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RESEARCH ARTICLE

Structure, properties, and photodegradation of polymer biocomposites based on polylactide filled with A-Len® thermoplastic compound

Ilya Yu. Vasilyev[1,✉], **Victoria S. Petrenko**[1], **Viktor A. Rod**[1], **Dmitrii A. Golobokov**[2]
[1] *Moscow Polytechnic University (Mospolitech), Moscow, 107023 Russia*
[2] *Enikolopov Institute of Synthetic Polymeric Materials, Russian Academy of Sciences, Moscow, 117393 Russia*
✉ *Corresponding author, e-mail: iljanaras@ya.ru*

Abstract

Objectives. The work set out to investigate the influence of the compounding and technological parameters of polymer biocomposite materials based on polylactide (PLA) filled with the A-Len® thermoplastic compound (Alen) on their structural, thermophysical, and mechanical properties, as well as to determine the mechanism of their destruction under the impact of ultraviolet radiation.

Methods. Films of PLA and Alen, as well as polymer composites based on them (PLA/Alen), were obtained using a flat die extruder (*MashPlast*, Russia). The structural and morphological properties of the films were determined with a JCM-6000 PLUS scanning electron microscope (*JEOL*, Akishima, Tokyo, Japan); their thermal properties were obtained using a DSC 204 F1 calorimeter (*NETZSCH*, Germany). The chemical composition and degradation mechanism were analyzed by infrared spectroscopy with an FSM-1201 spectrometer (Russia) fitted with an attenuated total internal reflection attachment. The mechanical properties were examined using a Z010 ZwickRoell tensile testing machine (*Zwick Roell Group*, Germany). The effect of ultraviolet radiation was assessed using an ultraviolet chamber.

Results. It was shown that the presence of Alen in mixtures with PLA affects the formation of a heterogeneous structure due to the presence of polyethylene in the Alen composition. This mediated a reduction in Young's modulus by 34% (to 1050 MPa) in comparison with the initial value for PLA (1600 MPa). In the polymer composites, the PLA phase recrystallizes to yield a denser-ordered supramolecular structure, while in the polyethylene phase, the segmental mobility of macromolecules decreases and crystallization process is hindered by their different polarities to form an interface between the components under study.

Conclusions. The photodegradation mechanism in PLA/Alen polymer composites is shown to be primarily due to the presence of a pro-oxidant additive in Alen, which initiates the oxidation process to degrade and subsequently fragment the low-molecular fraction of polyethylene. In addition, the PLA matrix photodegrades through the Norrish Type II mechanism, which describes the oxidation of polymer chains by a radical mechanism to produce C=C bonds. The developed polymer composites are promising for the fabrication of certain polymer products due to their optimal performance characteristics and accelerated photochemical degradation kinetics.

Keywords
polylactide, A-Len®, compound, biocomposites, UV radiation, photodegradation

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НАУЧНАЯ СТАТЬЯ

Структура, свойства и фотодеструкция полимерных биокомпозиционных материалов на основе полилактида, наполненного термопластичным компаундом A-Len®

И.Ю. Васильев[1],✉, В.С. Петренко[1], В.А. Род[1], Д.А. Голобоков[2]

[1] *Московский политехнический университет (Мосполитех), Москва, 107023 Россия*

[2] *Институт синтетических полимерных материалов им. Н.С. Ениколопова Российской академии наук, Москва, 117393 Россия*

✉ *Автор для переписки, e-mail: iljanaras@ya.ru*

Аннотация

Цели. Изучить влияние рецептурно-технологических параметров полимерных биокомпозиционных материалов на основе полилактида (ПЛА), наполненного термопластичным компаундом A-Len® (Alen), на структурные, теплофизические и деформационно-прочностные свойства, а также определить механизм деструкции под действием ультрафиолетового излучения.

Методы. В работе исследовали пленки на основе ПЛА и Alen, а также полимерные композиции на основе ПЛА и Alen (ПЛА/Alen), которые получали на плоскощелевом экструдере фирмы «*МашПласт*» (Россия). Для вышеуказанных объектов определяли структурно-морфологические свойства на сканирующем электронном микроскопе JCM-6000 PLUS (*JEOL*, Акисима, Токио, Япония), теплофизические свойства на калориметре DSC 204 F1 (*NETZSCH*, Германия). Химический состав, а также определение механизма деструкции осуществляли методом инфракрасной спектроскопии на приборе ФСМ-1201 (Россия) с приставкой нарушенного полного внутреннего отражения. Деформационно-прочностные свойства исследовали на разрывной машине Z010 ZwickRoell (Германия). Воздействие ультрафиолетового излучения определяли с помощью ультрафиолетовой камеры.

Результаты. Показано, что Alen в смесях с ПЛА влияет на образование гетерогенной структуры вследствие наличия в его составе полиэтилена (ПЭ). Этим опосредовано снижение модуля упругости на 34% (1050 МПа) в сравнении с исходной величиной ПЛА — 1600 МПа. При этом для фазы ПЛА в полимерных композициях осуществляется перекристаллизация с более плотным упорядочиванием надмолекулярной структуры, а для фазы ПЭ происходит снижение сегментальной подвижности макромолекул и затруднение процесса кристаллизации вследствие их разной полярности с формированием границы раздела фаз между исследуемыми компонентами.

Выводы. Механизм фотодеструкции полимерных композиций ПЛА/Alen осуществляется за счет наличия в составе термопластичного компаунда прооксидантной добавки, инициирующей процесс окисления, после чего происходит разрушение низкомолекулярной фракции ПЭ с последующей ее фрагментацией. Наряду с этим, фотодеструкция матрицы ПЛА протекает по механизму Норриша II, заключающемся в окислении полимерных цепей по радикальному типу с образованием С=С-связи. Таким образом, разработанные биокомпозиционные полимерные материалы являются перспективными для изготовления полимерных изделий из них, характеризуются оптимальными эксплуатационными свойствами и ускоренной кинетикой фотохимической деструкции.

