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Anna BILOUSOVA, Denys BAKLAN,
Oleksandr LINYUCHEV, Oleksiy SIKORSKY,
Serhii KHLYSTUN, Oleksiy MYRONYUK
National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute", Ukraine

PHOTODEGRADATION OF BIODEGRADABLE POLYMER COATINGS BASED ON POLYLACTIDE AND POLYCAPROLACTONE

Purpose. *Comparison of the photodegradation resistance of polylactide and polycaprolactone coatings depending on the nature of the polymer and the film-forming method.*

Methodology. *Aqueous dispersions were obtained by emulsifying a polymer solution in dichloromethane, followed by removal of the organic solvent, washing, and concentration. Films from the dispersions were formed by applying them to a metal substrate, followed by heat treatment until a continuous coating was formed.*

To assess changes caused by UV irradiation, the water contact angle and surface energy were determined, and the IR spectra of the films were analyzed. The features of film formation were analyzed, and the melt flow index of polylactide and polycaprolactone granules was determined to assess a possible relationship between the polymer's rheological characteristics and the particle coalescence temperature.

Findings. *It was found that under the influence of UV irradiation, the surface of both polymers rapidly becomes hydrophilic. For films prepared from solutions, after 8 hours of irradiation, the contact angle decreased from 74±3° to 24±3° for polylactide and from 85±3° to 40±3° for polycaprolactone. For coatings formed from aqueous dispersions, the corresponding values changed from 79±3° to 33±3° and from 58±3° to 43±3°. According to IR spectroscopy data, an increase in the area of the carbonyl peak shoulder in the 1700–1690 cm^{-1} region was observed, which is associated with the accumulation of carboxylic acids as photooxidation products. It was shown that coatings formed from aqueous dispersions exhibit slightly higher resistance to UV radiation compared to films obtained from solutions.*

Originality. *The relevance of the study stems from the need to combine the environmental benefits of biopolymers with sufficient operational stability of coatings under the influence of external factors, particularly ultraviolet radiation.*

The practical value *of this work lies in establishing the influence of the coating formation method on their resistance to UV aging, which can be utilized in the development of biodegradable polymeric materials with specified performance properties. The results obtained can be used to justify the choice of a method for forming biodegradable coatings with predictable resistance to photoaging.*

Keywords: *biopolymer coatings; photooxidation; aqueous dispersions; film formation; contact angle; surface energy; IR spectroscopy.*

Introduction. Petrochemical-based plastics, which have become a staple of modern living, are characterized by their durability, resilience to degradation, and, in many instances, their single-use nature. These characteristics contribute significantly to the global volume of municipal solid waste. Projections indicate that by the year 2050, global oil consumption for plastic production will have increased by 20% (Narancic et al., 2020), and the amount of plastic entering the environment will reach 1.3 billion tons (Lau et al., 2020).

Moreover, a significant proportion of greenhouse gas emissions, approximately 50%, are directly attributable to the manufacturing process of conventional plastics (Huang et al., 2025). In contrast, the production of bioplastics has been shown to result in a reduction of emissions by 14% to 62% (Chen et al., 2023).

In this context, the transition to bioplastics is becoming increasingly important to reduce the carbon footprint and ensure biodegradability. Of particular interest within the field of biopolymers are polylactic acid (PLA) and polycaprolactone (PCL). PLA is a highly promising biopolymer that has demonstrated significant commercial viability. It is distinguished by its low density, cost-effectiveness, and optimal balance of flexibility and rigidity (Wu et al., 2023). PCL is distinguished by its high biocompatibility, biodegradability, and good elasticity, which are attributable to its low glass transition temperature (Mokoena et al., 2025; Dwivedi et al., 2019).

The combination of these characteristics renders PLA and PCL promising materials for the formation of bio-coatings, including those using aqueous dispersion systems (Belletti et al., 2021; Calosi et al., 2024).

Concurrently, the implementation of bio-coatings is associated with a number of limitations. The eco-compatibility of such materials, particularly their biodegradability, does not always align with the requirements for long-term operational stability. For practical application, the material must maintain its integrity, adhesion, and functional properties throughout its specified service life, while its degradation must occur in a controlled manner (Glaskova-Kuzmina et al., 2021), preferably after the end of its use. In this regard, achieving an equilibrium between durability and biodegradability constitutes a pressing challenge in the contemporary context (Jahangiri et al., 2024).

Despite the growing popularity of biopolymer coatings, the issue of their service life remains under-researched. For PLA, the preparation of coatings from aqueous dispersions has been the subject of study Belletti et al. (2021), but their resistance to UV radiation has not been addressed. Most studies on PLA and PCL have been conducted independently of one another, notably in the works of Copinet et al. (2004) and Vukoje et al. (2022). However, no comparative analysis of the UV resistance of coatings made from these materials by applying solutions and aqueous dispersions was found in the available sources.

