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Plastics — Disintegration field test of plastics under water environmental conditions

Plastiques — Essai sur site de désintégration des plastiques dans les conditions d'un environnement aquatique

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Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 14, *Environmental aspects*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Marine plastic litter has been continuously increasing. Plastic waste released in the environment eventually reaches the rivers, the lakes and the sea, floats on the water surface, and some of them sink to the riverbed, lakebed and seabed. Floating plastic litter has a significant negative impact on aquatic life due to accidental ingestion or entangling. To minimize the risk of plastic waste, it is recommended that plastics become low molecular weight by microbial action, lose strength, and disintegrate. Biodegradable plastics are designed to disintegrate in this way. The biodegradation of plastics in the marine environment is defined in laboratory tests, such as ISO 18830, ISO 19679, ISO 22404, ISO 23977-1 and ISO 23977-2. ISO 23832 describes a test method for the determination of the degradation rate and disintegration degree. However, these test methods do not measure disintegration under environmental conditions. ISO 22766 applies to disintegration only in the sandy sublittoral and the sandy eulittoral zone.

This document provides a test method for determining the disintegration degree of biodegradable plastic materials floating on the water surface. It can only apply if the biodegradation test according to the ISO standard, for example ISO 18830, ISO 19679, ISO 23977-1 and ISO 23977-2, has been carried out and has given a positive result. This test method is simplified so that it can be conducted in various coastal area. The disintegration is thought to be due to combination of bio-action, non-biochemical action, and physical degradation, but it is not specified by this method.

Plastics — Disintegration field test of plastics under water environmental conditions

1 Scope

This document specifies test methods for the determination of the degree of disintegration of plastic materials floating in water.

NOTE The disintegration test is a field test performed under natural environmental conditions in a part of the sublittoral zone, the shores of lakes or rivers, and the test samples are immersed to a depth between 1,5 m to 3 m from water surface.

This document specifies the general requirements of the apparatus and the procedures for using the test methods described.

This document is not suitable for the assessment of disintegration caused by heat or light exposure.

The described field test is a disintegration test and not a biodegradation test. Therefore, it cannot be used for demonstrating biodegradation or for making unqualified claims such as “biodegradable in marine environment” and similar.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4591, *Plastics — Film and sheeting — Determination of average thickness of a sample, and average thickness and yield of a roll, by gravimetric techniques (gravimetric thickness)*

ISO 4593, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological 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/

3.1
biodegradation
degradation (3.2) caused by biological activity, especially by enzymatic action, leading to a significant change in the chemical structure of a material

[SOURCE: ISO 472:2013, 2.1680]

3.2
degradation
irreversible process leading to a significant change in the structure of a material, typically characterized by a change of properties (e.g. integrity, molecular mass or structure, mechanical strength) and/or by fragmentation, affected by environmental conditions, proceeding over a period of time and comprising one or more steps

[SOURCE: ISO 472:2013, 2.262]

3.3
disintegration
physical breakdown of a material into small fragments

[SOURCE: ISO 472:2013, 2.1757]

3.4
sublittoral zone
coastal seafloor that is permanently immersed and extends from the low-water line to the continental shelf edge at 200 m water depth

Note 1 to entry: The seafloor can consist of solid rock, or fragments that form sediments of different particle size, from coarse blocks and pebbles, to permeable sands, silt and clay. Sediments can form from fragmented rock or consist of fragments of biogenic origin (algae, shells, coral, etc.), or be a mixture of these compounds.

[SOURCE: ISO 22766:2020, 3.5]

3.5
dissolved organic carbon
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part of the organic carbon in water which cannot be removed by specified phase separation methods, for example by centrifugation at 40 000 ms^{-2} for 15 min or by membranes with pores of 0,2 µm to 0,45 µm diameter

[SOURCE: ISO 22404:2019, 3.4]

