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Biodegradable polymer materials and modifying additives: state of the art. Part II

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ABSTRACT: One of the most demanded materials on the planet is plastic, the excellent performance of which contributes to the accumulation of a significant amount of waste on its basis. In this regard, a new approach to the development of these materials hasbeen formed in scientific circles: the production of polymer composites with constant performance characteristics for a certainperiod and then capable of destruction under the influence of environmental factors. Analysis of the current state of the industry of polymeric materials shows that the most urgent is the use of such classical polymers as polyolefins and polyvinyl chloride. First of all, the optimal solution to this problem due to the lack of a suitable replacement for traditional polymers is the development of composites based on them with the use of biodegradable additives. In this case, a set of problems associated with waste disposal issolved: the decomposition period of the recycled waste is significantly reduced, the territories required for plastic waste are reduced. The paper outlines the preconditions for the emergence and further development of the field of biodegradable polymers. The main-quantitative characteristics of the production capacities of manufactured bioplastics by types, regions and industries of application-are given. Modern methods of reducing and regulating the degradation time of polymer materials are presented. The main global and domestic manufacturers of biodegradable polymers and their products are listed, as well as a list of the main manufacturers of biodegradable polymeric materials. Modern types of bioplastics based on renewable raw materials, composites with their use, aswell as modified materials from natural and synthetic polymers are listed. The main methods for determining the biodegradability of existing bioplastics are described.

KEYWORDS: biodegradation, biodegradable additives, petrochemical raw materials, polymers, plasticizers, plant sources.

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MAIN PART

Overview of Biodegradable Polymers

Biodegradable polymers are polymers that decompose in the environment under the action of microorganisms (bacteria or fungi) and physical factors (UV radiation, temperature, oxygen) [25].

Biodegradation is a biological process that changes the chemical structure of a material, resulting in natural metabolic end products.

There are two types of biodegradation of materials: aerobic and anaerobic [25].

Aerobic decomposition (composting) is the biological decomposition of organic matter PM with the consump-

tion of free oxygen or air during the life of microorganisms. The products in this case are carbon dioxide, water, minerals and biomass.

Anaerobic decomposition (biogasification) is the biological decomposition of organic substances PM in the absence of consumption of free oxygen or air during the life of microorganisms. The products in this case are methane, water, minerals and biomassa.

Classification of bioplastics by the type of raw material from which the polymer is made:

- renewable raw materials;
- non-renewable (fossil).

Another criterion for classification is the ability of PM to spontaneously degrade in the natural environment, that is, biodegradation:

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- biodegradable;

- non-biodegradable.

According to these characteristics, all plastics can be divided into four groups [26].

Group 1. Non-biodegradable plastics from fossil raw materials – all "traditional" large-scale polymers: polyethylene, polypropylene, PVC, polyethylene terephthalate, polystyrene, polybutylene terephthalate, polycarbonates, polyurethanes, etc.

Group 2. Biodegradable plastics from fossil raw materials – synthetic materials obtained by traditional methods based on petrochemical hydrocarbon raw materials, but due to their structural features are capable of biodegradation: polybutyrates (copolymers of adipic acid, dimethyl terephthalate and 1,4-butanediol (PBAT), polybutylene succinates (PBS), polyvinyl alcohol (PVAL), polycaprolactones (PCL), and polyglycolic acid (PGA) These include traditional plastics modified with depolymerization promoters (group 2a) or obtained with the introduction of copolymers unstable to hydrolysis (group 2b). It is mainly modified PET, where, for example, PBAT is used as a copolymer.

Group 3. Non-biodegradable plastics based on natural raw materials – classical polymers (biopolyethylene, bioPVC, terephthalic biopolyesters PET or PBTF), the raw material for which is biomass. These are bioethylene and biomonoethylene glycol obtained from it, as well as bio-1,4-butanediol and monoethylene glycol of direct fermentation of sugars. And also polyamide-11, which is made from vegetable oil.