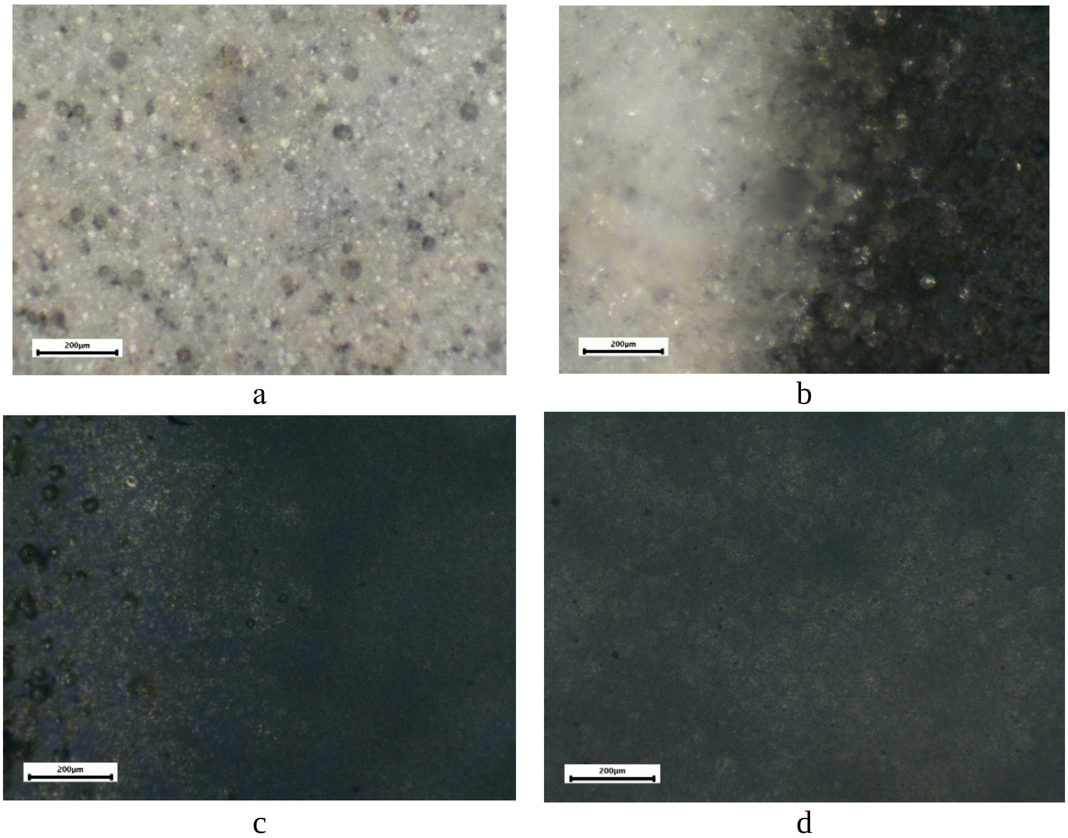
The objective of this study is to comparatively analyze the photodegradation resistance of coatings based on polylactide and polycaprolactone, obtained from solutions and aqueous dispersions. Also, a direct experimental comparison with melt-processed films was not included in the present study, since its main objective was to compare coatings formed from polymer solutions and aqueous dispersions.

Materials and Methods. In this study, PLA 4060D and PCL CAPA-6503 were used as polymers. The PLA 4060D grade was selected due to its amorphous structure (glass transition temperature of 55–60 °C), which improves its suitability for use in films. PCL CAPA-6503 was selected as a biodegradable thermoplastic polyester due to its excellent processability and ability to form flexible semi-crystalline materials. Its low glass transition temperature (−60 °C) ensures high flexibility and stability in film form. Dichloromethane was used as the solvent for PLA and PCL.

The procedure for preparing PLA and PCL dispersions was identical and consisted of the following steps. In the first step, sodium dodecyl sulfate (0.21 g) was dissolved in distilled water (53.6 g). Next, 50 g of a 5% PLA or PCL solution in dichloromethane was gradually added to the solution. The each PLA and PCL solution was mixed with the sodium dodecyl sulfate solution using a disperser at a speed of 27,000 rpm for 2 minutes. The next step was the gradual removal of dichloromethane, which was carried out on an rotary evaporator. The water bath temperature was 40 °C. To accelerate the removal of dichloromethane, a mild vacuum of up to -0.5 atm was created. After 4 hours of distillation, the resulting dispersions were cooled to room temperature and dried under vacuum in static conditions for an additional 2 hours. The next step was to wash the PLA and PCL dispersions to remove sodium dodecyl sulfate. To do this, water and dispersion particles were separated by centrifugation at 13,500 rpm for 20 minutes. The process was repeated 3 times, replacing the water remaining after centrifugation with distilled water. To concentrate the PLA dispersion for rapid water removal, a vacuum drying oven was used at 40 °C and full vacuum. Then, PCL and PLA particles from an aqueous dispersion were deposited onto a metal substrate using a coater with a gap width of 100 μm and dried at room temperature. In the next step, the metal substrate with the deposited sample was heat-treated until a continuous transparent film formed.

