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STRUCTURAL FEATURES AND MORPHOLOGY OF BIODEGRADABLE COMPOSITES BASED ON POLYLACTIDE AND CORN STARCH

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The aim of the work was to study the morphology and structural features of biodegradable composites based on polylactide, filled with corn starch formed by extrusion method. The characteristics of the obtained materials were investigated by scanning electron microscopy, X-ray diffraction, IR spectroscopy. It was established that the presence of polyethylene glycol PEG–4000 as a plasticizer and surfactant – glycerin monostearate in the initial mixture leads to the formation of a nonporous heterogeneous system, in which starch particles are statistically distributed in the matrix. The starch concentration in the composite varied from 20 to 55 wt. %. When the starch concentration increases to the limit values, agglomeration of filler particles occurs and a sharp deterioration in the mechanical properties of the composite. The XRD analysis of the samples showed that the initial PLA does not actually have a crystalline phase, and when PEG and starch are introduced into the composite, intense reflexes are observed on the diffractogram, which are most likely a superposition of two reflections - from the starch and the crystalline phase of the polylactide. The effect of PEG and starch on the composite structure was analyzed using the IR spectroscopy method. No new bands were found in the spectrum of the composite compared to the IR spectrum of the original polylactide, which indicates the absence of chemical interaction between the components. The most intense bands in both samples are located in the region of 1200-1750 cm^{-1} and relate to the valence vibrations of C–O bonds (C–O–C and / or C–O(H)), as well as to the valence vibrations of C–C bonds, i.e. only polylactide is present on the surface of the sample, and the surface. The composite is a plasticized polylactide, interspersed with starch particles coated with a polymer film. In addition, in the IR spectrum of the composite, the band corresponding to the valence vibrations of bound hydroxyl groups (3200–3500 cm^{-1}) has a significantly higher intensity and width, which indicates an additional contribution of OH- groups present in the PEG molecule. Biodegradability tests conducted in accordance with the international standard ISO 16929:2013 showed that the obtained materials undergo complete chemical and biological destruction in compost within 5–6 months.

Keywords: biodegradable composite, polylactide, corn starch

СТРУКТУРНЫЕ ОСОБЕННОСТИ И МОРФОЛОГИЯ БИОРАЗЛАГАЕМЫХ КОМПОЗИТОВ НА ОСНОВЕ ПОЛИЛАКТИДА И КУКУРУЗНОГО КРАХМАЛА

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Целью работы было изучение морфологии и структурных особенностей биоразлагаемых композитов на основе полилактида, наполненного кукурузным крахмалом, формируемых экструзионным методом. Характеристики полученных материалов были исследованы сканирующей электронной микроскопией, рентгенографией, ИК-спектроскопией. Установлено, что присутствие в исходной смеси пластификатора полиэтиленгликоля ПЭГ-4000 и поверхностно-активного вещества – глицерин моностеарата ведет к формированию беспористой гетерогенной системы, в которой частицы крахмала статистически распределены в матрице полилактида, Концентрацию крахмала в составе композита варьировали от 20 до 55 мас. %. При увеличении концентрации крахмала до предельных значений происходит агломерация частиц наполнителя и резкое ухудшение механических свойств композита. Исследование образцов методом РФА показало, что исходный полилактид фактически не имеет кристаллической фазы, а при введении в состав композита полиэтиленгликоля и крахмала на дифрактограмме наблюдаются интенсивные рефлексы, которые, скорее всего, являются суперпозицией двух отражений - от крахмала и кристаллической фазы полилактида. Анализ влияния ПЭГ и крахмала на структуру композита проводили с использованием метода ИК-спектроскопии. Новых полос в спектре композита по сравнению со спектром исходного полилактида не обнаружено, что свидетельствует об отсутствии химического взаимодействия между компонентами. Наиболее интенсивные полосы в обоих образцах расположены в области 1200–1750 см$^{-1}$ и относятся к валентным колебаниям связей С–О (С–О–С и/или С–О(Н)), а также к валентным колебаниям связей С–С, т.е. на поверхности образца присутствует только полилактид, а поверхность композита представляет собой, пластифицированный полимер, с вкраплениями частиц крахмала, покрытых пленкой полилактида. Кроме того, в ИК-спектре композита полоса, соответствующая валентным колебаниям связанных гидроксильных групп (3200 – 3500 см$^{-1}$), имеет значительно большую интенсивность и ширину, что указывает на дополнительный вклад ОН-групп, присутствующих в молекуле ПЭГ. Испытания на биоразлагаемость, проведенные в соответствии с международным стандартом ISO 16929:2013 показали, что полученные материалы подвергаются полному химическому и биологическому разрушению в компосте в течение 5–6 месяцев.