Ключевые слова

полилактид, A-Len®, компаунд, биокомпозиты, УФ-излучение, фотодеструкция

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INTRODUCTION

The growing relevance of using biodegradable polymers is driven by the possibility of creating promising, environmentally friendly materials to reduce environmental impacts for use in the packaging, medical, and agricultural industries [1]. The leading position of synthetic thermoplastics in the packaging industry is accompanied by an uncontrolled amount of polymer waste, most of which accumulates in landfills to pollute the environment. However, in many cases, polymers synthesized from petrochemical feedstocks [2–4] can be made biodegradable by modifying them with natural fillers.

The modification of synthetic polyolefins with natural polymers accelerates the fragmentation of the polymer matrix due to the rapid biodegradation of the filler. Such modifying additives include starch [5], chitin [6], chitosan [7], and other natural polymers [8–10]. However, the formation of such composites can be complicated by insufficient adhesion of the synthetic matrix to the natural filler. An additional complicating factor is the limited filler concentration (typically up to 30 wt %), which is due to the technological complexity of producing dispersed filled systems. Increasing the natural polymer content in the synthetic polymer matrix above 30 wt % gives rise to a large number of agglomerates, which may lead to defects in the production of such composites [11–13].

A promising direction in recent years involves the creation of biodegradable materials based on polymers synthesized from renewable raw materials, in particular polylactide (PLA), which reduces the dependence on petroleum-derived polymers [14–16]. PLA is a synthetic polymer made from the lactide monomer, which can exist in two optically active isomeric forms, L- and D-lactide, as well as in the optically inactive form of meso-lactide. PLA is a strong and rigid polymer comparable in performance properties to polyethylene terephthalate and, following appropriate plasticization, to polyethylene (PE) and polyvinyl chloride [17]. PLA can be processed both by extrusion to produce flexible films and by thermoforming to produce rigid containers [18]. Although PLA is a biodegradable polymer, it is important to note that its biodegradation only occurs under certain conditions (high temperature, pH, and humidity). Therefore, PLA, like other polymers, is typically modified with additives of various origins [19–21], although the desired properties are not always achievable due to of their low technological compatibility [22].

Another method for modifying synthetic thermoplastics is the use of pro-oxidant additives, e.g., transition metal (Mn, Fe, Mg) stearates, which initiate the decomposition of the polymer matrix and promote accelerated oxidative degradation [23]. The main advantage of using transition metal stearates consists in their ability to modify a wide range of thermoplastic polymers, including PLA, as well as their processing on existing equipment designed for thermoplastics, their low toxicity, and—unlike natural fillers—the short oxidative degradation time of polymer materials produced at a low mass fraction within the synthetic polymer [24].

Therefore, it is of scientific and practical interest to analyze the properties of ternary composites based on PLA filled with the thermoplastic compound A-Len® (Alen) and determine their degradation mechanism, which is currently insufficiently understood.

EXPERIMENTAL

Materials

The polymer matrix for the production of polymer composites was made of the aliphatic polyester PLA grade 524 (China). The additive used to initiate the degradation of the polymer matrix was the thermoplastic compound A-Len® TECH BD00TY (India) with an active (organic) substance content of 10 wt %. Table 1 presents the characteristics of the initial components.

Research methods

The components were mixed, plasticized, and subsequently cast using a flat die extruder (*MashPlast*, Russia) at a varying Alen content in mixtures with PLA from 5 to 20 wt %. The resulting polymer composites were films having a thickness of 60 ± 3 μm. Table 2 presents the temperature conditions to produce the initial PLA and Alen films, as well as the polymer composites based on them. Table 3 shows the compositions of the polymer composites.

The effect of ultraviolet (UV) radiation on the samples was determined using an UV chamber containing two PRK-4 quartz (mercury) lamps producing radiation with a wavelength of $\lambda = 254$ nm. 150 × 150 mm film samples were placed 30 cm from the UV lamps, isolated from any external radiation source, and exposed for 150 h with collection of control samples every 25 h.

The thermophysical properties of the samples were determined using differential scanning calorimetry on a DSC 204 F1 calorimeter (*NETZSCH*, Germany) in a temperature range from 20 to 200°C at a scanning rate of 10°C/min and a sample weight of 10 ± 1 mg under a flow of inert argon gas. When studying the thermophysical properties, the enthalpy of melting ΔH_m (J/g), melting point T_m (°C), and glass transition temperature T_g (°C) were determined. The degrees of crystallinity α (%) of Alen and PLA, as well as polymer composites based on

Table 1. Characteristics of the initial components

Component	Melt flow index, g/10 min	Young's modulus, MPa	Elongation at break, %	Density, g/cm^3
PLA	8.7	1600	4.2	1.24
Alen	17	600	3.2	0.97

Table 2. Temperature conditions to produce polymer composites

Polymer composites	Temperature conditions of flat die extruder zones, °C				
	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5
PLA	165	168	170	173	175
Alen	120	125	130	135	140
PLA/Alen	165	168	170	173	175

Table 3. Compositions of polymer composites

Polymer composites	Concentration, wt %	
	PLA	Alen
PLA	100	–
Alen	–	100
PLA/Alen5	95	5
PLA/Alen10	90	10
PLA/Alen15	85	15
PLA/Alen20	80	20

them, were found using the enthalpies of melting of fully crystalline PE (293 J/g) and for PLA (93.7 J/g) according to ISO 11357-1:2023 "Plastics. Differential scanning calorimetry (DSC). General principles"[1].