Group 4. Biodegradable plastics from natural raw materials, which are divided by the method of obtaining the polymer into subgroups:

Group 4a. Biodegradable plastics from natural raw materials, in which the polymer chain is formed naturally. The tasks of their production are reduced to their isolation from biological raw materials or to modifying their structure without synthesizing the polymer chain (biopolymers based on starch, modified cellulose);

Group 4b. Biodegradable plastics from natural raw materials, in which the polymer chain is formed due to the vital activity of microorganisms in a certain environment (polyhydroxyalkanoates (PHA));

Group 4c. Biodegradable plastics from natural raw materials, in which a monomer is formed as a result of a biological process, the polymer itself is assembled using synthesis (polylactic acid (PLA)).

For biodegradable composites, it is important that all ingredients are also biodegradable and non-toxic. Thus, standards for compostable PM require testing of polymers and all additives in order to exclude their negative impact on the biosphere [27–29].

In general, the biodegradability of PM does not depend on the nature of raw materials, but is directly related to the size and chemical structure of the molecule, the presence and nature of substituents, and the supramolecular micro- and macrostructure [30-35]. To activate biodegradation, composite must contain the following fragments [36]:

1) heteroatoms;

2) biodegradable bonds (R = CH₂; R = CH-R¹; R-CH₂-OH; R-CH(OH)-R; R-CO-H; R-CO-R¹ и др.);

3) fragments of the carbon chain of less than five groups CH_2 ;

4) bulky substituents;

5) natural foods for microorganism nutrition as fillers: starch, cellulose, lactose, magnesium, urea.

There are five main ways of degradation of synthetic polymers, depending on external factors [37–38]:

1) bacterial destruction;

2) chemical destruction;

3) photodegradation by exposure to sunlight;

4) thermal degradation;

5) mechanical degradation.

Biodegradation of polymers occurs through two mechanisms: biological hydrolysis or biological oxidation [39–40].

Biological hydrolysis occurs under the action of special depolymerase enzymes for each type of hydrolysable bond. Biological oxidation can proceed without enzymes. Both destructive processes work synergistically.

The first stage of polymer damage begins with the adsorption of microorganisms on the surface. The second stage of damage proceeds depending on the chemical composition of the monomer unit of the polymer. There is either the utilization of the polymer as a source of nutrition for microorganisms, or destruction under the influence of the metabolic products of microorganisms – metabolites, which destroy the sample throughout the volume.

Each polymer is associated with microorganisms capable of initiating its depolymerization. The resulting mono- and oligomers undergo mineralization under the action of enzymes.

Bacterial destruction is characteristic of natural polymers and PMs containing natural components. Methods for bacterial decomposition of polymers using specially selected microorganisms have also been developed. This expensive process allows the destruction of petrochemical-based plastics.

Chemical degradation is carried out using aqueous solutions of chemical compounds and is especially accelerated in an alkaline environment. Initially, PM is dissolved in an aqueous solution, then complete biodegradation of the aqueous solution is carried out due to the microbial reagent.

Photodegradation. Under the influence of UV irradiation, photodegradable polymers gradually crack, then crumble into pieces, which then turn into powder. Further decomposition of PM proceeds under the ac-

tion of microorganisms. Polyolefins are especially prone to photodegradation. To increase the efficiency of the process, the following additives are added to the PM formulation[41-44]:

- sensitizers initiators of photochemical reactions (benzophenone, diphenyl sulfide);
- compounds containing variable valence metals (Cu, Fe);
- compounds which, as a result of copolymerization of compound units, lead to the formation of aldehyde or ketone groups (comonomers vinyl ketones);
- compounds that contribute to the formation of a large number of carbonyl groups that absorb UV radiation. Biodegradation of basic synthetic polymers is usually

initiated by non-biological processes (thermal, photooxidative, mechanical degradation, etc.).