Ключевые слова: биоразлагаемые композиты, полилактид, кукурузный крахмал

POLILAKTID VA MAKKA KRAXMALI ASOSIDAGI BIOPARCHALANUVCHAN KOMPOZITLARNING TUZILISH XUSUSIYATLARI VA MORFOLOGIYASI

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Ishning maqsadi ekstruziya usulida hosil bo'lgan makkajo'xori kraxmal bilan to'ldirilgan polilaktid asosidagi biologik parchalanadigan kompozitlarning morfologiyasi va strukturaviy xususiyatlarini o'rganishdan iborat. Olingan materiallarning xarakteristikalari *elektron mikroskopiya, rentgen nurlari diffraktsiyasi, IQ spektroskopiyasi orqali o'rganilgan. Dastlabki aralashmada plastifikator va sirt faol modda - glitserin monostearat sifatida polietilen glikol PEG-4000 mavjudligi kraxmal zarralari matritsada statistik taqsimlangan g'ovak bo'lmagan heterojen tizimning shakllanishiga olib kelishi aniqlandi. Kompozitdagi kraxmal konsentratsiyasi 20 dan 55% mass oraliqda o'zgaradi. Kraxmal konsentratsiyasi chegaraviy qiymatlarga ko'tarilganda, to'ldiruvchi zarrachalarning aglomeratsiyasi va kompozitning mexanik xususiyatlarining keskin yomonlashishi sodir bo'ladi. Namunalarning XRD tahlili shuni ko'rsatdiki, dastlabki PLA aslida kristall fazaga ega emas va kompozitsiyaga PEG va kraxmal kiritilganda, diffraktogrammada kuchli reflekslar kuzatiladi, bu ikki-kraxmal va polilaktidning kristal fazasining aks ettirishning superpozitsiyasi bo'lishi mumkin. PEG va kraxmalning kompozit tuzilishga ta'siri IQ spektroskopiya usuli yordamida tahlil qilindi. Asl polilaktidning IQ spektri bilan solishtirganda kompozitning spektrida yangi chiziqlar topilmadi, bu komponentlar o'rtasida kimyoviy o'zaro ta'sir yo'qligini ko'rsatadi. Ikkala namunadagi eng qizg'in chiziqlar 1200-1750 sm^{-1} oraliqda joylashgan va C-O aloqalarining (C-O-C va / yoki C-O (H)) valentlik tebranishlari bilan bog'liq. C-C bog'lanishlarining valent tebranishlariga, ya'ni namuna yuzasida faqat polilaktid mavjud va kompozitning sirti plastiklashtirilgan polimer plyonka bilan qoplangan kraxmal zarralari bilan bog'langan polilaktiddan iborat. Bundan tashqari, kompozitning IQ spektrida bog'langan gidroksil guruhlarning (3200-3500 sm^{-1}) valentlik tebranishlariga mos keladigan tasma sezilarli darajada yuqori intensivlik va kenglikka ega, bu esa PEG molekulasida OH-guruhlarining qo'shimcha hissasini ko'rsatadi. ISO 16929:2013 xalqaro standartiga muvofiq o'tkazilgan biologik parchalanish sinovlari shuni ko'rsatdiki, olingan materiallar 5-6 oy ichida kompostda to'liq kimyoviy va biologik parchalanadi.*

Kalit so'zlar: biologik parchalanadigan kompozitlar, polilaktid, makkajo'xori kraxmal

