SIST

SLOVENSKI STANDARD
SIST-TP CEN/TR 17910:2023

01-marec-2023

Biorazgradljivi polimerni materiali - Stanje standardizacije in nove možnosti

Biodegradable plastics - Status of standardization and new prospects

Bioabbaubare Kunststoffe - Stand der Normung und neue Perspektiven

Plastiques biodégradables - État de la normalisation et nouvelles perspectives

Ta slovenski standard je istoveten z: CEN/TR 17910:2022

ICS:

01.120	Standardizacija. Splošna pravila	Standardization. General rules
13.020.20	Okoljska ekonomija. Trajnostnost	Environmental economics. Sustainability
83.080.01	Polimerni materiali na splošno	Plastics in general

SIST-TP CEN/TR 17910:2023 **en,fr,de**

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER REPORT

CEN/TR 17910

December 2022

ICS 83.080.01; 13.020.20

English Version

Biodegradable plastics - Status of standardization and new prospects

Plastiques biodégradables - État de la normalisation et nouvelles perspectives

Bioabbaubare Kunststoffe - Stand der Normung und neue Perspektiven

This Technical Report was approved by CEN on 18 December 2022. It has been drawn up by the Technical Committee CEN/TC 249.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Ref. No. CEN/TR 17910:2022 E

Contents

Page

European foreword

This document (CEN/TR 17910:2022) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

Introduction

Biodegradable plastics have been developed starting from late 80s of the last century in parallel with the development of methodologies for the characterization of degradation (including biodegradation, disintegration, detection of potential ecotoxic by-products produced during biodegradation) of solid materials. The industry developed together with standardization and certification bodies a reliable governing framework needed to develop the market. After 30 years, a wide range of biodegradable products are commercially available. Standard test methods and specifications are available enabling the characterization and certification of those products.

There is an increasing interest to find out more information on the nature of biodegradable plastics and their fundamental characteristics that was fulfilled by going to the source, i.e. by directly examining the technical standards. The analysis of standards by persons who are not experts in the science of biodegradation or standardization and therefore unaware of the underlying reasons for some test schemes, has led to the direct application of such schemes in the context of communications, creating paradoxical situations. Several criticisms did surface based on the erroneous interpretation of the testing schemes. For example, many were puzzled by the 90 % mineralization pass level (rather than 100 % i.e. “complete”) required by the standard specifications to show biodegradability, ignoring that biodegradation involves biomass formation, a very basic knowledge in biochemistry and microbiology. This commingling between technical requirements and media communication created a great deal of confusion among the public and put the Industry, Standardization, and Certification under increased pressure.

CEN experts acknowledge the communication issues and therefore created the underlying document that summarizes the state of standardization and enters into the merits of the individual tests to explain the reasons for some technical solutions and the criteria adopted. This exercise also becomes a preliminary step to highlight potential gaps, the need for updating some standards, or new frontiers to be explored to complete the characterization of biodegradable plastic materials.

1 Scope

This document summarizes the state of standardization in the field of biodegradable plastics and plastics products at CEN and ISO level. It explains the underlying scientific principles of biodegradation that provide the foundations for relevant test methods and enters into the merits of the individual tests to explain and clarify the reasons for the adoption of specific solutions and criteria.

This document primarily focusses on standards adopted by CEN covering environmental biodegradation testing and relevant specifications. It also includes information on disintegration and eco-toxicity tests. A full list of the international standards considered in this document is provided in Annex A.

In a second part, this document highlights areas where standardization in this field is currently lacking and where future developments may be anticipated and useful.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 Biodegradation

Biodegradation is a term used in ecology to refer to the natural processes carried out by the decomposing microorganisms (fungi, bacteria, protozoa) that convert the organic substances produced during photosynthesis back into CO_2, water, inorganic substances and new biomass. Biodegradation and photosynthesis function in opposite directions in the ecological cycles, most notably in the carbon (biogeochemical) cycle.