The chemical composition and photodegradation mechanism were determined by FTIR spectroscopy with an attenuated total internal reflection (ATR) attachment using an FSM-1201 spectrometer (Russia) with a resolution of 1.0 cm^{-1} (spectral wavenumber range of 4000–375 cm^{-1}) at a temperature of 23 ± 2°C according to GOST 57939-2017[2].

Performance properties were measured in accordance with ISO 527-3:2018 "Plastics. Determination of tensile properties"[3]. Tests were conducted on a ZwickRoell Z010 tensile testing machine (*ZwickRoell Group*, Germany). Specimens for testing strength and deformation properties were obtained using a type 1B punch (EN ISO 527-3:2018) with dimensions of 50 × 10 mm. The maximum permissible error of load measurements did not exceed 1%. The maximum deviation in specimen width was ±0.1 mm. The average value was determined from 10 measurements. Tests were carried out at a sample deformation rate of 50 mm/min. The strength properties were measured as Young's modulus (E) and tensile stress at break (σ), while the deformation properties were determined as elongation at break (ε).

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[1] ISO 11357-1:2023. Plastics — Differential scanning calorimetry (DSC). Part 1: General principles. https://www.iso.org/standard/83904.html. Accessed January 22, 2026.

[2] GOST 57939-2017. National Standard of the Russian Federation. Polymer composites. Infrared spectroscopy. General principles. Moscow: Standartinform; 2019.

[3] ISO 527-3:2018. Plastics — Determination of tensile properties. Part 3: Test conditions for films and sheets. https://www.iso.org/standard/70307.html. Accessed January 22, 2026.

The structural and morphological properties of gold-sputtered samples were examined using scanning electron microscopy with a JCM-6000 PLUS microscope (*JEOL*, Akishima, Tokyo, Japan) with a secondary electron detector at accelerating voltages of 10 and 15 kV.

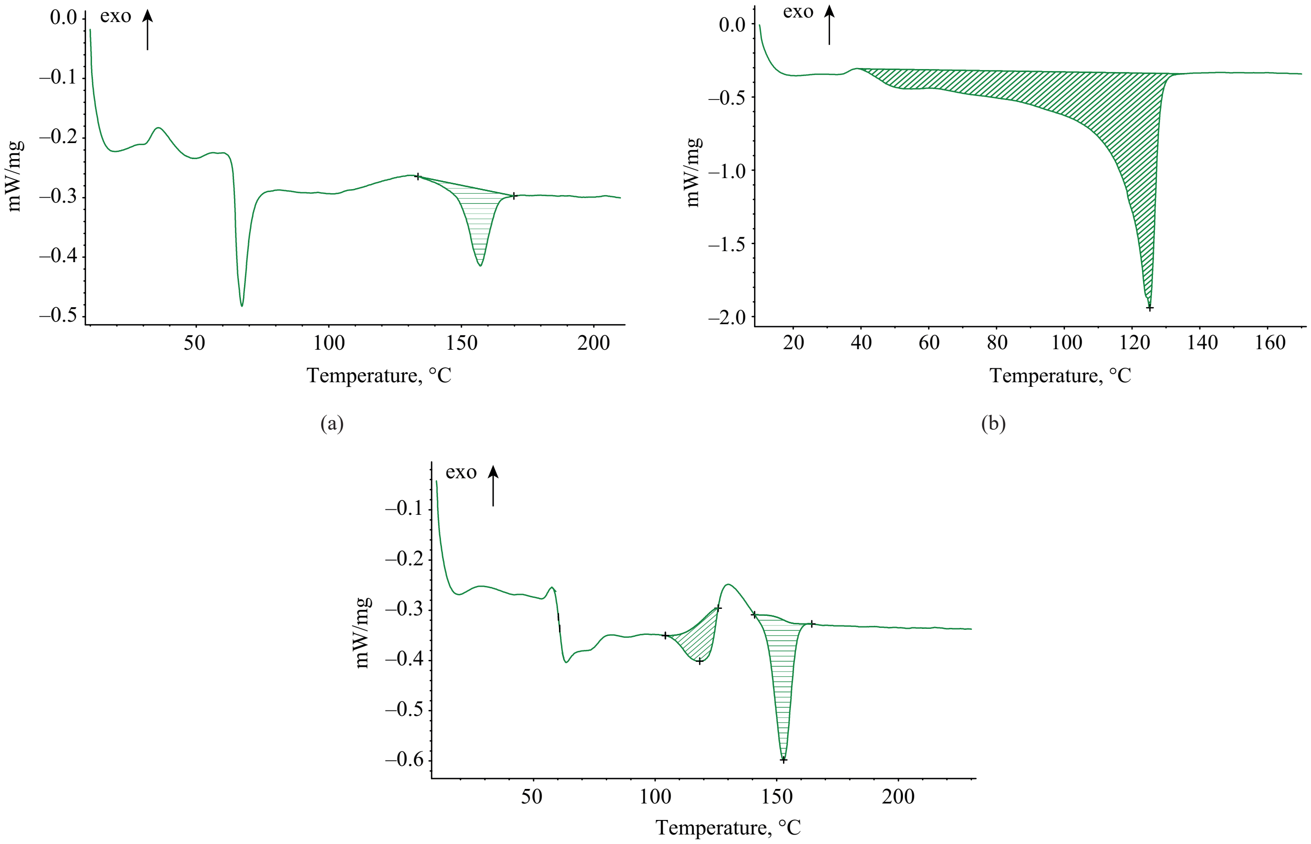
RESULTS AND DISCUSSION

Thermophysical characteristics

The thermal analysis curve of PLA (Fig. 1a) contains two peaks: the first at 65°C corresponds to the glass transition temperature, while the second identifies the presence of a crystalline phase at a melting point of 157°C. Figure 1b depicts a characteristic endothermic melting peak at 125°C corresponding to the melting point of PE, which is part of the thermoplastic compound. The thermal analysis curve of the PLA/Alen polymer composite containing 20 wt % Alen (Fig. 1c) contains two endothermic peaks corresponding to the melting points of PE at 118°C and PLA at 153°C.