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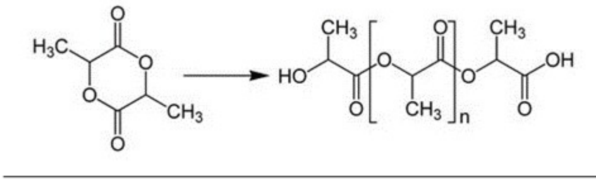
Introduction

The problem of creation, production and application of biodegradable materials is becoming increasingly relevant in connection with the global pollution of the environment by plastic waste, the growth of which is becoming alarming [1–3]. Biodegradable polymer composites cannot completely solve the problem of environmental pollution, but they can partially replace traditional plastics in consumer goods such as soft and hard packaging, various containers and tableware [4–6]. Many countries have adopted a number of legislative acts concerning the replacement of disposable plastic products - bags, cups, cutlery, etc. with those made of biodegradable materials. In recent years, intensive research and development has been carried out concerning the creation and production of materials based on biodegradable polymers filled with natural fibers and polysaccharides, including various types of starches [7–12]. The production of biodegradable polyesters is developing quite rapidly, among which polylactide (polylactic acid, PLA) is one of the most important and widely used biopolymers with many excellent characteristics, which allows it to be used, including for packaging and in medicine [13–15]. Polylactide has quite good mechanical properties, however, its brittleness, low softening temperature (about 60 °C) and high production cost prevent its widespread use in its pure form. It is also known that polylactide decomposes rather slowly in soil or compost [16]. Polylactide is a product of polymerization of lactic acid, which, in turn, is formed as a result of lactic acid fermentation of glucose (from corn, wheat, etc.) [17]:



A significant point in the production technology of PLA and products made from it is the presence of stereoisomers in the lactic acid molecule. The lactic acid polymer can exist in two versions (L and D), which are mirror images of each other. One hundred percent L-PLA has a crystalline structure with a certain melting point, while a mixture of isomers has an amorphous glassy structure. By varying the ratio of isomers, it is possible to achieve a wide range of properties in products depending on their purpose [18]. Methods for obtaining and research results of composite materials based on polylactide with various fillers, including starch, are described in a number of reviews and original articles [19–22]. However, the issues of combining components and increasing the biodegradability of composites remain unresolved.

This work is devoted to the study of the structural features and morphology of biodegradable composites formed by extrusion using polylactide as a matrix and corn starch as an organic biodegradable filler.

Research methods

To obtain experimental tape samples of biodegradable materials based on a mixture of polylactide and starch by extrusion, the following starting materials were used: Ingeo Biopolymer 4043D PLA (manufactured by Natureworks LLC, USA) in granules; polyethylene glycol PEG-4000, average molecular weight is in the range of 3500–4500; crystallization temperature is 50–55 ºC; mass fraction of water is no more than 1.0 %) (hereinafter designated as PEG), which served as a plasticizer and combining agent for the PLA-filler system.

Corn starch, which is a white powder with a grain size of 10–15 microns, was chosen as a biodegradable filler. Corn starch grains, unlike other types of starches, are characterized by smaller sizes and a small spread of particle sizes, therefore, corn starch powder (hereinafter referred to as CS) is preferred as a filler. To improve the rheological characteristics of the mixture, surface active agents was used glycerol monostearate GMS (purity is 98.1%, melting point is 64.5 ºC).

Titanium dioxide in the crystalline form of anatase TiONA AT-1 used in the composition as a photoactive agent that promotes the destruction of intramolecular bonds in polymers when exposed to the ultraviolet component of solar radiation [23]. In addition, TiO_2 is used in the composite as a white dye. Titanium dioxide is considered ecofriendly and is classified as a food additive E171.

The crystal structure was investigated by X -ray diffraction (XRD) measurements using DRON -7 diffractometer.

SEM measurements have been performed by Vega II LSH Tescan, Czech Republic scanning electron microscope.

IR spectra were performed by using Thermo Nicolet Nexus FT-IR Fourier transform spectrometer (USA) in the range of 4000–400 cm^{-1}.