Starting from atmospheric CO_2 and through the utilization of solar energy via photosynthesis, plants, algae, and all the autotrophic organisms synthesize sugars – the organic molecules that form the building blocks of the countless substances present in the biosphere. Through the food chain, the flow of substances and energy passes from plants (producers) to herbivores (primary consumers) and from those to carnivores (secondary consumers).

In the other direction, biodegradation breaks down these organic molecules into smaller intermediate constituents such as CO_2, CH_4, inorganic substances and new biomass. Ultimately, the carbon will be converted back into CO_2. Biodegradation is carried out by the decomposers which grow on dead organic matter; the solid waste of nature. The natural process of biodegradation is essential for the environment and its natural cycles that must get rid of waste and residues in order to make space for new life. Without biodegradation reactions releasing CO_2 back into the atmosphere, photosynthesis would be devoid of its key building block. Therefore, in the natural balance of the planetary ecosystem, biodegradation processes are in balance and harmony with photosynthesis process in the naturally occurring ‘circular economy’.

At the atomic level, once the carbon atoms which form part of CO_2 have reached the maximum state of oxidation they are considered as minerals and hence this final process of biodegradation is called mineralization. The complete burning of a wood log in a fireplace into CO_2, water and ashes with instant release of energy is a fast form of “mineralisation”. Microorganisms degrading the same wood log reproduce this process in a controlled way so that they can exploit the energy for living.

Biodegradation ultimately happens by means of aerobic respiration. As noted above, respiration is the opposite of photosynthesis, which is the reduction of carbon dioxide into organic carbon i.e. carbon atom bond with other carbon atoms or hydrogen. This reaction happens thanks to the solar energy.

photons

$6\ CO_2 + 6\ H_2O \rightarrow C_6H_{12}O_6 + 6O_2$

Respiration is the opposite reaction.

$C_6H_{12}O_6 + 6\ O_2 \rightarrow 6\ CO_2 + 6\ H_2O$ + energy

5 Biodegradation of polymers

Worldwide photosynthetic fixation of carbon dioxide is estimated to yield annually up to 150 X 10^9 tons of dry plant material (biomass). Almost half of this material consists of cellulose (28 % – 50 %); other major components are hemicelluloses (20 % – 30 %) and lignin (18 % – 30 %) [1].

A polymer is best described as a class of natural or synthetic substances composed of very large molecules, called macromolecules, that are multiples of simpler chemical units called monomers. Polymers make up many of the materials in living organisms, including, for example, proteins, cellulose, and nucleic acids where the polymer is produced by the cells of living organisms they are known as biopolymers. Biopolymers consist of monomeric units that are covalently bonded to form larger molecules. Cellulose is the most abundant biopolymer on earth, with plants producing about 180 billion tonnes per year globally.

The biodegradation of cellulosic biomass represents an important part of the carbon cycle within the biosphere. Microorganisms are able to provoke the complete degradation of cellulose which is ultimately converted into CO_2, water and microbial biomass under aerobic conditions. Most cellulose is degraded aerobically, but 5 % – 10 % is degraded anaerobically, and thus converted into CO_2, methane and biomass under anaerobic conditions.

Cellulose is a water-insoluble polymer and thus it cannot get into the cells and cellulolytic enzymes are by necessity secreted into the medium or bound to the outside surface of cellulolytic microorganisms.

Cellulose does not accumulate in the environment and therefore it can be considered as totally biodegradable in any environment, independently from the environmental conditions. For this reason, it is used as a reference material in biodegradation testing. The actual biodegradation rate of cellulose will be clearly affected by the environmental conditions found in that specific location.

Biopolymers are made up of macromolecules, which due to their large size, do not pass through the cell membrane and therefore cannot be absorbed directly by microorganisms [2, 3]. The first stage of biodegradation occurs outside the microorganisms and is caused by extracellular enzymes that erode the surfaces of solid materials [4]. In this extracellular phase, the main reactions are hydrolysis and oxidation. The macromolecules are split up to the constituent elements, i.e. the monomers and oligomers, which pass through the cell membrane and are metabolized becoming part of the biochemistry of the microorganisms and of the living mass ("biomass"). Low molecular weight additives do not need the depolymerisation phase to become available. The final degradation of the metabolites under aerobic conditions involves an oxidation process that requires oxygen and leads to the evolution of carbon dioxide [5].