The addition of Alen to PLA at a content of 20 wt % causes an increase in the enthalpy of melting and degree of crystallinity of the PLA phase (Table 4). This occurs as a result of recrystallization, which causes a denser ordering of the PLA supramolecular structure due to the presence of the crystalline PE phase in the polymer composite. The shift of the endothermic peak of PE melting to a lower temperature of 118°C indicates the formation of more defective small crystals, which is caused by a decrease in the segmental mobility of PE macromolecules which hinders the crystallization process.

Following 150 h of exposure to UV radiation, the thermophysical properties of films based on the initial PLA and Alen components, as well as the PLA/Alen polymer composites, are observed to change (Fig. 2). For the irradiated PLA-based film, in comparison with the initial, the enthalpy of melting increases by more than 3.5 times and a cold crystallization peak appears on the thermal analysis curve at 105°C. At the same time, the melting point decreases by 5.2°C. The increase in the enthalpy of melting under UV irradiation is due to the partial degradation of the amorphous phase of PLA, which leads to a change in the supramolecular structure of the crystalline phase, namely, the formation of denser and larger crystals. Another explanation is that the presence of a metastable crystalline structure of PLA under UV irradiation is likely to promote continued crystallization by breaking stressed tie molecules, thereby increasing its degree of crystallinity [25].



(a)

(b)

(c)

Fig. 1. Thermal analysis curves of melting of (a) PLA, (b) Alen, and (c) PLA/Alen20

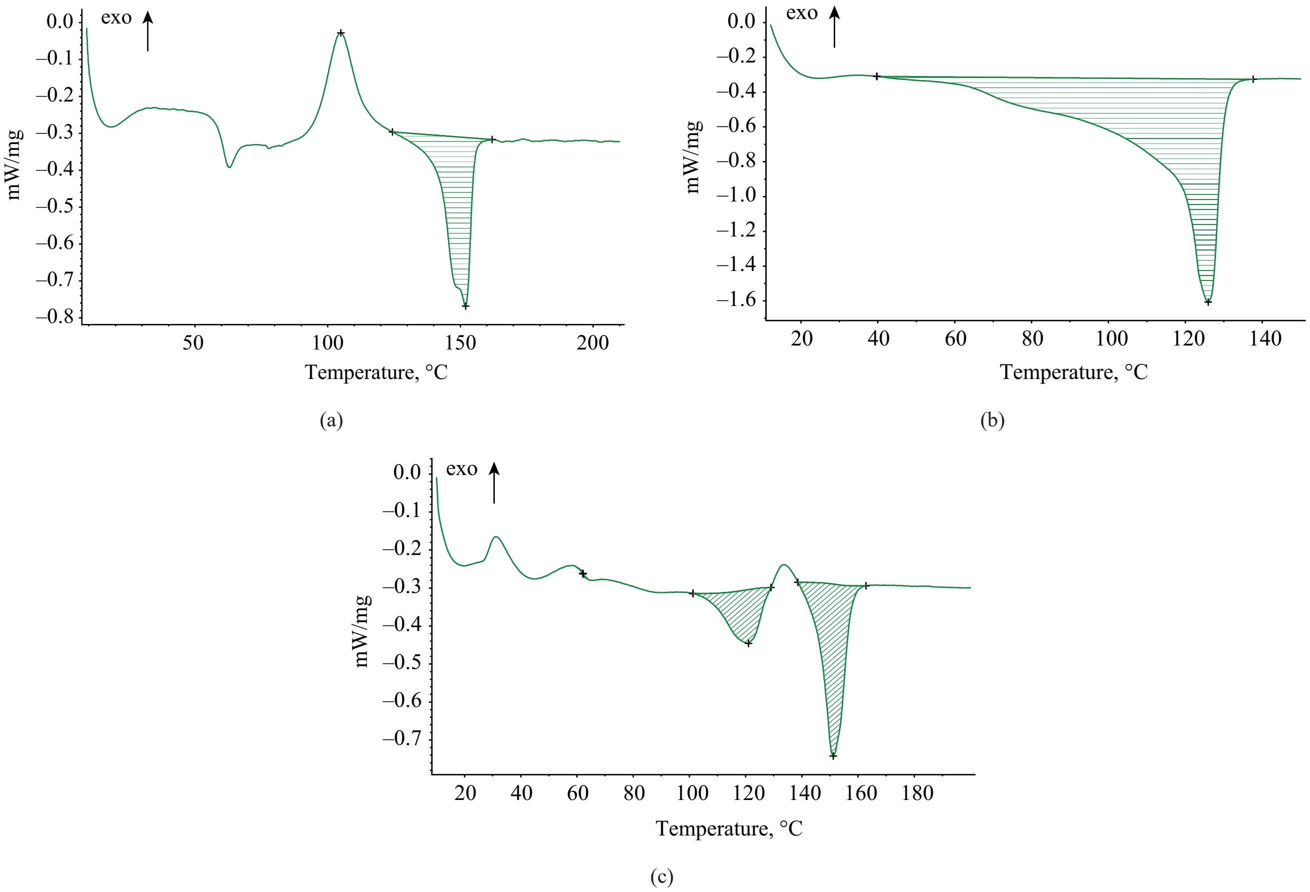


Fig. 2. Thermal analysis curves of melting of (a) PLA, (b) Alen, and (c) PLA/Alen20 following 150 h of UV irradiation

