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RADIATION CHEMISTRY

Gamma- and Electron Beam Radiation-Induced Degradation of Poly-L-Lactide

L. P. Krul'[a, b, *], **G. V. Butovskaya**[b], **A. A. Fedorenko**[a, b], **L. P. Roginets**[c], and **I. A. Sal'nikova**[c]

[a] *Belarusian State University, Minsk, 220030 Belarus*
[b] *Research Institute of Physical and Chemical Problems, Belarusian State University, Minsk, 220030 Belarus*
[c] *Joint Institute for Energy and Nuclear Research, National Academy of Sciences of Belarus,*
PO Box 119, Sosny, Minsk, 220109 Belarus
**e-mail: krul@bsu.by*

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Abstract—Comparative data have been obtained on the effect of the absorbed dose of γ-radiation and electron beam on the radiation-induced degradation of poly-L-lactide in the presence of oxygen provided dose distribution through the irradiated polymer bulk is uniform and high-energy radiation heating is insignificant to cause change of relaxation and phase transitions in the polymer matrix. The efficiency of radiation-induced degradation of poly-L-lactide exposed to γ-radiation has been shown to be higher than that in the case of electron beam due to a longer irradiation time in the former case resulting in a more essential contribution of an oxidative component in degradation.

Keywords: poly-L-lactide, electron beam, gamma-radiation, molecular weight, radiation-chemical yield

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Semicrystalline poly-L-lactide (PLL) is the most important representative of biodegradable biocompatible thermoplastic aliphatic polyesters synthesized from lactic acid or its cyclic dimers (lactides) [1]. The most widely polylactides (**PL**s) are used in medicine [2–11] and as packaging materials for food products [12, 13]. Modern PL-based medical polymer materials and packaging films are often specially subjected to ionizing radiation of various types. For example, γ-radiation or electron-beam treatment of medical items made from polylactides ensures their sterilization [14]. In addition, the radiation processing of polylactides makes it possible to control the rate of degradation of such products in the human body [2–10]. Electron beam dispersion of a PLL powder is used in technology of nanoscale antibacterial coatings deposition onto surgical implants from the active gas phase [11]. Phytosanitary treatment for killing insects and their larvae in vegetables and fruits, as well as microbiological pathogens in meat of mammals and poultry, with fast electrons or γ-radiation is used to increase the safety of food products packed in PLL films [12]. In addition, radiation treatment makes it possible to speed up the composting of used PLL packaging and this polymer industrial waste [15, 16].

Already in the early works related to the effects of ionizing radiation on polylactides, it was shown that these polymers are predominantly degradable [2, 17–19]. However, reliable comparable experimental data on the effect of the most common types of ionizing radiation (γ-rays and high-energy electron beams) on the occurrence of radiation-induced degradation processes in semicrystalline PLL are still missing. This complicates the choice of both the type of ionizing radiation source that is the most suitable for solving specific practical problems and radiation treatment conditions that provide the desired degree of polymer degradation during irradiation.

Overwhelming majority of the works published [2, 4–13, 15–25] deal with transformations of PLL macromolecules under the effect of only one particular kind of ionizing radiation, the irradiation being carried out not only in the absence of oxygen, but also in air, since the latter option is of practical importance. It is impossible to make a correct comparison of the published data on the degradation of PLL by the action of γ-radiation and high-energy electron beam. These data, as a rule, relate to polymers synthesized under different conditions and to experimental samples that differed in form (granules, powders, finished products) and methods and conditions for their preparation. There are quite few works devoted to the comparative study of the effect of γ-radiation and electron beam on experimental samples that have the same form and have been prepared from PLL of the same origin according to the same procedure [3, 16]. Nuutinen [3] studied the effect of sterilizing doses of ionizing radiation on the crystallinity and mechanical properties of surgical sutures made of poly-L-lactide, as well as on the characteristic viscosity of their solutions before and after radiation sterilization, but this is not enough to understand the characteristics of PLL degradation induced by radiation of various types. Vargas et al. [16] reported data characterizing the radiation-induced degradation of the polymer during electron-beam irradiation of polylactic acid plastic drinking cups and compared the values of the radiation-chemical yield of chain scission (G_s) with their previously published results on the γ-irradiation of this plastic [15].