The biodegradation of synthetic polymers consists of two stages: depolymerization and mineralization of residues [45]. Extracellular enzymes are capable of destroying macroceps by initiating a depolymerization reaction. Endoenzymes have the form of a cavity in the form of a "lodge" and randomly destroy internal bonds in polymers. The active center of exoenzymes has the shape of a cavity in the form of a "pocket" and sequentially break macroceps into mono- and oligomeric fragments (Fig. 5) [39–40].

Their coherent serial-parallel catalytic transformations contribute to the rapid destruction of PM [39–40].

Currently, methods for producing recombinant enzymes from genetically modified (mutant) strains of microorganisms have been developed and polyenzyme complexes with the desired set of individual components have been created.

The obtained low molecular weight fragments are further mineralized with intracellular enzymes to form final products (carbon dioxide, water, salts, methane).

Classification of additives that accelerate the decomposition of PM [46]:

 Oxo additives. All toxic properties are preserved, and the complete decomposition of small pieces is not accelerated.

- Additives that accelerate the breakdown of polymer molecular chains under the influence of sunlight, a certain air temperature, moisture and other activating factors.
- Additives that cause the release of carbon and hydrogen molecules from plastic, which are readily absorbed by bacteria and fungi.

Biodegradable plastics based on traditional polymers. To create such PMs, special additives are used during processing. Some of them make it possible to modify the period of PM biodegradation up to 1-3 years, depending on the polymer structure. There are *OXO-degradable and hydrodecomposed PM*.

The d2w additive is a catalyst for the oxidation of carbon bonds in a polymer molecule under the action of ultraviolet light, atmospheric oxygen after a certain period of time. The resulting PMs are called OXO-degradable. In this case, after the oxidation process, the growth of microorganisms on the polymer surface is observed.

Hydrodegradable PM are materials based on renewable sources of plant raw materials or synthetic plastics that contain special additives (starch) that are decomposed by a hydrolysis reaction without oxygen. For this reason, methane is released during the biodegradation of these plastics. The final decomposition products are similar to OXO-degradable PM [37]. In both cases, the surface of the oxidized polymer has hydrophilic properties, in contrast to the initial PM, and therefore is easily exposed to the action of water and bacteria. The general scheme of destruction is shown in Figure 6 [24].

Biodegradable PM based on natural polymers

In the production of biodegradable packaging, the most widespread are polysaccharides (starch, dextrins, chitosan, cellulose, wood processing waste) [47–48].

Typically, starch contains 30–40% bound moisture, which is used as the most readily available plasticizer in the production of starch-based PM. It is also known to use urea, mono-, di- and polyethylene glycols as plasticizers. Foamed starch-based PMs are used as soundproof packaging. By extrusion of mixtures of corn starch and micro-







Fig. 6. General scheme of biodegradation of polymeric materials

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crystals of cellulose and methylcellulose, PM is obtained to protect food products from weight loss and spoilage.

Mixtures of chitin and chitosan are becoming increasingly important as a basis for the production of biodegradable packaging films and textile fibers. Films based on chitosan are formed from acetic acid solutions, adjusting the solubility and swelling of the films by crosslinking chitosan with glutaraldehyde or oligomeric diepoxide.

Composite biodegradable polymers

When creating biodegradable polymer composites, as a rule, the following principle is applied: biodegradable natural polymer mixtures (starch, cellulose, chitin) or synthetic ones are added to a synthetic polymer binder [37]. Biodegradation of polyolefins in the presence of starch is a very complex process, in which various factors play an important role, including the oxidation reactions of carbon chain macromolecules [49–50].

For example, a complex of valuable characteristics inherent in chitin polysaccharide: low density, layered structure, ability to film formation, tendency to selective interaction with microorganisms, radio absorption and radioprotective properties, suggests the possibility of developing biodegradable electromagnetic screens based on its mixtures with polyethylene [51–52].

Another example, biodegradable copolymers of the product of joint polymerization of ethylene with caprolactone were prepared by graft copolymerization [53].

