамін Б.