4 Principle

The disintegration test is a field test performed under natural environmental conditions in the sublittoral zones especially in the coast, the shores of lakes or rivers, and the test samples are immersed to a depth between 1,5 m to 3 m from water surface. This test is a short-term immersion method with the goal to reduce the probability of coastal microorganisms inhibiting microbial surface contact to the test material or damaging the sample as it was observed for long-term immersion tests. Therefore, the sample used in this test is preferably a film, a fibre, or the like, and is not suitable for a film so thin that it disappears quickly. The sample is used uncovered or covered with a mesh or wrapped in a nonwoven fabric and put in a specially designed perforated plastic container which is immersed in the sea and which allows the samples to move freely. The degree of disintegration of the sample is determined by the weight loss or the film thickness reduction (µm). The film thickness decrease can be obtained by multiplying the film thickness before immersion by the weight loss rate (%). When films of different thicknesses are immersed for the same period, the apparent weight loss rate is greater for thin films because in principle both thick and thin films are disintegrated from the surface to the same depth. On the other hand, the film thickness decrease is essentially constant for both thick and thin films, therefore, it is preferred to use the film thickness decrease rate as an index of disintegration in case the sample films are of different thickness. As the disintegration progresses, it becomes impractical to collect all the fragmented samples that causes variation, therefore, the test condition needs to be adjusted to maintain the weight retention ratio of 50 % or more to prevent fragmentation of the test sample in order to obtain reliable data. In the case of weight retention of 50 % or less, further fragmentation occurs during the cleaning process because the film strength is greatly reduced. Since it is impractical to completely collect these fragments, it is recommended to recover them before they fragmentate too much. In this way, a reliable weight reduction rate or film thickness decrease rate can be obtained. In order to recover samples with a weight retention rate of 50 % or more, it is efficient and recommended to immerse samples with different film thicknesses. Further, since the disintegration

degree varies greatly depending on the immersion sea location (e.g. sea temperature and the number of microorganisms), it is also recommended to take samples multiple times with different periods so that the weight retention rate remains at 50 % or more, and then calculate the weight loss rate from these samples to evaluate the degree of disintegration. In addition, when immersion is performed for the purpose of collecting data on physical properties, strength, or shape changes after immersion, observing the progress of disintegration, comparing data between samples, etc., immersion may be continued until significant disintegration occurs. The immersion period is preferably within 3 months. The immersion for a longer period is possible if the disintegration rate is slow. If there is a large amount of sludge or marine organisms in the container, it is advisable to remove samples at appropriate intervals, rinse lightly, and then transfer them to a new container to continue the test.

5 Test procedure

5.1 Test material

Use the test material preferably in the form of film in an identical form (e.g. shape, thickness) as for the intended final use. The thickness of the film shall be either determined in accordance with ISO 4591 or ISO 4593.

Other forms than films, for instance articles such as fibres, nonwoven fabrics, or foams, can also be tested if test procedure and test evaluation are in accordance with this document.

5.2 Reference material

A poly(3-hydroxybutyrate-co-hexanoate) (PHBH) film[1)] and/or a polycaprolactone (PCL) film1) of 50 µm to 500 µm thickness and/or ashless cellulose filters shall be used as a positive control as these materials are thought to be biodegradable. Low-density polyethylene (LD-PE), high-density polyethylene (HD-PE), polypropylene, polystyrene and polyvinylchloride are thought to be non-biodegradable and these film of 20 µm to 100 µm thickness shall be used as a negative control. Use pellets to blow or to extrude a film, or dissolve pellets in a solvent and cast to make a film of desired thickness.

5.3 Preparation of test and reference materials

Test samples shall not be subjected to conditions or procedures, such as a pretreatment by heat and/or an exposure to radiation, designed to accelerate disintegration prior to testing according to this document.

A plastic material preferably in the form of a film having a thickness of 100 µm, the same thickness as the positive control, and at least two other film thickness, for example, 35 µm and 200 µm, is cut into pieces of 30 mm × 40 mm in size.

NOTE By using a sample with three thicknesses of 35 µm, 100 µm, and 200 µm, even if the 35 µm sample is lost due to more collapse than expected, the 100 µm and 200 µm samples can be recovered and the test will be completed.

In the case of immersion tests using thin films, it might be difficult to distinguish whether the film has completely disintegrated or whether only a portion of it has disintegrated and flowed out. By using a thick film and calculating film thickness decrease rate from the weight loss and initial thickness, the disintegration behaviour of a thin film due to immersion can be determined. Record the weight and thickness of each cut film and take a photograph of the film. The film can be used as it is for the test, or can be covered with non-biodegradable plastic mesh of 40 mm × 50 mm in size with 1 mm × 1 mm mesh size on both sides. Typical non-biodegradable meshes are made of polyamide, polyethylene, or polypropylene. If the test sample is a fibrous material or a fragile material and is easily lost through the mesh, it may be wrapped in a nonwoven fabric (see A.1).

