
Textiles — Test method for accelerated hydrolysis of textile materials and biodegradation under controlled composting conditions of the resulting hydrolysate

Textiles — Méthode d'essai pour hydrolyse accélérée des matières textiles et la biodégradation dans des conditions de compostage contrôlées de l'hydrolysates résultant

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

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

Textile fibres can be classified into natural fibres and man-made fibres according to ISO/TR 11827. Some of man-made fibres manufactured from organic materials are biodegradable and can be divided into three major categories in relation to their origin, i.e. natural material base, biology base and petroleum base. The representative bio-based, man-made biodegradable fibre is polylactide and petroleum-based, man-made biodegradable fibres are manufactured from polyethylene terephthalate succinate, polycaprolactone, polypropylene carbonate, polybutylene succinate or copolymer using them.

The biodegradation of petroleum-based fibres is relatively slow compared to biology-based or natural fibres due to the chemical structure. In addition, the rate of biodegradation of textile materials such as fibres and yarns can also be affected negatively by high molecular weight, degree of crystallinity and orientation occurred during the spinning. Although some standards refer to the instrument analysis, such as gas chromatograph or infrared analysis, the process and calculation method are not standardized. Therefore, it is difficult to determine the biodegradation of petroleum-based textile materials using the existing standards available for natural fibres, biology-based fibres or plastic materials used for packaging.

To overcome these difficulties, the new test method is proposed by a combination of accelerated hydrolysis and biodegradation using instrument analysis for analysis of evolved carbon dioxide.

Under the composting of textile materials both mechanisms, abiotic and biotic processes, operate together and the microorganisms eventually remove the hydrolysate in a synergistic process. It is difficult and time consuming to reproduce this in the laboratory. For convenience, the accelerated hydrolysis, which is an abiotic process, should be carried out followed by biodegradation subsequently. The rate and extent of molecular weight loss is measured as indicative of losses in physical properties from accelerated hydrolysis and then the biodegradability of hydrolysate is estimated by direct measurement of evolved carbon dioxide with a gas chromatograph.

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Textiles — Test method for accelerated hydrolysis of textile materials and biodegradation under controlled composting conditions of the resulting hydrolysate

1 Scope

This document specifies a test method for the determination of the biodegradability of the hydrolysate of textile materials obtained after accelerated hydrolysis under controlled composting conditions by measurement of the amount of evolved carbon dioxide with a gas chromatography.

This test method can be applied to petroleum-based man-made biodegradable textile materials which are manufactured from polyethylene terephthalate succinate, polycaprolactone, polypropylene carbonate, polybutylene succinate or copolymer using them.

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 1628-1, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1: General principles*

ISO 13885-1, *Binders for paints and varnishes — Gel permeation chromatography (GPC) — Part 1: Tetrahydrofuran (THF) as eluent*

ISO 14855-1, *Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions — Method by analysis of evolved carbon dioxide — Part 1: General method*

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 <http://www.electropedia.org/>

3.1

compost

organic soil conditioner obtained by biodegradation of a mixture principally consisting of various vegetable residues, occasionally with other organic material and having a limited mineral content

3.2

composting

aerobic process designed to produce compost

3.3

total dry solids

amount of solids obtained by taking a known volume of test material or compost and drying at about to constant mass

3.4

volatile solids

amount of solids obtained by subtracting the residue of a known volume of test material or compost after incineration at about from the total dry solids of the same sample

Note 1 to entry: The volatile-solids content is an indication of the amount of organic matter present.