The process of biodegradation of the (bio)-polymers in an environment can be divided into four stages (adapted from Folino 2020 [6]):

1. Firstly, the stage of bio-deterioration, in which the polymers undergo chemical, mechanical, and physical change, as a result of the microorganisms' biological activity on the surface of the material. The porosity highly influences this step.

2. The second stage is that of bio-depolymerisation, where the microbial activity causes the breaking down of polymers into oligomers and monomers.

3. The third stage describes the assimilation of the oligomers and monomers, in which the compounds are taken up by the microorganisms.

4. In the fourth stage the assimilated compounds are converted to end products, such as H_2O and biomass and the carbon mineralised into CO_2.

The depolymerisation (Stage 2) releases monomers that are assimilated by the surrounding microorganisms (the "central dogma" for biodegradation of polymers; [7]). The enzymes and microbes in the liquid phase interact with the constituents of plastics at the surface of the solid phase. The available solid/liquid interphase is thus a potential limiting factor of the depolymerisation and consequently of the overall biodegradation, including mineralization. Stage 2 is considered the limiting factor, while the subsequent assimilation of monomers by the microbes (Stage 3) is expected to be immediate [8]. After the assimilation (Stage 3), the organic substance is mineralized into CO_2 and H_2O (Stage 4). This conversion is fast in the early stages of biodegradation. The ultimate stage of biodegradation is actually the respiration process i.e. the conversion of simply organic molecules into CO_2, water and energy. Afterwards, when there are no further polymers to biodegrade, the microbes are under starvation conditions, and mineralization affects the biomass which is stored and the metabolites formed in Stage 2. This latter phase can be very long.

The processes which result in the final mineralisation of biopolymers and polymers are identical.

6 The principles of the biodegradation tests for plastics

6.1 Measuring biodegradation

The biodegradation of a plastic material is measured under specific laboratory conditions. It is generally not possible to accurately and consistently replicate stage 1. As described before, four Stages have been identified in biodegradation: in Stage 2 the plastic (the original reactant) is depolymerised into monomers and oligomers; in Stage 3 the monomers and oligomers are taken up as biomass; in Stage 4 respiration of biomass consumes O_2 and produces CO_2 (under aerobic conditions).

The measurement of reactant consumption (i.e. the plastic material) is inconclusive, because it does not prove whether the process has actually been completed or has stopped, for example, at depolymerisation (Stage 1). Quantitative measurement of biomass formation presents technical difficulties that have not been sustainably overcome to date. Therefore, all the standardized methods for determining biodegradation of plastics are based on the measurement of respiration, i.e. the conversion into CO_2 of the carbon initially present in the plastic through the use of the oxidant (O_2).

The overall reaction, which includes the four stages, can be represented as follows:

$$C_{\text{Plastic (polymer)}} + O_2 \rightarrow C_{CO2} + C_{\text{biomass}}$$

The respirometric methods differ from each other in the state (solid, liquid and sometimes biphasic), the origin of the microorganisms (the sampling environment of the microbial inoculum, for example soil, compost, etc.), the temperature and the type of measurement (consumption of the O_2 reagent or evolution of the CO_2 product). It should be remarked that all these methods measure the degree of mineralisation, rather than the degree of biodegradation, because the production of biomass is not accounted. Reliable methods for the measurement of biomass formed during a biodegradation process and the production and use of energy are still missing and thus only one product of biodegradation – CO_2 – can be monitored. For simplicity, this value is called the degree of biodegradation despite the fact that new microbial biomass growth is not considered.

The test methods are also meant to determine the so-called "Ultimate biodegradation" i.e. the level of biodegradation achieved when the test compound is totally utilized by microorganisms resulting in the production of carbon dioxide, water, mineral salts (if elements present in original polymer) and new microbial cellular constituents (biomass). The outcome of these test methods is the percentage of biodegradation. Percentage of biodegradation is a percentage yield i.e. it is the actual CO_2 produced compared to the maximum possible or theoretical CO_2 ($ThCO_2$) that can be produced in case of total oxidation of the carbon present in the test material.