1) PHBH-pellets from KANEKA (https://www.kaneka.co.jp/en/solutions/phbh/) and PCL-pellets (Mn 80,000) from Sigma-Aldrich(https://www.sigmaaldrich.com/US/en/product/aldrich/440744) are examples of a suitable reference material. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the suppliers named. Equivalent products may be used if they can be shown to lead to the same results.

Figure 1 shows, from left to right, a film as it is, a film covered with non-biodegradable plastic mesh, and a fibrous material wrapped in a nonwoven fabric.

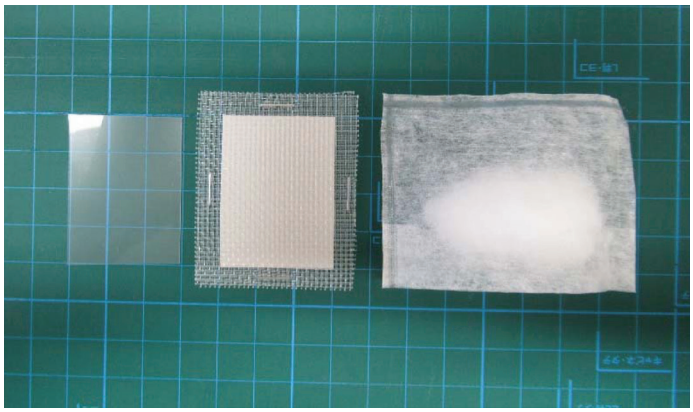


Figure 1 — Test samples

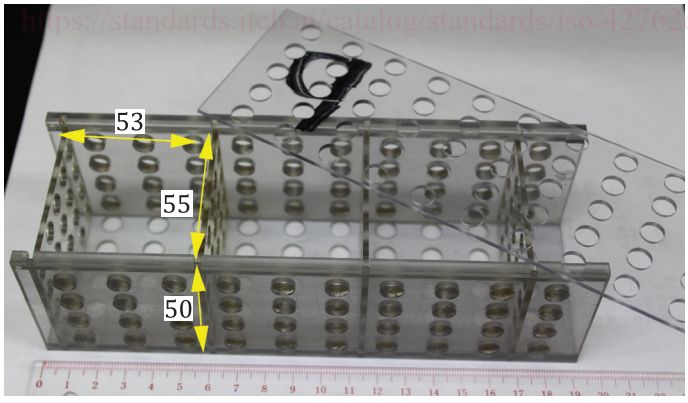
The film samples thus prepared are put in a non-biodegradable plastic container having a perforated or mesh structure on each surface with an opening rate of over 25 % to allow ambient water flows freely from all directions. The container has, for example, three rooms (see Figure 2) and each room hold one sample, and the largest cross-section of the room is more than twice the film area to allow the film moves freely in the room.

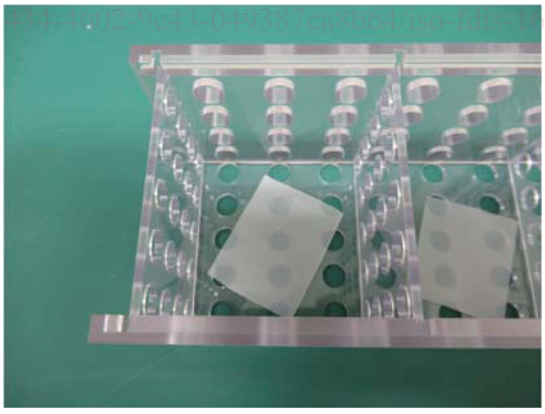
Example of the container

Outer size: 190 × 65 × 55 mm — Inner size: (53 × 55 × 50) mm × 3 rooms

Pore diameter: 8 mm — Opening ratio: 26 %

Largest cross section of a room: 40 cm^2 — Sample area: 12 cm^2





NOTE Film sample: 30 mm × 40 mm

Figure 2 — Perforated plastic container with 3 rooms

5.4 Number of replicates

Provide a sufficient number of samples prepared according to 5.3, at least:

— three replicates of each test material;