3.5

abiotic process

process without the action of living organisms

3.6

biotic process

process through the actions of living organisms

3.7

hydrolysis

degradation identified as resulting from hydrolytic cleavage of macromolecules

3.8

accelerated hydrolysis

hydrolysis under high temperature and humidity

3.9

hydrolysate

product of hydrolysis

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3.10

theoretical amount of evolved carbon dioxide

ThCO₂

maximum theoretical amount of carbon dioxide evolved after completely oxidizing a chemical compound, calculated from the molecular formula and expressed as milligrams of carbon dioxide evolved per milligram or gram of test compound

3.11

maximum level of biodegradation

degree of biodegradation, measured as a percentage, of a chemical compound or organic matter in a test, above which no further biodegradation takes place during the test

3.12

plateau phase

time, measured in days, from the end of the biodegradation phase until the end of the test

4 Principle

WARNING — Any claim on biodegradation or (industrial) compostability of the textile material is not allowed and considered misleading. For claims on biodegradation under controlled composting conditions, testing shall be carried out in accordance with ISO 14855-1.

The test method determines the ultimate biodegradability of test material after accelerated hydrolysis under conditions simulating an intensive aerobic composting process. It aims to determine the ultimate biodegradability of the hydrolysate by using a small-scale reactor. The degradation rate is periodically measured by determining the amount of the evolved carbon dioxide using gas chromatography.

Firstly, the test material is hydrolysed under the constant temperature and humidity until the substantial loss of molecular weight in order to initiate the biodegradation process shortly.

During the aerobic biodegradation of the hydrolysate, carbon dioxide, water, mineral salts and new microbial cellular constituents (biomass) are the ultimate biodegradation products. The carbon dioxide produced is continuously monitored, or measured at regular intervals, in test and blank vessels to

determine the cumulative carbon dioxide production. The percentage biodegradation is given by the ratio of the carbon dioxide produced from the test material to the maximum theoretical amount of carbon dioxide that can be produced from the test material. The maximum theoretical amount of carbon dioxide produced is calculated from the measured total organic carbon (TOC) content.

5 Reference material

5.1 TLC (thin-layer chromatography) grade cellulose used with a particle size of less than 20 µm as the positive-control reference material.

5.2 For the calibration of gas chromatograph, preferably internal standards should be selected for the CO₂/N₂ mixture.

NOTE Examples of suitable internal standards are:

- CO₂(CAS No. 124-38-9);
- N₂(CAS No. 7727-37-9).

6 Apparatus

6.1 Environmental chamber for hydrolysis, to accelerate the hydrolysis of textile materials. The chamber shall be able to control relative humidity to within ±5 % when the set point temperature has an operational tolerance of ±1 °C.

6.2 Gel permeation chromatography (GPC), in accordance with ISO 13885-1 to measure the molecular weight distribution of a polymer.

It is essential that all components which come into contact with the eluent or the sample solution are resistant to them and do not exhibit absorption or memory effects in any form. The eluent varies depending on polymer. The individual components of the GPC apparatus, which in the case used THF as eluent, shall be linked with stainless-steel or titanium capillary tube.

6.3 Composting vessels, in accordance with ISO 14855-1.

Use bottles or columns that ensure a supply of water-saturated, carbon-dioxide-free air to the contents. A suitable volume is 1 l to 5 l. If the loss in mass of the test material is to be determined, weigh each composting vessel empty before starting the test.

6.4 Air-supply system, capable of supplying each composting vessel with dry or water-saturated, if required carbon-dioxide-free, air at a pre-set flow rate which shall be high enough to provide truly aerobic conditions during the test.

6.5 System for the determination of carbon dioxide, designed to determine carbon dioxide directly with a gas chromatograph.

The carbon dioxide in the exhaust air is measured continuously, and exact control or measurement of the air-flow rate is required. Depending on the measurement instrument, it may be necessary to remove water from the air, for example by cooling. If several composting vessels are connected up to a single measuring instrument, a suitable gas switch may be required. Take care to analyse the concentration of carbon dioxide on a sufficiently frequent basis in order to produce a reliable cumulative carbon dioxide production over the course of the test (for example, every 3 h to 9 h).

6.6 Gas chromatograph, provided with a flame ionization detector (FID) to analyse the concentration of carbon dioxide. An example of apparatus is given in [Annex A](#) for additional information.