It is important to understand the difference between the potential of a material to undergo biodegradation (biodegradability) and the rate at which that biodegradation occurs under given conditions. The rate of biodegradation of any material known to have a chemical structure which enables it to undergo complete mineralisation is limited by the environment. For example if we consider peat bogs or deep ocean sediments then the time to biodegrade cellulose is in the centuries whereas the rate will differ again between fertile tropical soils compared to a desert. In all of these environments, the fact remains that cellulose is intrinsically biodegradable, what changes depending on the environmental conditions is the rate at which biodegradation occurs. Due to the fact that cellulose is intrinsically biodegradable, cellulose is used as a reference material in all of the biodegradation tests which have been established for plastics including packaging.

During the biodegradation of a product three stages occur (example: see Figure 1):

1. lag phase:

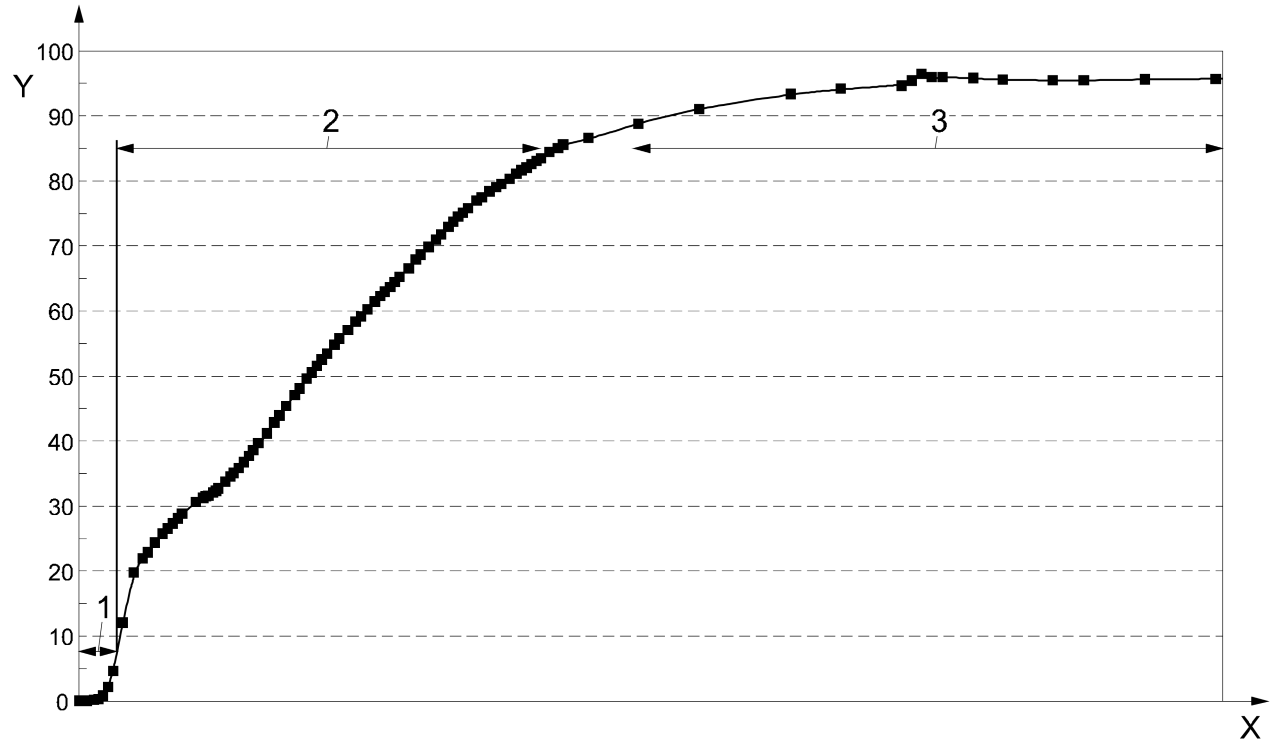
   it is the time, measured in days, from the start of a test until adaptation and/or selection of the degrading microorganisms is achieved and the degree of biodegradation of a chemical compound or organic matter has increased to about 10 % of the maximum level of biodegradation (source: ISO 15985:2014)

2. biodegradation phase:

   it is the time, measured in days, from the end of the lag phase of a test until about 90 % of the maximum level of biodegradation has been reached (source: ISO 15985:2014)

3. plateau phase:

   it is the time, measured in days, from the end of the biodegradation phase until the end of a test (ISO 14855-1:2012)



Key

X Time

Y Biodegradation (%)

1 Lag phase

2 Biodegradation phase

3 Plateau phase

Figure 1 — Example of the different stages of biodegradation.