To protect PM from the effects of microorganisms, modification of PM properties is controlled using the following factors [43, 25]:

 by varying the structure of the material: an increase in the degree of crystallinity and orientation of polymers, crosslinking of macromolecules with the formation of network structures (the crystalline supramolecular structure of the polymer provides greater resistance to biodegradation, since the compact arrangement of structural fragments of such polymers prevents swelling in water and the penetration of enzymes into the polymer matrix);

- by varying the chemical structure: by introducing substituents into the polymer macromolecules that sterically hinder the approach of aggressive metabolites to chemically unstable bonds;
- varying the composition of the material: introducing mineral fillers capable of diffusing to the surface and creating a protective layer at the interface;
- modification of the surface of the material: the formation of an insulating layer on it with other physicochemical properties, resistant to metabolites;
- modification of the nature of the stress state of the surface layers of the material, parts, product: the creation of residual compressive stresses that prevent the facilitation of cracking of the material in the presence of metabolites;
- modification of environmental conditions in order to minimize the rates of sorption, chemical, electrochemical processes.

To protect PM from biodegradation, compounds with biocidal properties are used: phenol derivatives, quaternary ammonium compounds, salicylanilides, copper 8-hydroxyquinolate, trilan, trichloroethyl phosphate, 1-fluoro-3-bromo-4,6-dinitrobenzene, phenyl mercury salicylate, phenyl mercury phthalate, pentachlorophenol [25]. Organometallic, organochlorine, quaternary ammonium and inorganic compounds are also used as biocides. Biocides can be divided into four groups:

- inorganic (ZnO, CuSO₄, NaF, NH₄BF₄, Ca(CN)₂);
- organic (phenol derivatives, phosphorus-containing compounds, amines, etc.);



- organometallic (containing mercury, lead, tin, sodium, silver);
- polyfunctional (synthetic biologically active compounds).

It has been found that calcium carbonate as a filler reduces, and asbestos and talc increases the growth rate of microorganisms; zinc oxide in the composition of polymer composites inhibits the growth of microorganisms, titanium dioxide is inert, and iron and magnesium oxides stimulate it [25].

However, PMs that are absolutely resistant to the action of living organisms do not exist [25]. Studies have shown that materials such as neoprene and nylon are attacked by the fungi A. niger, A. Flavus and noticeably deteriorate their properties. There is no unambiguous opinion regarding a number of other polymers: caprone, polyurethane, cellulose acetate, cellulose acetate butyrate, phenol aniline formaldehyde resin, melamine formaldehyde resin.

It has been established that the products of fungi vital activity are acetic, propionic, butyric, fumaric, succinic, malic, citric, tartaric, gluconic, and oxalic acids [25]. The resulting acids, due to their aggressiveness, lead to a change in the physical and mechanical characteristics of PM and are food for the development of fungi.

However, the resistance of PM to fungi does not mean their resistance to bacteria [45, 25]. For example, shale PVC is a fungus, but is destroyed by dinitrophying and hydrocarbon-oxidizing bacteria.