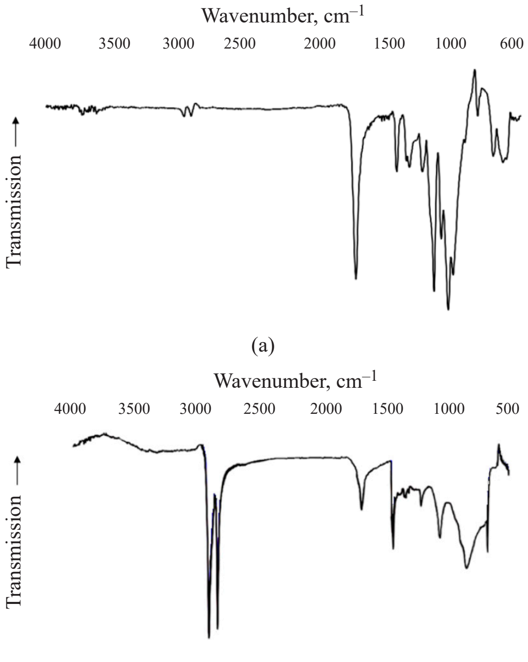
Table 4. Thermophysical properties of polymer composites

Exposure, h	Sample	T_m, °C	ΔH_m, J/g	α, %
0	PLA	157	8.7	9.3
	Alen	125	176	60.0
	PLA/Alen20	153/118	14.5/5.9	15.5/2.0
150	PLA	152	30	32
	Alen	125	155	52.9
	PLA/Alen20	151/121	23.9/12.0	25.5/4.1

For the Alen-based film (Fig. 2b), the enthalpy of melting decreases by 12%. The difference in the melting point before and after UV irradiation does not exceed 1°C. The specific energy of the endothermic process of the PLA/Alen20 polymer composite (Fig. 2c) also increases in comparison with the initial (Table 4). For example, for PE, the enthalpy of melting doubles, while for PLA it increases by 1.7 times. Furthermore, the melting point for the PE phase increases by 3°C, while for the PLA phase it decreases by 1.6°C.

IR spectrum characteristics

The presence of intense absorption bands in the IR spectrum of PLA (Fig. 3a) in the range of 1180–1077 cm^{-1} indicates the presence of rocking vibrations of the methyl CH_3 group, which identifies high-molecular-weight PLA obtained by ring-opening polymerization. Thus, PLA is an optically active poly(D,L-lactide). The IR spectrum of Alen (Fig. 3b) contains absorption bands at 2914 and 2847 cm^{-1}, indicating the presence of an asymmetric CH_2 group and a symmetric CH_2 group, as well as an absorption band at 1462 cm^{-1} characteristic of a scissoring CH_2 group. The IR spectrum also includes a pronounced absorption band near 1740 cm^{-1}; this band is not characteristic of PE, but indicates that Alen includes a second component, which contains a functional carbonyl C=O group.



(a)

(b)

Fig. 3. IR spectra of (a) PLA and (b) Alen

The presence of PE and the carbonyl C=O group constitutes an additive—a masterbatch, which also contains active transition metal elements (Mn, Fe, Mg) initiating the decomposition of the polymer matrix through oxidative degradation [26]. Thus, Alen is a PE composite with a pro-oxidant additive in the form of transition metal stearates.

Apparently, the photodegradation mechanism of PLA/Alen polymer composites is mediated by the pro-oxidant additive in Alen, which then decomposes and fragments the low-molecular-weight PE fraction. The PLA matrix photodegrades by the Norrish Type II mechanism [27], a radical oxidation to form a C=C bond, which appears as an absorption band in the IR spectrum at 1654 cm^{-1} (Fig. 4). The IR spectrum also demonstrates a change in the intensity of the characteristic absorption bands of the PLA/Alen polymer composite, which also confirms the process of photodegradation.

Scanning electron microscopy

The surface of the extruded PLA film (Fig. 5) is characterized by a minimum number of defects, which are presumably related to the manufacturing process. After 150 h of UV irradiation, the surface remained unchanged, indicating no visible signs of photodegradation throughout the entire experiment.

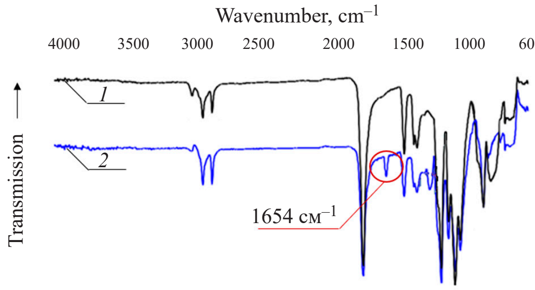
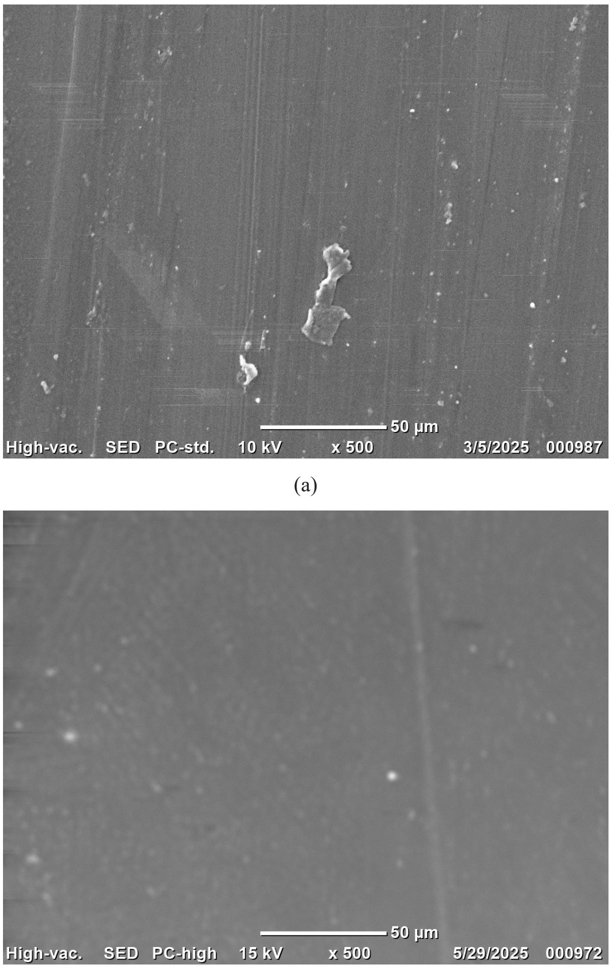