UV irradiation of the samples was conducted in accordance with ASTM International (ASTM D 4329) using a 250 W UVC fluorescent lamp. The samples were exposed for 8 hours at 60 ± 5 °C. Optical microscopy images were obtained on a Konus Academy optical microscope using a UCMOS 1300 digital camera (Sigeta Optics) and ToupView software. Chemical analysis was performed using FTIR spectroscopy in ATR mode (IRSpirit, Shimadzu). For water contact angle and evaluation surface tension values were used contact angle goniometer (BGD-190, Biuged Precision Instruments, Guangzhou, China) with parameters 5 μL drop volume, 10 different positions on the sample surface. The melt flow index was determined for samples in accordance to the ISO 1133-22 procedure.

Results and Discussion. The main stages of the process of forming a polycaprolactone film from dispersions are shown in (Fig. 1). During drying at room temperature, a dry, uniform layer of polymer microparticles (Fig. 1a) with a thickness of 120–150 μm was formed. This layer was heated on a heating plate at a rate of 10 °C/min. Fig. 1b shows the phase boundary of the material discrete particles a continuous film with air inclusions. The coalescence of particles is due to the fact that as the temperature rises to 60 °C, the interaction between particles intensifies due to surface forces, resulting in a significant number of particles deforming and beginning to coalesce, forming a film (Belletti et al., 2021). At this stage, small particles coalesce first, while larger particles and aggregates remain visible on the surface (Fig. 1c). At this stage, the film is not fully transparent. To complete the formation of a continuous coating, including the coalescence of larger particles and aggregates, the thermal treatment temperature was increased to 80 °C and maintained for 5 min (Fig. 1d).



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Source: *developed by the authors of this study based on the findings of experimental research.*

Fig. 1. **Optical microscopy of film formation from PCL dispersion particles:**
a) 25 °C; b) 50 °C; c) 60 °C; d) 80 °C

The experiment revealed that the initial contact angle for films produced from dispersed particles is abnormally low, at approximately 40–45°. After rinsing the film with clean warm water, the contact angle returns to the typical range for polycaprolactone and polylactide of 60–80° (Fig. 2).

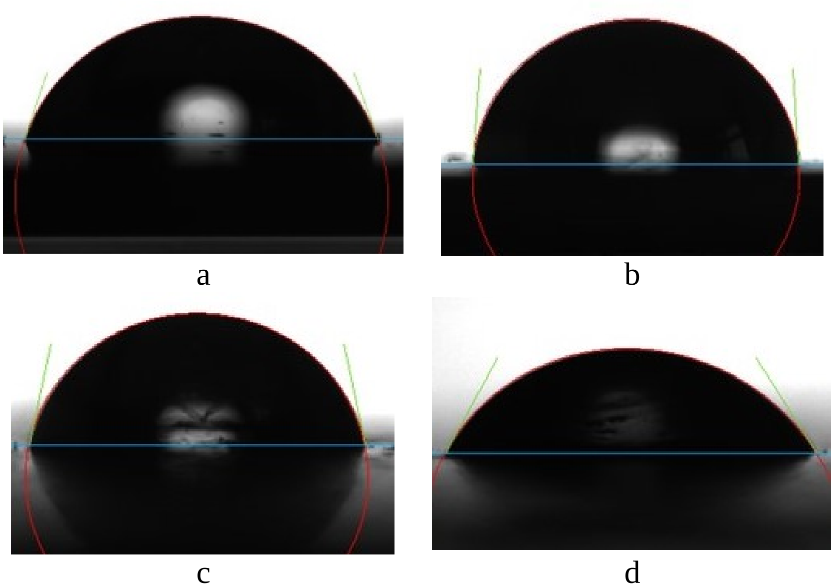

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Fig. 2. **Initial contact angles of films**:
a) PLA solution; b) PCL solution; c) PLA dispersion; d) PCL dispersion

This may be due to the presence of residual sodium dodecyl sulfate (SDS) remaining after the dispersion stage (Tzitzinou et al., 1999). During the thermal sintering of the particles, the surfactant likely migrates to the surface of the film, which leads to a decrease in the contact angle.

The effect of heat treatment itself on surface hydrophilization has not been confirmed, since the films obtained from the solution did not show a decrease in the contact angle even after 30 minutes of heat treatment under similar conditions.

In this regard, during sample preparation prior to measuring the contact angle, it was decided to rinse them with warm water and dry them in an oven at 40 °C.

The film-forming process of polylactic acid films differed from PCL only in the characteristic temperatures of the film-forming processes and was studied in detail by us in O. Myronyuk et al. (2025). In particular, for unplasticized PLA, the onset temperature of film formation is 110 °C, and the formation of a continuous film (final removal of entrapped air bubbles) is 160 °C. In principle, film formation from such dispersions is similar to the formation of films from aqueous polymer emulsions, but requires additional heating after water evaporation.

It is known that film formation from aqueous polymer dispersions occurs through the sequential evaporation of the dispersion medium and the coalescence of polymer particles under the action of capillary forces (Brock et al., 2010). The determining parameters of this process are both the value of the capillary pressure (determined by the size of the capillaries between the particles) and the softness of the polymer particles, their ability to deform (Semerdzhiev et al., 2024). Accordingly, a hypothesis was formulated regarding a probable correlation between the ratio of polymer viscosity and the minimum temperature of their film formation.