The main reason for the lack of reliable comparative data in the literature on the PLL degradation induced by treating with various types of radiation is apparently due to unresolved problems that complicate the interpretation of the results of radiation processing of polymers using high-energy electron beams. These problems include the uneven distribution of absorbed dose (D) through the irradiated polymer bulk [26] and insufficiently high accuracy of determining its absolute value. The latter circumstance is due to the fact that even knowingly high doses are often measured using industrial dosimetric films that are certified for a range of values whose upper boundary (200–300 kGy) is lower than the values of D to be determined. Possible uncontrolled heating of PLL samples during irradiation is also a problem [20], which can lead to the transition of the polymer from one relaxation (and even phase) state to another, thereby completely ruling out the correct comparison of the effects of various types of ionizing radiation on radiation-induced degradation of the polymer.

The purpose of this work was to obtain comparative data characterizing the radiation degradation processes in PLL during gamma- and electron beam irradiation in the presence of atmospheric oxygen.

EXPERIMENTAL

We used industrial polylactide resin of the 4043D brand manufactured by NatureWorks LLC (USA), in which the concentrations of L- and D-units were 94.8 and 5.2 mol %, respectively. The samples under study were ellipsoidal pellets with linear dimensions along the major and minor axes of 4.5–4.9 and 2.5–2.8 mm, respectively; the average mass of one pellet was 0.17 g.

Polylactide samples were irradiated in 6 × 8 cm polyethylene bags equipped with Zip-Lock fasteners. The portion of the polymer placed in the bag was 7 g, which made it possible to arrange the pellets in one layer with the thickness corresponding to their minimum linear size.

The samples were γ-irradiated at room temperature on a UGU-420 cobalt facility (420 kCi activity). The dose rate was 1.4 Gy/s. The absorbed dose values 100, 250, 500, 700 and 1000 kGy) were set by the duration of irradiation.

The source of accelerated electrons was a UELV-10-10 multipurpose linear accelerator. The electron beam energy was 6.5 MeV with a total electron beam current of 850 μA. Irradiation was carried out at room temperature under conditions of fifteen-fold forced ventilation in the working chamber of the accelerator. Bags with polymer pellets were placed in one layer on the bottom of a 45 × 70 × 10 cm pallet made of 2 mm thick aluminum sheet. The pallet was installed across the direction of movement of the conveyor belt. The speed of the pallet under the beam was 0.57 cm/s. Thermocouples were used to monitor the polymer temperature; standard SO PD(F)P 30/200 films (Russia), certified for measurements in the fields of electron radiation in the range of D values from 30 to 200 kGy, were used for dosimetry, as well as Fujifilm FTR-125 dosimetric films (Japan), designed for measurements of D from 5 to 300 kGy. The difference in D values determined using SO PD(F)P 30/200 and Fujifilm FTR-125 dosimetric films did not exceed 5%.

The minimum dose of 120 kGy was absorbed in one irradiation cycle. It consists of rapid (4 cm/s) move of the pallet from loading to irradiation zone, stop, absorption of a dose for three slow (0.57 cm/s) passes (forward–backward–forward) under the beam, stop, and rapid (4 cm/s) return of the pallet from irradiation to loading zone. Stopping the pallet before receiving the dose needed for switching the conveyor speed (the duration of stop did not exceed 10 s). The stops before the reversing motion were made when the pallet was completely removed from the beam coverage area. After completion of the cycle, the pallet was taken out of the irradiation zone for 15 min to measure the temperature and cool, after which it was again sent for irradiation. Values of D ranged from 120 to 1200 kGy. Doses of 360, 600, 840, and 1200 kGy were accumulated for 3, 5, 7, and 10 irradiation cycles, respectively. The total time spent on the irradiation procedure to have the maximum dose was 3 h.

The distribution of D through the thickness of the irradiated polymer, as well as along the direction of movement of the conveyor and along the electron beam sweep, was determined experimentally using the dosimetric films. The films were placed between layers of PLL pellets poured into a box of 10 × 10 × 10 cm in size in the first case and under the plastic bags with PLL pellets in the second and third cases.

The molecular structure characteristics of the initial and irradiated PLL polymer, by which the efficiency of its radiation-induced degradation was assessed, were experimentally determined values of average molecular mass and polydispersity index.

The values of the viscosity-average molecular mass $\langle M_v \rangle$ of the polymer were determined by capillary viscometry using the characteristic viscosity [η] of polymer solutions in chloroform with a concentration of 0.5–2.0 g/dl. The measurements were made at 30°C with an Ostwald viscometer (capillary diameter of

0.56 mm). The $\langle M_v \rangle$ values of the samples were calculated according to the Mark–Kuhn–Hauwink–Sakurada equation (1):

$$[\eta] = K \times \langle M_v \rangle^{\alpha}, \qquad (1)$$

where K and α are constants equal to 1.31×10^{-4} dl/g and 0.777, respectively [27].