Fig. 4. IR spectra of the PLA/Alen20 polymer composite: (*1*) initial and (*2*) after 150 h of UV irradiation

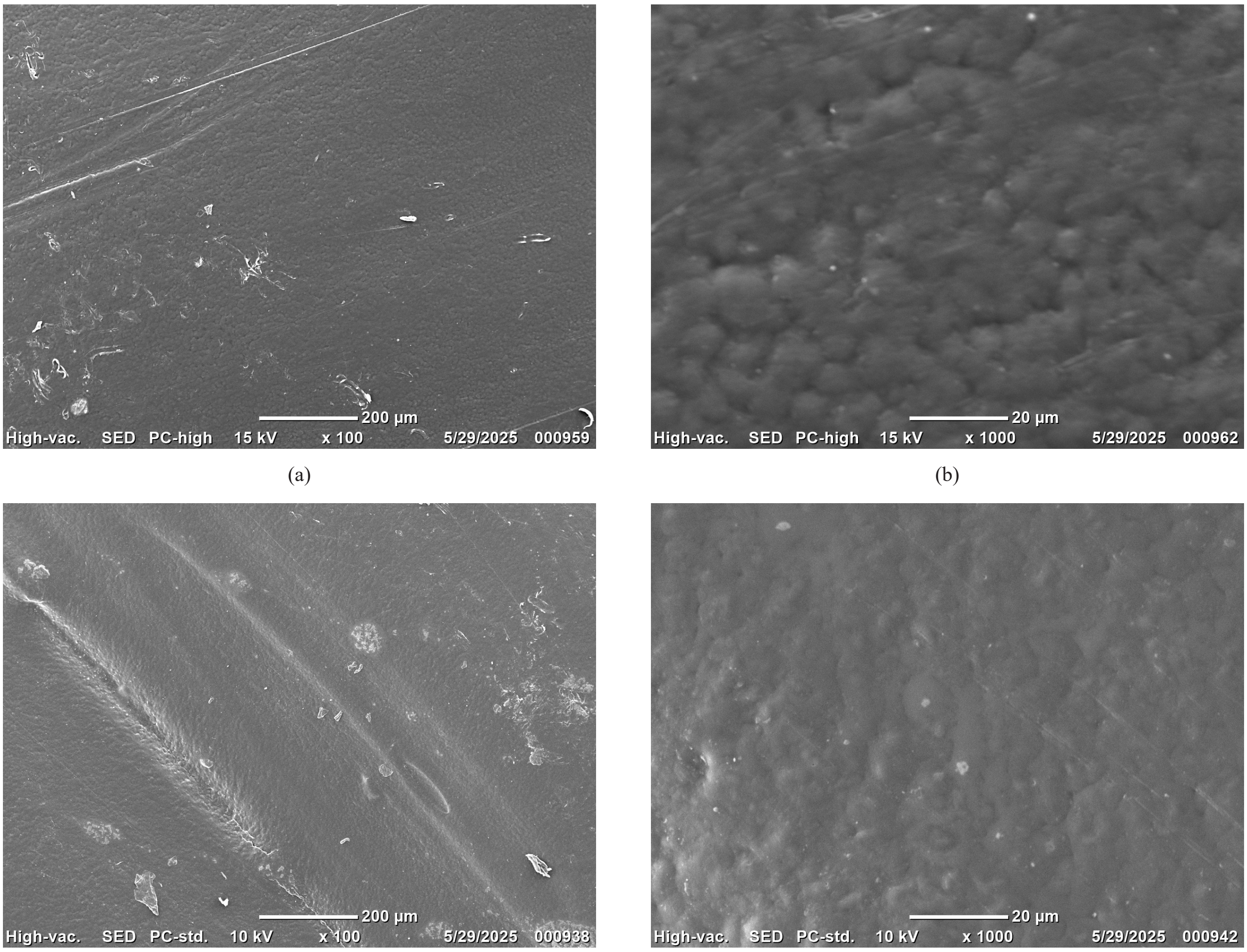


(a)

(b)

Fig. 5. Morphology of the surface of the PLA film: (a) initial, (b) after 150 h of UV irradiation

The surface of the Alen film (Fig. 6a) shows scratches, presumably formed during the extrusion process, and spherulites (Fig. 6b) (average size in the image is ~7 µm). As the result of photodegradation following 150 h of UV exposure, wide through cracks (Fig. 6c) formed on



(a) (b)

(c) (d)

Fig. 6. Surface morphology of the Alen film: (a) and (b) initial, (c) and (d) after 150 h of UV radiation

the film surface, the number of defects increased, and the spherulitic structure became less visible (Fig. 6d).