Tape samples of composites with a thickness of 0.5 mm were drawn on a single-screw extruder HAAKE RHEOCORD-90.

Biodegradability tests were carried out on disc samples cut from experimental tapes in accordance with ISO 16929:2013.

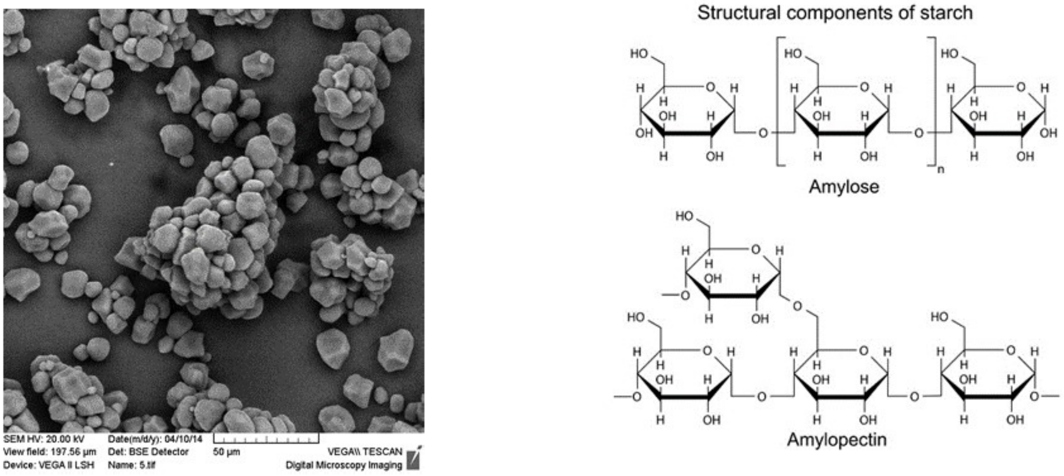
Results and Discussion

Starch in corn grains is present in the form of granules, the size of which ranges from 2 to 10 μm (Fig. 1, a). During storage, starch particles stick together, forming agglomerates that are easily dispersed under mechanical forces. From a chemical point of view, starch consists of a mixture of polysaccharides, which can be divided into two fractions - amylose and amylopectin, which differ in the structure of the carbohydrates they contain (Fig. 1, b) [24].

When preparing PLA–filler composites, the composition of the initial charge was varied as follows: 35–60 wt.% granulated PLA, 20–55 wt.% CS, 5–15 wt.% PEG, 4–5 wt.% GMS and 1 wt.% TiONA.

The biodegradable composite is made according to the following scheme. PLA granules are mixed with PEG in a high-speed heated turbo mixer at a temperature of 80 ºC, added to the CS mixture, and kept at a temperature of 100 ºC for 30 minutes; then add GMS, TiONA powder to the mixer, continue heating and rotating until a homogeneous mixture is obtained, then cool the mixture to 25–30 ºC and after that the mixture is loaded into a single-screw extruder for melting and homogenization. The temperature of the melt at the exit from the slotted extruder head is 180–185 ºC. The temperature of the melt at the exit from the slotted extruder head is 180–185 ºC. The melt enters the calender, cooled and formed in the form of a tape (Fig. 2, a).

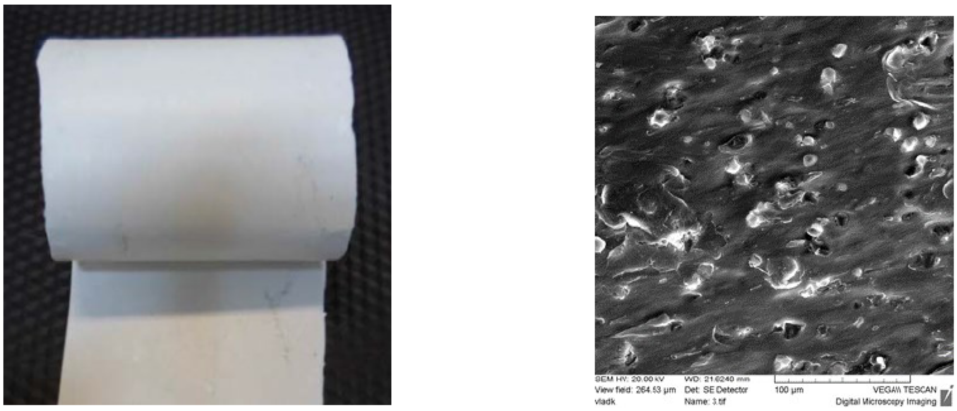
From the analysis of electron microscopic images of the tape composite, we can conclude that the presence of a plasticizer (PEG) and surfactant in the initial mixture contributes to the formation of a fairly homogeneous structure in which starch particles are statistically distributed in the matrix (Fig. 2, b). In the absence of CS in the composition, the