To test this hypothesis regarding a possible correlation between the melt flow index (MFI) and the particles' ability to coalesce (Voogt et al., 2019), MFI measurements were conducted for polylactide and polycaprolactone pellets (Fig. 3).

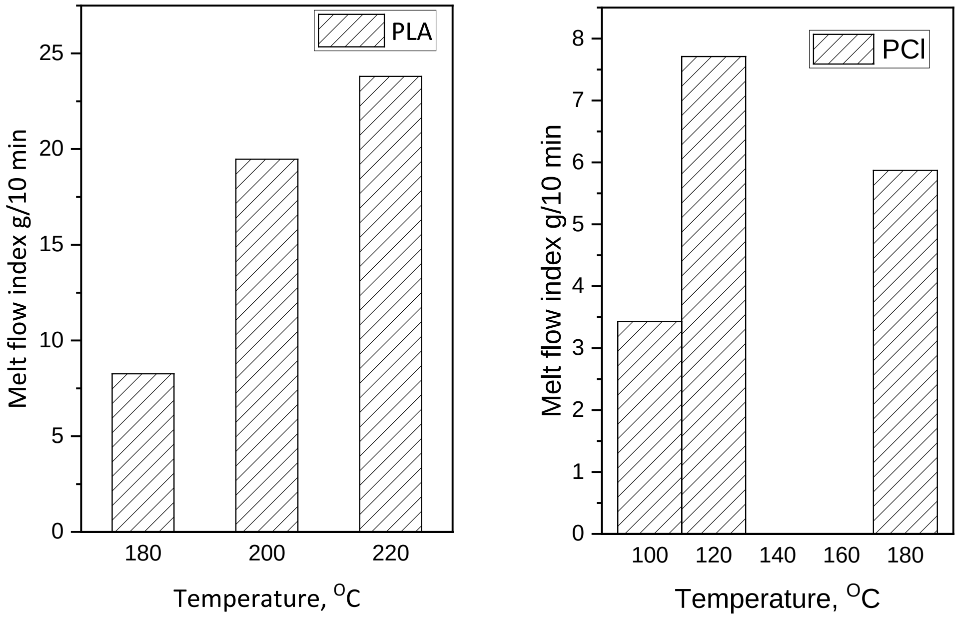

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Fig. 3. **Melt flow index of PLA and PCL pellets**

It was expected that a higher MFI, as an indicator of greater melt fluidity, would correspond to a lower film-forming temperature. However, the results did not confirm this relationship. For PCL at 180 °C, the MFI value was lower than that for PLA, at 5.87 versus 8.26, while the film-forming temperature of polycaprolactone is lower than that of polylactide. For PCL, film formation begins as early as 60 °C (30 sec), but to ensure complete fusion of all aggregates, the temperature was increased to 80 °C (10 sec). For PLA, film formation begins at 110 °C (60 sec), and the film becomes fully transparent at 130 °C in 30 sec.

One possible explanation for this discrepancy is that particle coalescence occurs predominantly not in the temperature range corresponding to the viscoelastic state, but in that characteristic of the highly elastic plateau. As described in Man and Russel (2008), the particles deform, filling the free volume, accompanied by surface diffusion of the mobile segments of the polymer chains. In this case, the determining parameters are the elastic modulus of the particle material at a given temperature and their “stickiness”, which is due to segmental mobility.

Degradation of films under UV light. To compare the effects of UV irradiation on the surface of the films, the water contact angle was measured. The results of the study (Table 1) indicate that under the influence of UV radiation, the surface of both polymers rapidly becomes hydrophilic (Koo & Jang, 2008), with PLA being more sensitive to UV radiation than PCL.

Table 1

Change in the water contact angle of PLA and PCL films as a function of UV irradiation duration

Sample	Water contact angle, ° (0 hours)	Water contact angle, ° (4 hours)	Water contact angle, ° (8 hours)
PLA, solution-based film	74±3	58±3	24±3
PCL, solution-based film	85±3	76±3	40±3
PLA dispersion-based film	79±3	56±3	33±3
PCL, dispersion-based film	58±3	56±3	43±3