Figure 7 presents the surface morphology of the PLA/Alen polymer composite. When this composite is produced by extrusion, a structurally heterogeneous film forms (Fig. 7a). Higher magnification reveals domains formed by phase separation of PLA and Alen (Fig. 7b). Following 150 h of exposure in a UV chamber, microcracks form on the surface (Figs. 7c and 7d), indicating photodegradation, most likely of the Alen phase.

Mechanical properties

For the initial PLA and Alen films, the tensile stress at break is 67.2 and 19.2 MPa, respectively, and the Young's modulus is 1600 and 600 MPa. Increasing the Alen content in PLA blends to 20 wt % decreases the tensile stress at break by 53% (to 31.5 MPa), elongation at break by 29% (to 3.0%), and the Young's modulus by 34% (to 1050 MPa) (Fig. 8). This is due to the formation of a heterogeneous structure because of low intermolecular interactions for the reason of the different polarities of the initial components. The elongation at break for the initial PLA film is 4.2%, while for the Alen film it is 3.2%.

Following 150 h of exposure to UV radiation, the tensile stress at break for the PLA film decreases by 67%, the elongation at break by 33%, and the elastic modulus by 50% (to 800 MPa) (Table 5). The mechanical properties of the Alen film cannot be determined because of the presence of a large number of through holes, which also act as stress concentrators. For the PLA/Alen20 polymer composite, the tensile stress at break, like the Young's modulus, decreases by 82% of the initial value, primarily failing at the PLA/PE interface. The elongation at break, which remains virtually unchanged in comparison with the initial value of the PLA/Alen20 polymer composite, amounts to 2.8%.

Thus, the developed polymer biocomposite materials are promising for the manufacture of polymer products due to their optimal performance properties and accelerated photochemical degradation kinetics.

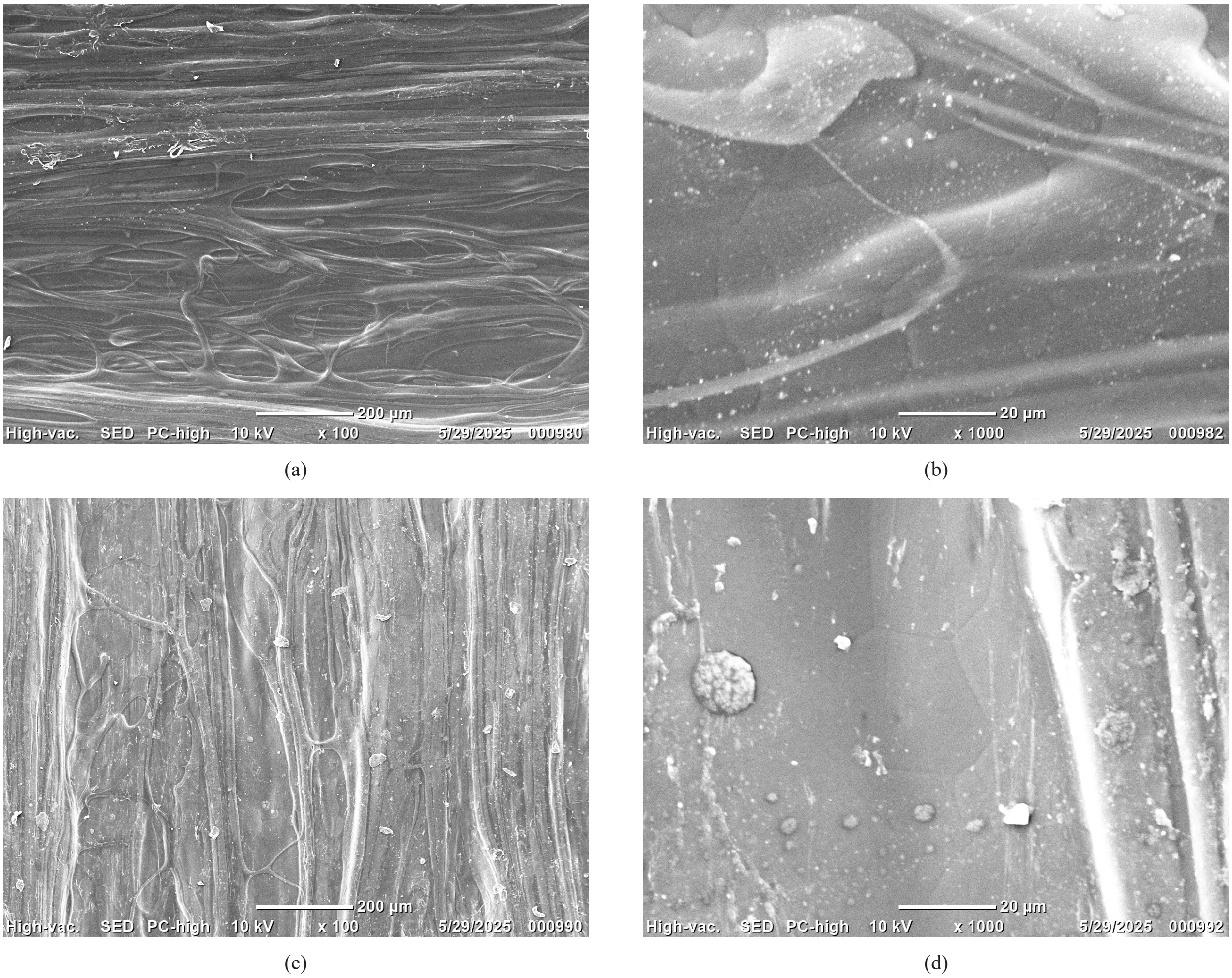


Fig. 7. Surface morphology of the PLA/Alen20 film: (a) and (b) initial, (c) and (d) after 150 h of UV radiation