a

b

Figure 1. SEM image of CS granules (a), chemical structure of starch (b).

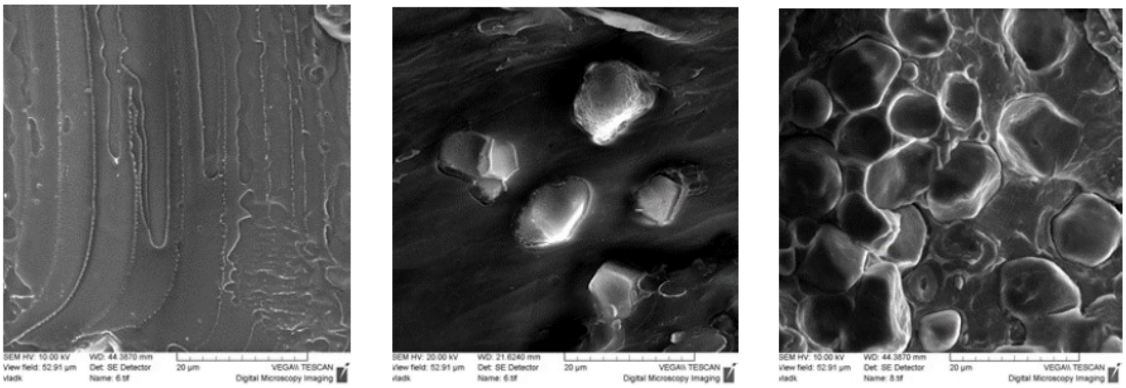


a b

Figure 2. Sample of composite extrusion tape (a); SEM image of the tape surface (b).

tape acquires a banded morphology, which indicates the heterogeneity of the composition in the direction of movement of the tape when exiting the slotted extruder head (Fig. 3, a), at the same time, the presence of crystalline particles of CS helps to improve the homogeneity when mixing the initial mixture in the extruder (Fig. 3, b). When the starch concentration increases to the limiting values (55 wt.%), agglomeration of filler particles occurs (Fig. 3, c) and a sharp deterioration in the mechanical properties of the composite.

X-ray diffraction analysis showed (Fig. 4) that the original PLA actually does not have a crystalline phase, showing nuclei (reflection at 2Θ



a b c

Figure 3. SEM – image of the composite (surface) at different CS contents: a – 0%; b – 20%; c – 55%.