Table 2

Methods for assessing the biological degradation of polymer composite materials

Method	The essence of the method	Experi- ment time	Experimental conditions	Applied cultures	Standard
Mycological test	Determination of ma- terial resistance to the effects of mold cultures	28 days	Temperature 29±2°C Humidity over 90% Illumination 200–300 lx	Mold and micro- scopic fungi	ISO 846 ASTM G 21-96 State standard 9.049-91
Bacterial test	Determination of ma- terial resistance to bac- teria cultures	_	Temperature 20°C Humidity over 58% Illumination 200–300 lx	Bacterial cells	ISO 846
Biodegradability of insoluble substances	Determination of the amount of O_2 absorbed during aerobic degradation	4 months	Anaerobic conditions, Buffer mineral medium, Solid medium	Inoculant from activated sludge or waste water	ISO 10708 OECD 301 D
Free space test at 25/50°C	Determination of the amount of CO ₂ re- leased during incuba- tion of the material	48 days	Temperature 25–50°C Humidity 60–70% Illumination 200–300 lx Anaerobic environment	Mixed population of microorganisms	ASTM D5988-96 OECD 301 A
Shturm Method	Determination of the released CO_2 in the process of material degradation	6 months	Temperature 20–25°C Illumination 200–300 lx Water solution Aerobic environment	Fungal or bacterial flora	ISO 9439 ISO 14852 DIN EN 29439 ASTM D 5209
Determination of released CO_2 (aerobic test in compost)	Determination of re- leased CO_2 in the pro- cess of material degra- dation in compost	6 months	Aerobic environment Compost based on the organic fraction of municipal solid waste	Aerobic bacteria	ISO 14855 ASTM D 6400
Determination of the degree of decomposition in compost	Determination of the degree of decomposi- tion of material in a model industrial compost	90 days	Temperature 58±2°C Air exchange Darkness or stray light Industrial compost	Inoculant, ther- mo-philic bacteria	ISO 16929 ISO 20200 BS EN 14045
Soil test	Determination of ab- sorbed O_2 or released CO_2 in the process of material degradation in natural soil of the upper layer of fields	6 months	Temperature 28±2°C Soil moisture 30±5% pH–7,5 Soil biologi- cal activity coefficient 0,65–1,5	Soil microorgan- isms	ISO 17556 DIN 53739 State standard 9.060-75



Major producers of biodegradable additives

Symphony Environmental (Great Britain) produces additives for the production of conventional plastics, biodegradable plastic products. The main product is d2w (degradable to water), used in more than 60 countries [25].

Environmental Products Inc. (EPI, Canada) produces oxo-biodegradable plastics, the developer of Totally Degradable Plastic Additives TDPA. The additive is added at a ratio of 2-3% to the total volume of the base plastic (polyethylene, polypropylene, polystyrene). Non-toxic and safe for food contact [25].

Willow Ridge Plastics (WRP, USA) – manufacturer of oxo-biodegradable additives for plastics. The additives of the company are non-toxic, safe for use in the food industry [25].

Bio-Tec Environmental (USA) manufactures oxo additives EcoPure, which are used for more than 15 polymers, are biodegradable [25].

ECM BioFilms (USA) produces additives for polystyrene, polyurethanes and PET. The additive decomposes by microorganisms [25].

Nor-X Industry AS (Norway) produces an iron-based ingredient Renatura, which is used for biodegradation of polyolefins [25].

Wells Plastics Ltd (Great Britain) releases additives and masterbatches containing prodegradants from metal ions to give the main polymer photo- and thermal decomposability, a biodegradation enhancer of the second stage, in which a reaction rate modifier is used to control the initiation and duration of oxobiodegradation[25].

P-Life Japan Inc. (Japan) produces a mixture of catalysts based on fatty acids of a special proprietary formulation of the P-Life company. Content in the base polymer (PE or PP): 0.3% to 1% [25].

TOSAF(Israel). The product OX5854 based on polysaccharides is introduced in a ratio of 2-8% of the total polymer weight. For decomposition within a month, UV radiation, high humidity and temperatures up to 90°C [54].

ADD-X BIOTECH (Switzerland). Add flex grade HE01010 FF for LDPE. When 1.5% of the additive is added, spontaneous decomposition of the LDPE film heated to 60°C is observed after 140 hours. The Addiflex brand is presented in four grades for polymers for various purposes: A-grade, The HES grade, The HEV grade, The BOPP grade [55].

Methods for assessing the biodegradability of PM

The most commonly used methods for testing the biodegradability of composite materials are shown in Table 2.

The most productive is an integrated approach to the study of PM biodegradation. To determine reliable parameters and elucidate the mechanism of the process, a comparative analysis of the results of a number of independent physicochemical, biochemical, microbiological experiments carried out in laboratory and in vivo is necessary.

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