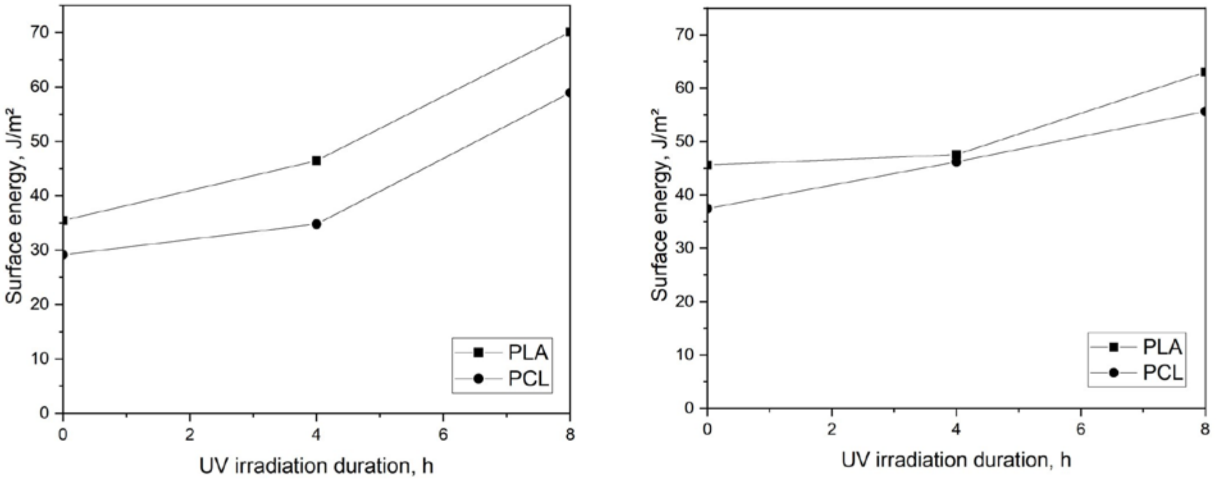
Source: *developed by the authors of this study based on the findings of experimental research.*

For films prepared from solution, the initial contact angle of PLA was 74±3°, and that of PCL was 85±3°. After 4 hours of UV irradiation, this value decreased to 58±3° for PLA and 76±3° for PCL, and after 8 hours – to 24±3° and 40±3°, respectively. A similar pattern was observed for films formed from dispersed particles. The initial contact angle for PLA was 79±3°, and for PCL, 58±3°. After 4 hours of irradiation, both samples had a contact angle of 56±3°, and after 8 hours, this value decreased to 33±3° for PLA and 43° for PCL.

Change in surface energy. The hydrophilization of PLA and PCL polymer films is primarily attributed to an oxidation process involving ultraviolet light quanta and oxygen molecules (or ozone), which, upon interaction with surface micromolecules, lead to the formation of a set of polar groups – ranging from unstable hydroperoxides in the initial stages of the process to relatively stable carboxyl groups that accumulate on the surface. Consequently, these groups increase the growth of the polar component of surface energy, the wettability by polar liquids, and, in particular, water (Koo & Jang, 2008).

Fig. 4a shows that under UV irradiation, the surface energy of both polymers increases at a rate of approximately 1.8–1.9 times after 8 hours of irradiation. PCL has a lower initial surface energy and, consequently, a lower surface energy after irradiation as well.

The same trend is observed when coatings are obtained from polymer dispersions, but for them the overall rate of energy increase is lower, as is the resulting value after 8 hours of irradiation. Also noteworthy is the difference between the surface energy values for soluble films and dispersions after 4 and 8 hours of irradiation.



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Source: *developed by the authors of this study based on the findings of experimental research.*

Fig. 4. **Change in surface energy as a function of UV irradiation duration:**
a) films formed from solutions; b) films based on dispersed particles

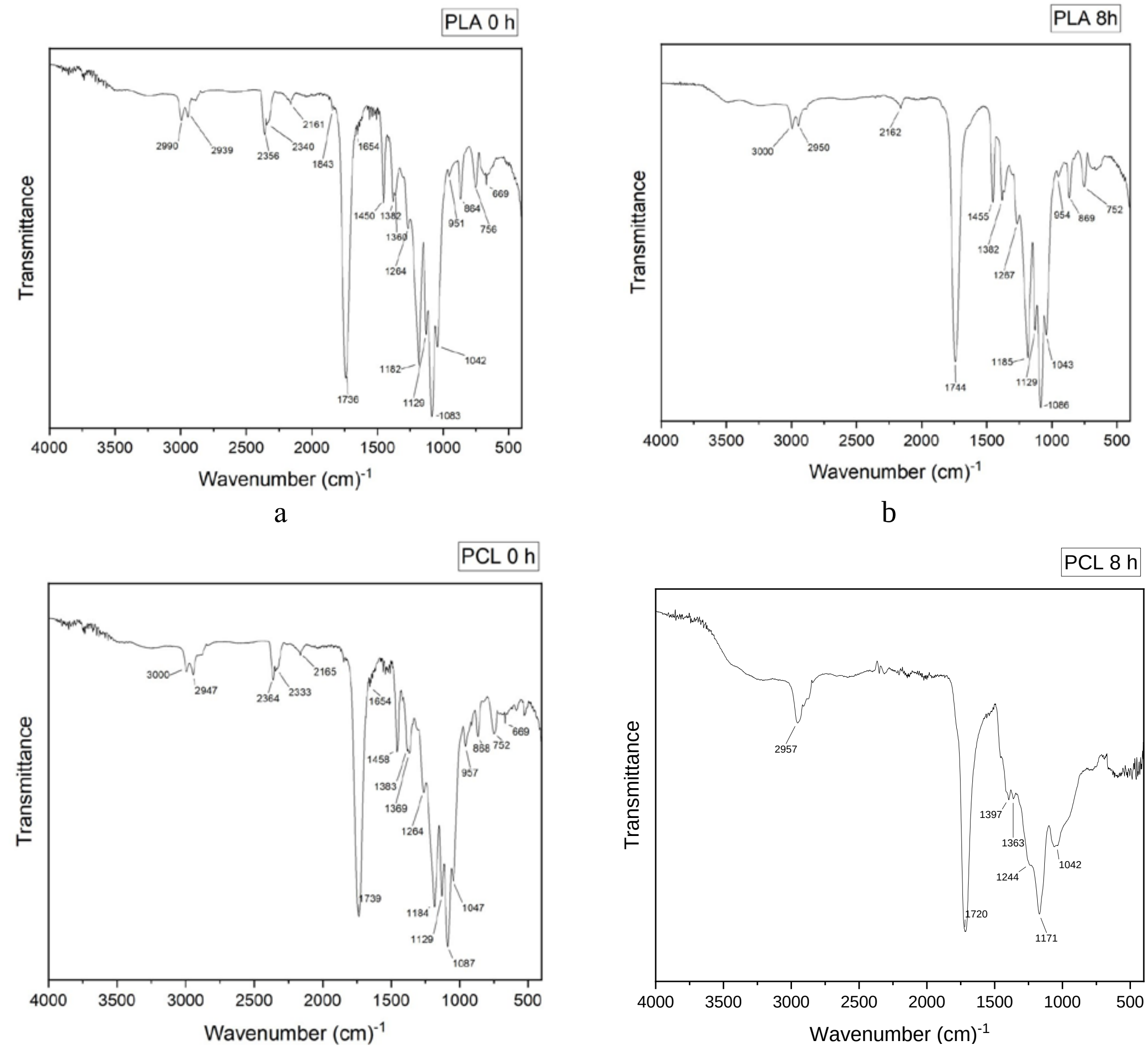
For films prepared from a solution, the rate increases significantly after 4 hours of irradiation, whereas for those prepared from dispersions, it remains nearly constant.