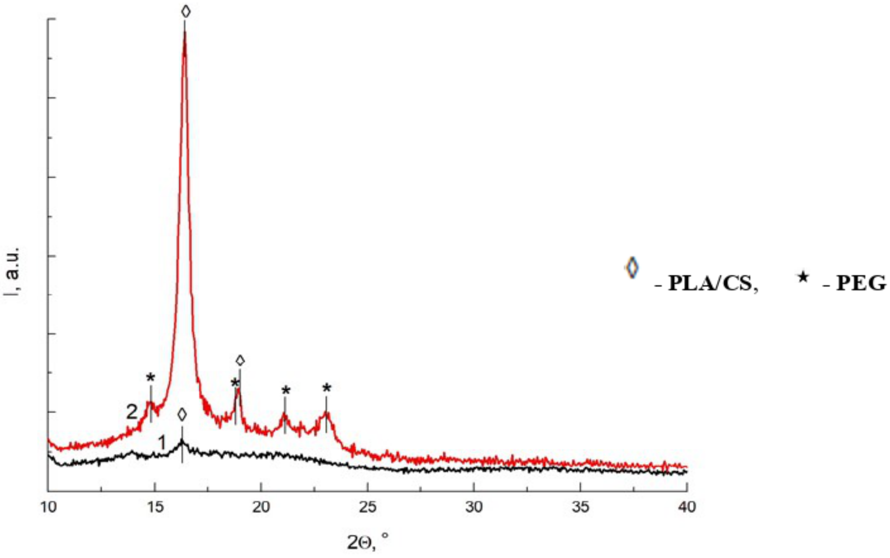


Figure 4. XRD patterns for samples:
1 – pure (initial) PLA; 2 – composite PLA (59 wt.%), CS (20 wt.%), PEG (15 wt.%), GMS (5 wt.%, TiONA (1 wt.%).

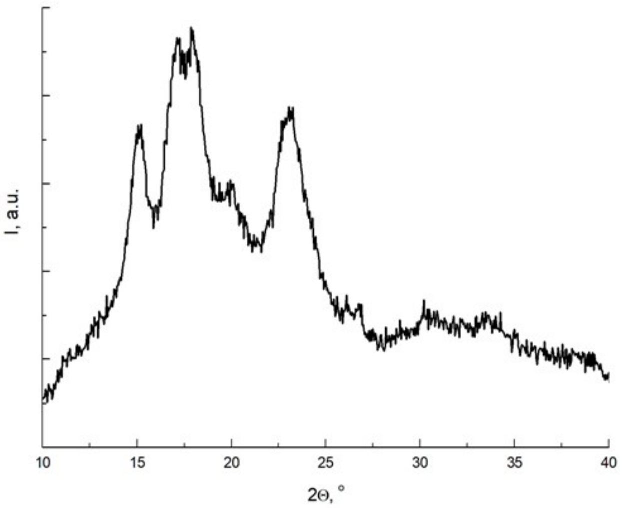


Figure 5. XRD patterns for CS powder sample.

equal to 16.7 º). When PEG and CS are introduced into the composite, intense reflections are observed in the diffraction pattern, which are most likely a superposition of two reflections: from crystalline starch (reflections 15.1; 17.6; 23.25 º) (Fig. 5) and PLA (16.7 and 19.1º), characteristic of the structure of the L-isomer [25]. Reflexes 18.7; 21.3; 23.1º corresponds to the crystalline form of PEG [26].

The degree of crystallinity of a sample of tape obtained from a charge of the original composition: 59 wt.% PLA, 20 wt.% CS, 15 wt.% PEG, 5 wt. % GMS and 1 wt.% TiONA, calculated from the ratio of the area of the amorphous halo to the entire area under the diffraction pattern of the composite, was 52%. Thus, it can be stated that the resulting composite has a semi-crystalline structure, the formation of which is facilitated by the addition of PEG to the composition.

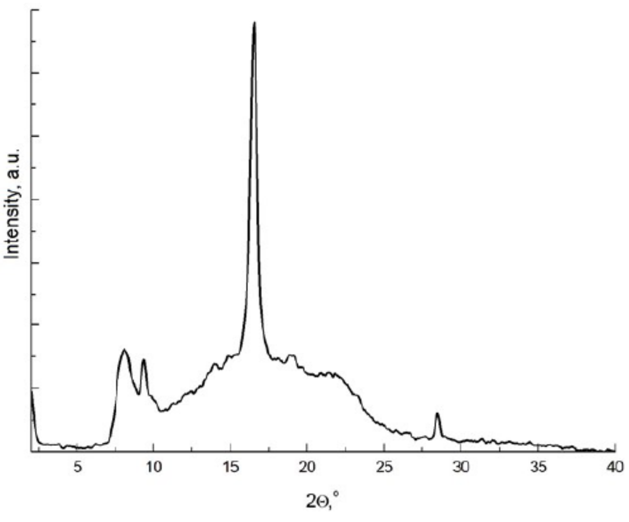


Figure 6. XRD patterns for extrusion tape with the composition: PLA (60 wt.%), flaxseed (20 wt.%), PEG (15 wt.%), GMS (5 wt.%).