The number-average molecular mass ($\langle M_n \rangle$) of PLL samples was found according to Eq. (2) [28] using experimentally determined values of the viscosity-average molecular mass $\langle M_v \rangle$:

$$\langle M_v \rangle = \langle M_n \rangle [(1+\ \alpha)\Gamma(1+\alpha)]^{1/\alpha}, \qquad (2)$$

where α is the constant of Eq. (1) and Γ is the gamma function.

In addition, for unirradiated PLL and PLL irradiated with the minimal (100–120 kGy) and maximal (1000–1200 kGy) doses, we used gel permeation chromatography (**GPC**) to find number-average molecular mass $\left\langle M_n^{\mathrm{GPC}} \right\rangle$, weight-average molecular mass $\langle M_w \rangle$, and polydispersity index (P) defined as $\langle M_w \rangle / \left\langle M_n^{\mathrm{GPC}} \right\rangle$. The instrument used was a Thermo-Scientific Ultimate 3000 chromatograph with a refractometric detector, an Agilent PLgel 5 μm MIXED-C column, and a PLgel 5 μm guard column thermostated at 30°C; the mobile phase was tetrahydrofuran (**THF**) passed at a flow rate of 1 mL/min. Samples to be investigated were solutions of PLL (2 mg/ml) in THF. Calibration was carried out using a set of polystyrene standards with molecular masses of 580, 3070, 6930, 29 400, and 188 700 g/mol, the P value for which did not exceed 1.12.

The G_s values were calculated according to Eq. (3) [15–17, 19]:

$$G_s = N_A \left(\langle M_{n,D} \rangle^{-1} - \langle M_{n,0} \rangle^{-1} \right) / (6.24 \times 10^{16} \times D), \qquad (3)$$

where $\langle M_{n,\mathrm{D}} \rangle$ is the value of $\langle M_n \rangle$ or $\left\langle M_n^{\mathrm{GPC}} \right\rangle$ for PLL irradiated with a dose of D (kGy), $\langle M_{n,0} \rangle$ is the value of $\langle M_n \rangle$ or $\left\langle M_n^{\mathrm{GPC}} \right\rangle$ for unirradiated PLL, and N_{A} is the Avogadro number.

The glass transition temperature of the amorphous phase of PLL (T_g) and the melting temperatures of its crystallites (T_{m1} and T_{m2}) in two known crystalline forms (disordered and ordered) [1] were determined from thermograms recorded in a nitrogen stream with a STA 449C thermal analyzer in the differential scanning calorimetry/thermogravimetry mode as in [22].

RESULTS AND DISCUSSION

The specific problems arising in the electron beam processing of PLL were solved using the cyclic irradiation regime. This technique was used in [8, 20]. The use of dosimetric films certified for measurements in the D range from 5 to 300 kGy in each irradiation cycle made it possible to reliably determine the absolute values of sufficiently high D (360, 600, 840, and 1200 kGy).

Table 1. Glass transition and melting temperatures of electron beam irradiated PLL

D, kGy	T_{g}, °C	T_{m1}, °C	T_{m2}, °C
0	58.5 ± 0.5	148.7 ± 0.1	151.6 ± 0.1
120	57.5 ± 0.5	146.4 ± 0.9	151.5 ± 0.2
360	56.0 ± 0.5	134.3 ± 0.6	144.0 ± 0.1
600	54.5 ± 0.5	135.3 ± 0.1	139.5 ± 0.1
840	52.0 ± 0.5	125.4 ± 0.1	131.4 ± 0.2
1200	51.5 ± 0.5	113.8 ± 0.1	122.9 ± 0.1

One should take into account that PLL samples during irradiation with a high-energy electron beam may get heated. The irradiation procedure used enabled us to avoid PLL samples heating to temperatures exceeding the glass transition of the amorphous phase and the melting points of the crystalline phase of the polymer. It was found that during electron beam irradiation, PLL pellets had not been heated above 45°C. This temperature, according to the simultaneous thermal analysis data (Table 1), was lower than the glass transition temperature and, moreover, the melting temperature of both unirradiated PLL and PLL irradiated up to the maximum dose of 1200 kGy. Consequently, during the irradiation, the amorphous phase of the polymer remained in the glassy relaxation state and the crystalline phase remained in its two (disordered and ordered) forms. Thus, the experimental data, obtained in this study, characterizing the processes of PLL degradation under gamma- and electron beam irradiation refer to the irradiated polymer, the relaxation and phase state of which remains essentially the same as that of the unirradiated polymer.