This difference can be explained by the fact that films formed from a solution in dichloromethane are characterized by a denser packing of macromolecules and a smaller number of structural defects and amorphous interparticle boundaries, which reduces scattering and shielding of UV radiation and promotes more efficient photodegradation. At the same time, films from aqueous dispersions have a heterogeneous morphology with interparticle boundaries and residual stabilizers, which can partially absorb or scatter UV radiation and slow down degradation (Tzitzinou et al., 1999).

IR spectroscopy. The primary mechanism of polylactide photooxidation involves the photolysis of the ester bond, followed by radical chain scission via the Norrish II mechanism and the formation of hydroperoxides, which subsequently decompose to form carboxyl and unsaturated groups (Olewnik-Kruszkowska et al., 2015). At the same time, given the significantly higher intensity of the absorption bands of the valence vibrations of carboxyl groups compared to the corresponding vibrations of unsaturated bonds, analysis of this specific part of the spectrum is the most accurate.

The primary mechanism of polycaprolactone photodegradation involves auto-oxidation leading to the formation of hydroperoxides and subsequent β-cleavage of the macromolecular chain. In the IR spectrum, this manifests as an increase in the intensity of the carbonyl (~1700–1730 cm^{-1}) and hydroxyl (3600–3200 cm^{-1}) groups, as well as a decrease in the intensity of the C–O–C ester bond bands (França et al., 2018).

To characterize the degradation processes based on the analysis of spectra of polymer films obtained from solutions (Fig. 5), the ratio of the area of the "shoulder" of the main carbonyl peak at 1700–1690 cm^{-1} in the fresh film to that in the film after 8 hours of degradation was used as an indicator.



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Source: *developed by the authors of this study based on the findings of experimental research.*

Fig. 5. **Changes in the IR spectrum as a function of UV irradiation duration for solution-cast films**

For polylactide, this value is 1.45, and for polycaprolactone, it is 1.5. This confirms previous findings regarding the similarity in the photodegradation rates of these materials.

At the same time, for the dispersions (Fig. 6), this coefficient is lower – 1.32 and 1.27, respectively – which indicates a slight slowing of the oxidation process in this case.