To confirm the role of PEG in the crystallization of PLA, an experiment was carried out to obtain an extrusion tape with the replacement of CS with amorphous flaxseed powder (20 wt.%, average particle size is 40 μm) and the absence of a crystalline phase of titanium oxide. In this case, the diffraction pattern (Fig. 6) also shows an intense reflection corresponding to the crystalline phase of PLA (16.7 º) [27]. Reflections corresponding to starch (15.1; 17.6; 23.25 º) is not available.

The influence of PEG and CS on the structure and chemical interaction between the components of the composites was also analyzed using the IR spectroscopy method. Figure 7 shows the IR spectrum of the original polylactide (1) and the composition of PLA (59 wt.%), CS (20 wt.%), PEG (15 wt.%), GMS (5 wt.%) and TiONA (1 wt.%) (2). As seen in Figure 7, no new peaks were detected in the spectrum of the composite compared to the IR spectrum of the original PLA, which indicates the absence of chemical interaction between these components. The most intense bands in both samples are located in the region of 1200–1750 cm^{-1} and belong to the stretching vibrations of C–O bonds (C–O–C and/or C–O(H)), as well as to the stretching vibrations of C–C bonds, i.e. only polylactide is present on the surface of the sample, and the composite is thus starch coated with a film of plasticized PLA [28]. The band corresponding to the C=O stretching vibrations (1756 cm^{-1}) broadens significantly when PEG is added to PLA, which may indicate a probable interaction between the hydroxyl groups of PEG and the carbonyl groups of the main chain of PLA [29]. The band corresponding to symmetrical stretching vibrations in the CH_3 group at 2952 cm^{-1} also broadens noticeably. In addition, in the IR spectrum of the composite, the band corresponding to the stretching vibrations of bound hydroxyl groups (3200 – 3500 cm^{-1}) has a significantly greater intensity and width, which indicates the contribution of OH groups present in the PEG molecule [30].

Based on the results of biological tests in accordance with the international standard ISO16929:2013, it was found that biodegradable composites containing polylactide, starch and auxiliary components decompose in compost much faster than pure polylactide [31, 32]. When samples are placed in wet compost (relative humidity

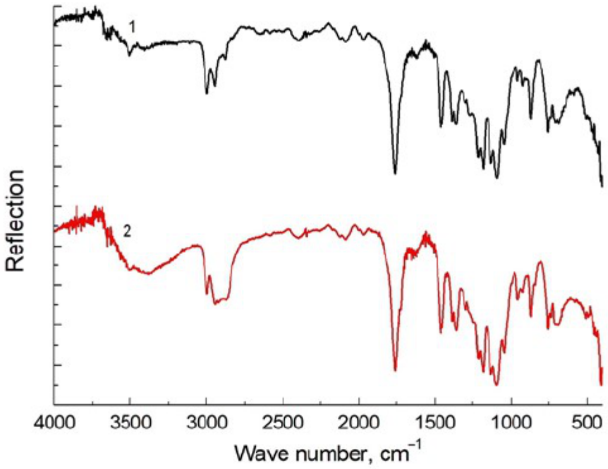


Figure 7. Comparative IR spectra of pure PLA (1) and a composite 59 wt.% PLA, 20 wt.% CS, 15 wt.% PEG, 5 wt. % GMS and 1 wt.% TiONA (2).