Tests to assess the biodegradation of chemicals are long established. The most widely applied test methods come from the 1992 OECD 301 series where “ready biodegradability” is assessed. The principle of these tests is not to replicate every environment but to provide consistency and replicability of the test.

6.2 Reliability and replication

For the purposes of standardization and regulation, it is an absolute necessity that performance tests are both reliable and replicable, not just as a test within a single laboratory but also between laboratories anywhere in the world. For this reason, several of the ISO biodegradation test methods (see Clause 7 below) have been subjected to round robin assessments. The following Table 1 shows some information:

Table 1 — Test methods and associated published round robin tests

Test method	References
ISO 14852:2021 Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium — Method by analysis of evolved carbon dioxide	Muller et al., 1992 [9]
ISO 14855-1:2012 Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions — Method by analysis of evolved carbon dioxide — Part 1: General method	Pagga et al. 1996 [10]
ISO 17556:2019 Plastics — Determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved	Annex G of ISO 17556:2019 [13]
ISO 19679:2020 Plastics — Determination of aerobic biodegradation of non-floating plastic materials in a seawater/sediment interface — Method by analysis of evolved carbon dioxide and ISO 18830:2016 Plastics — Determination of aerobic biodegradation of non-floating plastic materials in a seawater/sandy sediment interface — Method by measuring the oxygen demand in closed respirometer	Tosin M. and Degli Innocenti F. 2015 [11]

In biology, there is a general classification of organisms, thermophiles are those which operate at higher temperatures (those between 46 °C and 75 °C), mesophiles which operate in a lower range (45 degrees down to 15 °C) and psychrophiles which operate below 15 °C. Organisms which exist in a temperature zone will always function, what will change is the rate of activity which is also related to the amount of available 'food'. For the purposes of consistency across test regimes, thermophilic biodegradation testing occurs at 58 degrees ± 2 and mesophilic biodegradation testing occurs between 20 °C and 28 °C ± 2. It must be remembered that the purpose of the tests are to demonstrate the fundamental ability of microbes which exist in a temperature zone to biodegrade the material under examination.

As stated above, it is not the rate of biodegradation which determines whether or not something is biodegradable, a material or chemical is either biodegradable or not biodegradable e.g. polyethylene is not biodegradable. It is known that when milled to nano-scale, the biodegradation rate of certain plastics is extremely fast since the surface area available for enzymatic attack is very high and the microbes are able to consume at a high rate. As stated above, when the biomass is form of a tree the rate of biodegradation is very slow. Therefore, in order to enable a comparison between the biodegradation performance of a plastic material and the reference material, the particle size needs to be the same as the reference/known biodegradable material – cellulose. For this reason, plastics are milled to the same particle size as found in micro-crystalline cellulose powder which is the preferred reference material.

The principles of biodegradation given in Clause 5 give four distinct stages all of which influence the rate and ability of a (bio)-polymer to biodegrade. Of these, Stage 1 – bio-deterioration is not accounted for in ISO test methods. Test methods do exist to consider the exposure of light, heat and water on plastic materials. These high intensity methods have long been applied to applications where durability and safety is paramount e.g. structural and construction plastics and colour fading. In the case of testing plastics which may enter the environment these treatments are not considered appropriate as it is impossible to predict what level of exposure to light, heat or water a plastic may undergo.

Currently, neither the ISO/TC 61 or CEN/TC 249 for plastics foresee pre-treatment which seek to mimic environmental exposure prior to the assessment of biodegradation within their standards.