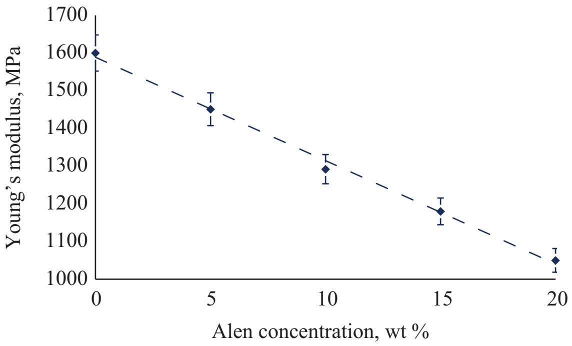


Fig. 8. Effect of the Alen concentration on the Young's modulus of the PLA/Alen polymer composite

CONCLUSIONS

The photochemical degradation of polymer composites based on PLA filled with A-Len® thermoplastic compound was studied. During extrusion of the polymer composites, the PLA phase mixed with A-Len® undergoes a change in its supramolecular structure, which manifests itself as an increase in crystallinity. This is caused by the presence of the PE phase. For this phase, the segmental mobility of macromolecules is reduced, which causes the formation of more defective and smaller crystals, resulting in decreased crystallinity. The identified structural features of the polymer composites contribute to a decrease in their performance properties due to the different polarities of the initial components.

Exposure of Alen and PLA/Alen to UV radiation leads to the formation of structural defects along with a reduction in deformation and strength properties. The most likely explanation for photodegradation is the presence of a pro-oxidant additive in Alen. In this case, the low-molecular-weight fraction of PE undergoes primary degradation, followed by fragmentation of the PE matrix. PLA undergoes photochemical degradation via the Norrish II mechanism, an oxidative degradation by a radical mechanism to form a C=C bond.

Table 5. Deformation and strength properties of polymer composites

Composites	Deformation and strength properties		
	Tensile stress at break, MPa	Elongation at break, %	Young's modulus, MPa
0 h of exposure			
PLA	67.2	4.2	1600
Alen	19.2	3.2	600
PLA/Alen5	55.1	3.8	1451
PLA/Alen10	44.0	3.4	1292
PLA/Alen15	37.8	3.2	1181
PLA/Alen20	31.5	3.0	1050
150 h of exposure			
PLA	22.4	2.8	800
Alen	–	–	–
PLA/Alen5	19.8	3.0	660
PLA/Alen10	12.6	2.9	421
PLA/Alen15	7.2	2.9	239
PLA/Alen20	5.7	2.8	189

The determined characteristics are of utility for the development of biocomposite materials having accelerated photochemical degradation kinetics.

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Authors' contributions

I.Yu. Vasilyev—experiment planning, writing and editing the text of the article.

V.S. Petrenko—conducting the study.

V.A. Rod—conducting the study.

D.A. Golobokov—conducting the study, writing and editing the text of the article.

The authors declare no conflicts of interest.

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About the Authors

Ilya Yu. Vasilyev, Cand. Sci. (Eng.), Associate Professor, Moscow Polytechnic University (38, Bolshaya Semyonovskaya ul., Moscow, 107023, Russia). E-mail: iljanaras@ya.ru. Scopus Author ID 57195569317, ResearcherID ABW-6525-2022, RSCI SPIN-code 2038-4156, https://orcid.org/0000-0001-8488-5907

Victoria S. Petrenko, Student, Moscow Polytechnic University (38, Bolshaya Semyonovskaya ul., Moscow, 107023, Russia). E-mail: petrenkoviktoria480@gmail.com. https://orcid.org/0009-0006-4131-8623

Viktor A. Rod, Student, Moscow Polytechnic University (38, Bolshaya Semyonovskaya ul., Moscow, 107023, Russia). E-mail: vikdor.andreevich@mail.ru. RSCI SPIN- code 5509-7202, https://orcid.org/0009-0008-8333-4749

Dmitrii A. Golobokov, Junior Researcher, Enikolopov Institute of Synthetic Polymeric Materials of the Russian Academy of Sciences (70, Profsoyuznaya ul., Moscow, 117393, Russia). E-mail: d.golobokov@ispm.ru. ResearcherID MBG-4040-2025, RSCI SPIN-code 7346-6740, https://orcid.org/0000-0002-4678-9866

Об авторах

Васильев Илья Юрьевич, к.т.н., доцент, ФГАОУ ВО Московский политехнический университет (Московский Политех) (107023, Россия, Москва, ул. Большая Семеновская, д. 38). E-mail: iljanaras@ya.ru. Scopus Author ID 57195569317, ResearcherID ABW-6525-2022, SPIN-код РИНЦ 2038-4156, https://orcid.org/0000-0001-8488-5907

Петренко Виктория Сергеевна, студент, ФГАОУ ВО Московский политехнический университет (Мосполитех) (107023, Россия, Москва, ул. Большая Семеновская, д. 38). E-mail: petrenkoviktoria480@gmail.com. https://orcid.org/0009-0006-4131-8623

Род Виктор Андреевич, студент, ФГАОУ ВО Московский политехнический университет (Мосполитех) (107023, Россия, Москва, ул. Большая Семеновская, д. 38). E-mail: vikdor.andreevich@mail.ru. SPIN-код РИНЦ 5509-7202, https://orcid.org/0009-0008-8333-4749

Голобоков Дмитрий Александрович, младший научный сотрудник, ФГБУН Институт синтетических полимерных материалов им. Н.С. Ениколопова Российской академии наук, (117393, Россия, Москва, ул. Профсоюзная, д. 70). E-mail: d.golobokov@ispm.ru. ResearcherID MBG-4040-2025, SPIN-код РИНЦ 7346-6740, https://orcid.org/0000-0002-4678-9866

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