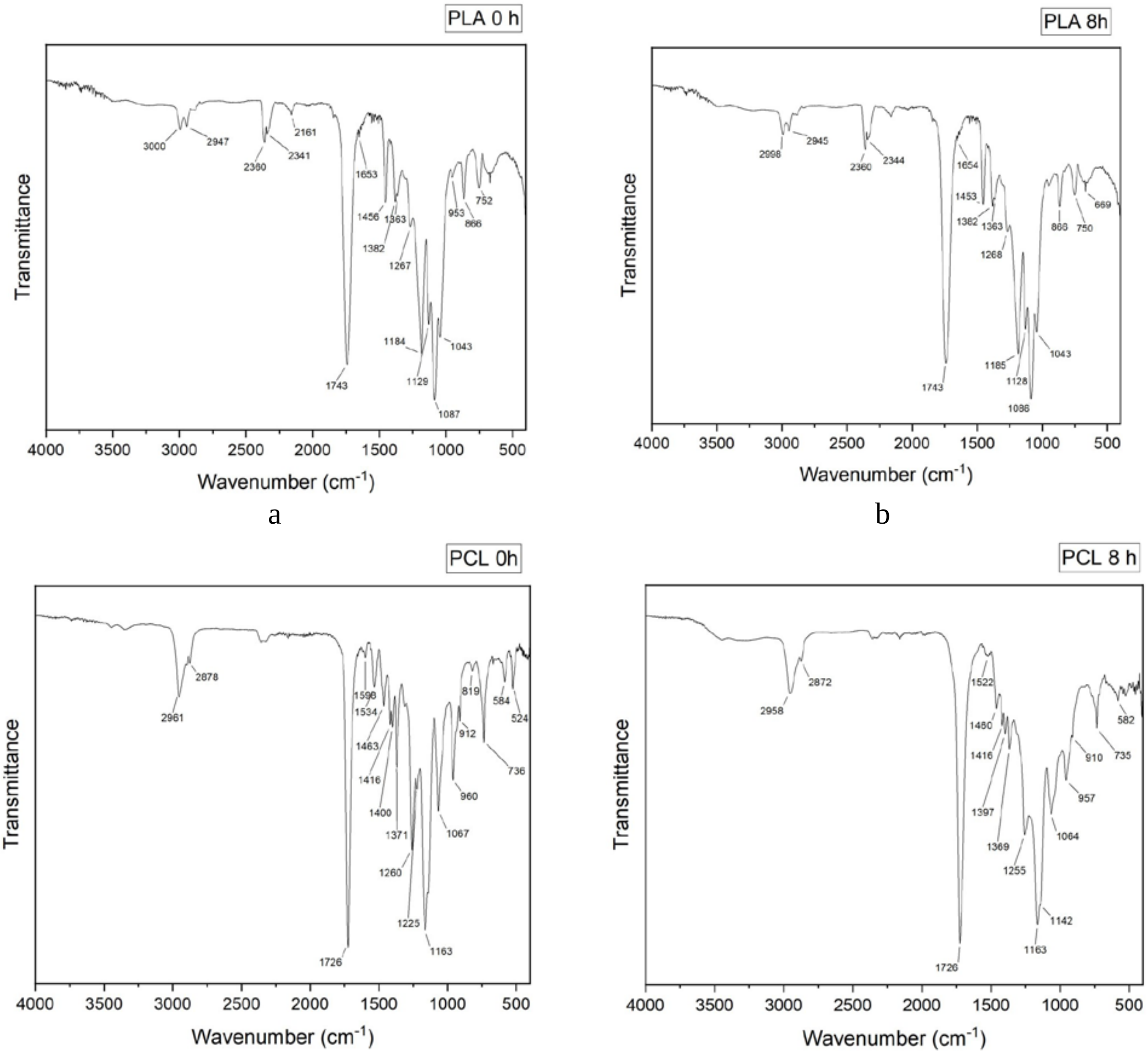

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Fig. 6. **Changes in the IR spectrum as a function of UV irradiation duration for films from dispersions**

Overall, the results obtained are consistent with observations of changes in the surface energy of polymers during photooxidation. It can be concluded that the main reason for this increase is the accumulation of carboxylic acids, which are produced during the decomposition of hydroperoxides formed as a result of contact between polymer molecules and atmospheric oxygen (Olewnik-Kruszkowska et al., 2015b).

Future research in this area should focus on expanding comparisons of various methods for forming biodegradable polymer coatings, particularly those involving melt-formed films, and on evaluating the effects of coating thickness, crystallinity, and morphology on their resistance to UV aging. It is also advisable to study long-term aging under the combined action of UV radiation, temperature, humidity, and oxygen, as well as to investigate changes in mechanical properties, adhesion to the substrate, molecular weight, and biodegradation behavior after irradiation. In future work, special attention should be paid to the influence of residual stabilizers, surfactants, plasticizers, and functional fillers on the photooxidation processes of PLA and PCL coatings, which will allow for the targeted regulation of the balance between operational stability and the materials' ability to undergo controlled degradation.

Conclusion. It has been shown that the photodegradation rates of polylactide and polycaprolactone are very similar: the area of the "shoulder" at 1700–1690 cm^{-1}, which is an indicator of the amount of carboxylic acids products of photodegradation increases by 1.45 for polylactide and by 1.5 for polycaprolactone after 8 hours of irradiation. This is accompanied by an increase in the surface energy of these materials from 35 to 70 J/m^2 and from 30 to 62 J/m^2, respectively. It has been established that films made from dispersions are somewhat more resistant to the effects of ultraviolet radiation compared to films made from solutions. Thus, for them, the area of the specified "shoulder" increases only by a factor of 1.32 and 1.27, while the surface energy increases to 64 J/m^2 for polylactide and 56 J/m^2 for polycaprolactone. It has been proven that the ratio of the minimum film-forming temperatures of the studied polymers does not correlate with the ratio of their melt flow indices.

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Conflict of Interest. None.