60%, T = 60 °C), colonies of microorganisms (bacteria, fungi) are formed on the surface of the material and microcracks are formed, allowing moisture to penetrate deep into the sample. Swelling follows and dissolution of starch particles and hydrolysis of the biodegradable polymer - polylactide, which leads to cracking of the composite material. Large and small fragments are exposed to intense microorganisms. In the process of fouling of composite fragments with soil bacteria and micromycetes, destruction of the material and a significant drop in its strength occur [33] (Fig. 8). Microbial enzymes and metabolites, together with water and chemical components of the soil, cause further biodegradation of the remaining material. Under the influence of enzymatic systems present in living organisms, polymer fragments are involved in hydrolytic and redox reactions, as a result of which the formation of new free radicals continues. Thanks to them, the macromolecules of polylactide are intensively destroyed, as a result of which its molecular weight is significantly reduced. Microfragments of polylactide with a molecular weight of about 5000 and below are absorbed by soil microorganisms with the release of CO_2, H_2O and humus, which, in turn, are a nutrient medium for soil microflora. If the composite is on the surface of the compost and UV radiation hits TiO_2 particles, additional mobile radicals are formed, which interact with the polymer chains and cause their destruction to low molecular weight fragments. The products of complete decomposition of the composite material are carbon dioxide, water, humus and minor amount of titanium dioxide. Thus, within 5–6 months, the composite material is completely destroyed, which is mixed with compost and does not emit environmentally harmful substances.

Conclusion

The morphology and structural features of biodegradable composites based on polylactide filled with corn starch, formed by the extrusion method, have been studied. It was found that the presence of the PEG-4000 plasticizer and surfactant glycerol monostearate in the initial mixture leads to the formation of a pore-free heterogeneous system in which starch particles are statistically distributed in the polylactide matrix. The concentration of starch in the composite varied from 20 to 55 wt. %. When the starch concentration increases to the limiting values, agglomeration of filler particles occurs and a sharp deterioration in the mechanical properties of the composite occurs. An XRD study of the samples showed that the original PLA actually does not have a crystalline phase, and when PEG and starch are introduced into the composite, intense reflections are observed in the diffraction pattern, which, most likely, are a superposition of two reflections - from starch and the crystalline phase of PLA. The influence of PEG

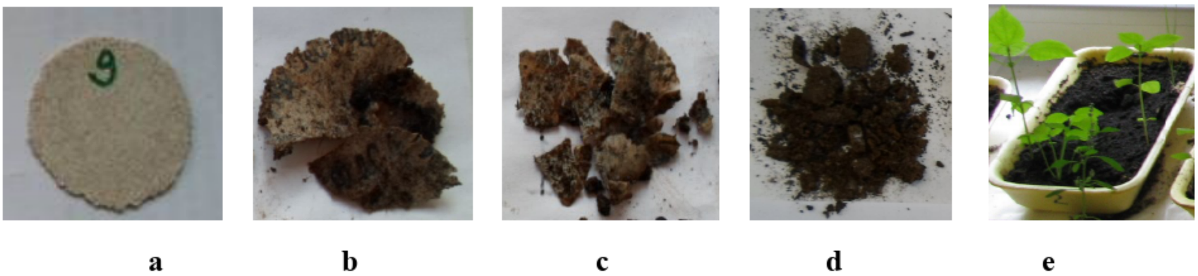


Figure 8. Stages of biodegradation of samples based on polylactide when kept in compost according to the ISO 16929:2013 standard: a – initial composite PLA (59 wt.%), CS (20 wt.%), PEG (15 wt.%), GMS (5 wt.%) and TiONA (1 wt.%); b – after 2 months; s – after 4 months; d – after 6 months; e – compost after the experiment.

and starch on the structure of the composite was analyzed using IR spectroscopy. No new bands were found in the spectrum of the composite compared to the IR spectrum of the original PLA, which indicates the absence of chemical interaction between the components. The most intense bands in both samples are located in the region of 1200–750 cm^{-1} and belong to the stretching vibrations of C–O bonds (C–O–C and/or C–O(H)), as well as to the stretching vibrations of C–C bonds, i.e. only polylactide is present on the surface of the sample, and the surface of the composite is plasticized PLA interspersed with starch particles coated with a PLA film. In addition, in the IR spectrum of the composite, the band corresponding to the stretching vibrations of bound hydroxyl groups (3200 – 3500 cm^{-1}) has a significantly greater intensity and width, which indicates an additional contribution of OH groups present in the PEG molecule. Biodegradability tests carried out in accordance with the international standard ISO 16929:2013 showed that the resulting composite materials undergo complete chemical and biological degradation in compost within 5–6 months.

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