Figure 1 shows the profile of dose distribution through the thickness of the fill of irradiated pellets. In the case of irradiation of PLL pellets arranged in five layers, the ratio of maximum to minimum D absorbed by the sample was 1.39. For the pellets packed in one layer (2.5–2.8 mm), this ratio decreased to 1.15, which is an acceptable level of absorbed dose uniformity ratio; thus, PLL granules were placed in one layer in plastic bags in all the subsequent experiments, both with electron beam and γ-irradiation.

The use of dosimetric films showed that the distribution of the dose D along the direction of conveyor movement and along the electron beam sweep was uniform. Such a result was achieved in the case of irradiation on the moving conveyor with a uniform pulling speed and invariability of the established irradiation mode, which ensures uniform electron beam current density along the scanning strip. Since the uniform distribution of D along the length and width of the

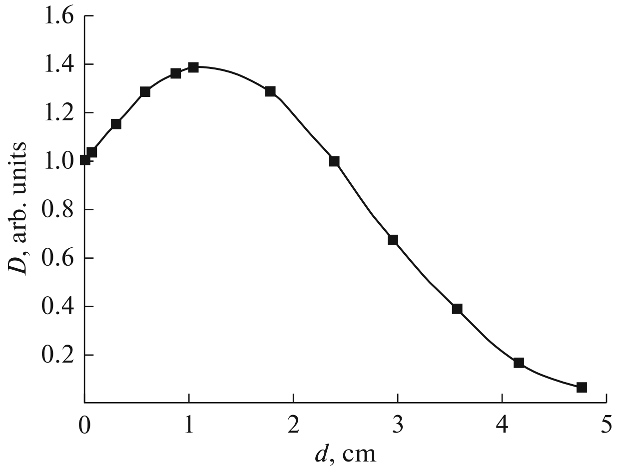

Fig. 1. Dose distribution profile through the thickness of the bulk of electron beam-irradiated PLL pellets.

irradiated bags with PLL samples is supposed to be uniform, it can be assumed that the polymer sample was uniformly irradiated through the entire volume.

Thus, the values of $\langle M_n \rangle$, $\left\langle M_n^{\text{GPC}} \right\rangle$, $\langle M_w \rangle$, P, and G_s were determined for the polymer irradiated under conditions allowing for maximally comparable data to be obtained to characterize the degradation of PLL macromolecules subjected to different types of ionizing radiation.

A comparative assessment of the efficiency of radiation degradation of macromolecules of this PLL plastic treated with γ-radiation and electron beam was made by comparing the values of their average molecular masses and the polydispersity index before and after irradiation, as well as the G_s values calculated from them.

The experimental data presented in Table 2 show that unirradiated PLL and the PLL after γ-irradiation ($D = 100$ and 1000 kGy) or electron beam treatment ($D = 120$ and 1200 kGy) do not differ much in polydispersity index ($P = 2.15 \pm 0.30$ at a confidence level of 0.95). Provided that the polydispersity index of the polymer is close to 2 (Table 2), it is possible to calculate $\langle M_n \rangle$ from the $\langle M_v \rangle$ values found by capillary viscometry using Eq. (2).

Table 2. Effect of ionizing radiation type and absorbed dose on the GPC-determined $\left\langle M_n^{\text{GPC}} \right\rangle$, $\langle M_w \rangle$, and P values for PLL

Radiation type*	D, kGy	$\left\langle M_n^{\text{GPC}} \right\rangle$, g/mol	$\langle M_w \rangle$, g/mol	P
–	0	96700	181200	1.87
γ	100	23400	53500	2.29
ē	120	24500	56200	2.29
γ	1000	3700	6700	1.89
ē	1200	5200	12600	2.42

* γ and ē denote γ-rays and electron beam, respectively.

The data given in Table 3 show that the $\langle M_v \rangle$ values and the $\langle M_n \rangle$ values calculated from the former are lower in the case of γ-irradiation than electron beam irradiation over the entire investigated range of D from 120 to 1200 kGy, thereby definitely indicating a higher efficiency of degradation of PLL by irradiation with γ-rays.