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BILOUSOVA ANNA
PhD Student,
Department of Chemical Technology
of Composite Materials,
National Technical University of Ukraine
"Igor Sikorsky Kyiv Polytechnic Institute", Ukraine
https://orcid.org/0000-0002-2818-8450
Scopus Author ID: 58668975800
E-mail: a.bilousova@kpi.ua

BAKLAN DENYS
PhD, Assistant,
Department of Chemical Technology
of Composite Materials,
National Technical University of Ukraine
"Igor Sikorsky Kyiv Polytechnic Institute", Ukraine
https://orcid.org/0000-0002-6608-0117
Scopus Author ID: 57194569043
E-mail: d.baklan@kpi.ua

LINYUCHEV OLEKSANDR
PhD, Associate Professor,
Department of Ecology and Plant Polymers Technology,
National Technical University of Ukraine
"Igor Sikorsky Kyiv Polytechnic Institute", Ukraine
https://orcid.org/0000-0003-1683-5937
Scopus Author ID: 57205182822
E-mail: o.liniuchev@xtf.kpi.ua

SIKORSKY OLEKSIY
PhD, Senior Lecturer,
Department of Chemical Technology
of Composite Materials,
National Technical University of Ukraine
"Igor Sikorsky Kyiv Polytechnic Institute", Ukraine
https://orcid.org/0000-0002-3730-2016
E-mail: o.sikorsky@kpi.ua

KHLYSTUN SERHII
PhD Student,
Department of Chemical Technology
of Composite Materials,
National Technical University of Ukraine
"Igor Sikorsky Kyiv Polytechnic Institute", Ukraine
https://orcid.org/0009-0006-8765-2360
E-mail: khlystun.s.o.-khk41f@edu.kpi.ua

MYRONYUK OLEKSIY
Doctor of Technical Sciences, Associate Professor,
Head of Department, Department of Chemical
Technology of Composite Materials,
National Technical University of Ukraine
"Igor Sikorsky Kyiv Polytechnic Institute", Ukraine
https://orcid.org/0000-0003-0499-9491
Scopus Author ID: 57190497257
E-mail: o.myronyuk@kpi.ua

Анна БІЛОУСОВА, Денис БАКЛАН, Олександр ЛІНЮЧЕВ,
Олексій СІКОРСЬКИЙ, Сергій ХЛИСТУН, Олексій МИРОНЮК
Національний технічний університет України «Київський політехнічний інститут імені Ігоря Сікорського», Україна

ФОТОДЕСТРУКЦІЯ БІОРОЗКЛАДНИХ ПОЛІМЕРНИХ ПОКРИТТІВ НА ОСНОВІ ПОЛІЛАКТИДУ І ПОЛІКАПРОЛАКТОНУ

Мета. *Порівняння стійкості до фотодеструкції покриттів на основі полілактиду та полікапролактону залежно від природи полімеру та способу формування плівки.*

Методика. *Водні дисперсії одержували методом емульгування розчину полімеру в дихлорметані, з подальшим видаленням органічного розчинника, промиванням і концентруванням. Плівки з дисперсій формували нанесенням на металеву підкладку з наступною термічною обробкою до утворення суцільного покриття.*

Для оцінки змін, спричинених УФ-опроміненням, було визначено кут змочування водою, поверхневу енергію, а також проаналізовано ІЧ-спектри плівок. Проаналізовано особливості плівкоутворення та визначено показник текучості розплаву гранул полілактиду і полікапролактону для оцінки можливого зв'язку між реологічними характеристиками полімеру та температурою коалесценції частинок.

Результати. *Встановлено, що під дією УФ-опромінення поверхня обох полімерів швидко гідрофілізується. Для плівок, отриманих із розчинів, після 8 годин опромінення кут змочування зменшувався з 74±3° до 24±3° для полілактиду та з 85±3° до 40±3° для полікапролактону. Для покриттів, сформованих із водних дисперсій, відповідні значення змінювалися з 79±3° до 33±3°та з 58±3° до 43±3°. За даними ІЧ-спектроскопії встановлено зростання площі плеча карбонільного піку в області 1700–1690 см$^{-1}$, що пов'язано з накопиченням карбонових кислот як продуктів фотоокиснення. Показано, що покриття, сформовані з водних дисперсій, виявляють дещо вищу стійкість до дії УФ-випромінювання порівняно з плівками, отриманими з розчинів.*

Наукова новизна. *Актуальність дослідження зумовлена необхідністю поєднання екологічних переваг біополімерів із достатньою експлуатаційною стабільністю покриттів під дією зовнішніх чинників, зокрема ультрафіолетового випромінювання.*

Практична значимість *роботи полягає у встановленні впливу способу формування покриттів на їх стійкість до УФ-старіння, що може бути використано під час розроблення біорозкладних полімерних матеріалів із заданими експлуатаційними властивостями. Отримані результати можуть бути використані для обґрунтування вибору способу формування біорозкладних покриттів із прогнозованою стійкістю до фотостаріння.*

Ключові слова*: біополімерні покриття; фотоокиснення; водні дисперсії; плівкоутворення; кут змочування; поверхнева енергія; ІЧ-спектроскопія.*