One of the most important characteristics of radiation-induced degradation of macromolecules, along with the G_s value, is the radiation-chemical yield of crosslinking (G_x), which can be calculated with the proviso that the quantities $\{[\langle M_{n,\,\text{D}} \rangle]^{-1} - [\langle M_{n,\,0} \rangle]^{-1}\}$ and $\{[\langle M_{w,\,\text{D}} \rangle]^{-1} - [\langle M_{w,\,0} \rangle]^{-1}\}$ linearly depend on D [29, 30]. Published data on the G_s and G_x values for PLL differ markedly depending on the characteristics of the virgin polymer and irradiation conditions; however, G_s is significantly higher than G_x in all cases [5, 13, 18, 23], thereby definitely showing the predominance of chain scission over crosslinking. Therefore, for a comparative assessment of the effect of the type of ionizing radiation on the degradation of PLL macromolecules, to a first approximation, it is entirely possible to limit ourselves to comparison of the G_s values only, especially taking into account the fact that this approach is often used to study the radiation-induced degradation of this polymer [15–17, 19].

It was found that in the D range of 100 to 1200 kGy, the dependence of $\{[\langle M_{n,\,\text{D}} \rangle]^{-1} - [\langle M_{n,\,0} \rangle]^{-1}\}$ on D is linear (Fig. 2), thereby allowing the calculation of G_s values for PLL by Eq. (3). The values of G_s in the case of electron beam irradiation amounted to 2.20 versus 2.86 for γ-irradiation, which is consistent with the data given in [15–17, 19].

The higher G_s values in the case of γ-irradiated PLL samples are apparently due to a significantly longer time of their radiation treatment in air. Indeed, a γ-radiation dose of 1000 kGy was attained within about 192 h, whereas the electron beam irradiation of the polymer to have the same dose took less than 1.5 h. As a result, the contribution of oxidative degradation of PLL during γ-irradiation in air is significantly higher. A similar trend was noted in [16]: the value of G_s was 0.52 in the case of electron beam irradiation [16] versus 2.18 upon γ-irradiation [15].

CONCLUSIONS

In the range of absorbed doses from 100–120 to 1000–1200 kGy, a comparative study has been carried out on the degradation of macromolecules of brand 4043D PLL, manufactured by NatureWorks LLC, in polymer pellets under irradiation with γ-rays and a beam of fast electrons in air at room temperature. The

Table 3. Effect of ionizing radiation type and absorbed dose on the viscosity-average molecular mass $\langle M_v \rangle$ and the number-average molecular mass $\langle M_n \rangle$ calculated according to Eq. (2)

Radiation type*	D, kGy	$\langle M_v \rangle$, g/mol	$\langle M_n \rangle$, g/mol
–	0	146000	91000
γ	120	31600**	19800
ē		32000	21000
γ	360	13300**	8200
ē		18000	11000
γ	600	8400**	5200
ē		12000	7500
γ	840	6100**	3800
ē		9300	5600
γ	1200	4400**	2700
ē		5300	3300

* γ and ē denote γ-rays and electron beam, respectively;
** obtained from the linear relationship of $1/\langle M_v \rangle$ with D.

use of the cyclic regime of electron beam irradiation ensured the uniform distribution of the absorbed dose over the volume of the irradiated polymer, the high accuracy of determining its absolute value, and the same invariance of the PLL phase and relaxation state as in the case of γ-irradiation. A comparison has been made of the changes in the molecular mass characteristics of PLL and the calculated radiation-chemical yields of the main chain scission (G_s) in the polymer by ionizing radiation of two types, which are most often used for radiation processing of medical devices and biodegradable food packaging. The values of G_s upon γ-irradiation of PLL and its electron beam processing are found to be 2.86 and 2.20, respectively.

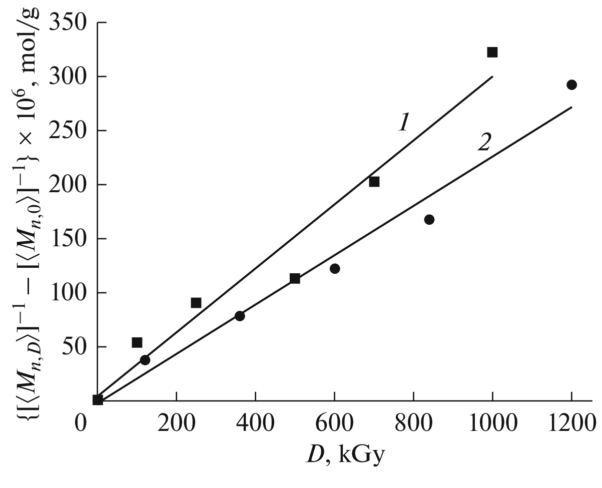

Fig. 2. Plot of $\{[\langle M_{n,\,D}\rangle]^{-1} - [\langle M_{n,\,0}\rangle]^{-1}\}$ as a function of dose (D) for PLL in the cases of (*1*) gamma- and (*2*) electron beam irradiation.

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