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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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Foreword

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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, polybuthylene 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.

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, polybuthylene 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

3.10
theoretical amount of evolved carbon dioxide
$ThCO_2$
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 CO_2/N_2 mixture.

NOTE Examples of suitable internal standards are:

— CO_2(CAS No. 124–38–9);

— N_2(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.

6.7 Gas-tight tubes, to connect the composting vessels with the air supply and the carbon dioxide measurement system.

6.8 pH-meter.

6.9 Analytical equipment, for the determination of dry solids (at 105 °C), volatile solids (at 550 °C) and total organic carbon (TOC), for elemental analysis of the test material and, if required, for the determination of dissolved inorganic carbon (DIC).

7 Test procedure

7.1 Preparation of test material

Select requirements for specimens either from the product or based on the anticipated product. Normally, a minimum of 200 g is required for the hydrolysis and biodegradation tests. The test material should be used as small pieces of fibres, yarns, fabrics or other textile products. The maximum surface area of any individual piece of test specimen shall be about 2 cm × 2 cm.

7.2 Preparation of the inoculum

The compost inoculum should be well-aerated compost coming from the organic fraction of municipal solid waste and sieved on a screen of <10 mm. The age of the compost should preferably be between 2 months and 4 months. If such compost is not available, compost from plants treating green or farmyard waste or mixtures of green waste and municipal solid waste may be used. It is recommended that the compost inoculum produces between 50 mg and 150 mg of carbon dioxide per gram of volatile solids over the first 10 days of the test, and has an ash content of less than 70 % and a pH between 7 and 9. Total dry solids should be between 50 % and 55 %.

The compost inoculum should be as free from larger inert materials (glass, stones, metals, etc.) as possible. These items should be removed manually as much as possible to produce a homogeneous compost inoculum.

It is recommended to use compost of sufficient porosity to enable conditions to be as aerobic as possible. Addition of structural material, such as small wood particles, or persistent or poorly biodegradable inert material may prevent the compost from sticking together and clogging during the test.

7.3 Accelerated hydrolysis test (abiotic test)

Expose the test specimens in the environmental chamber. Set the temperature and humidity at which chemical decomposition becomes significant. Example of accelerated hydrolysis test results is given in Annex B for additional information.

The example of accelerated test condition is 90 °C and 95 % of relative humidity for fibres and yarns in the case of polyethylene terephthalate succinate (PETs).

At intervals appropriate to the test specimen (chosen by trial and error), measure the weight-average molecular weight using GPC according to ISO 13885-1 or intrinsic viscosity according to ISO 1628-1. Expose the test specimens until the weight-average molecular weight below 1 000 or below intrinsic viscosity 0,1. The test conditions of hydrolysis such as temperature, humidity, total exposure time, are to be described in the test report.

7.4 Biodegradation test (biotic test) of hydrolysate

Test the biodegradation of the hydrolysate according to the procedures of ISO 14855-1. The carbon dioxide in the exhaust air should be measured directly with a continuous gas chromatograph. In this case, the concentration of carbon dioxide measured is usually expressed in mg/kg or µg/kg. Therefore,

the mg/kg (or µg/kg) shall be converted to amount of carbon dioxide evolved in grams to calculate the degree of biodegradation.

7.5 Gas chromatographic analysis

Calculate, using linear regression of peak height over concentration, a straight line for the range of the complete series of calibration CO_2/N_2 mix gas. Determine the deviations between the measured values and the straight line. When the deviations are less than 5 %, assume linearity to exist for the complete range. When these deviations are more than 5 %, reduce the range by deletion of the measured value of the highest concentration and again calculate a straight line by linear regression and check.

Record the gas chromatogram of the working standard.

Determine, on the basis of this chromatogram, the relative retention times of carbon dioxide to the standard.

Identify the peaks of the standards by using the absolute retention times. Determine, for all the other relevant peaks in the gas chromatograms, the relative retention times with respect to the standards.

8 Calculation and expression of results

8.1 Amount of carbon dioxide in grams

The amount of carbon dioxide evolved should be calculated from the concentration determined by gas chromatograph by the following process.

a) Record the flow rate of air Q (m^3/s) in the test system.

b) Find the sampling volume V_0(m^3) (usually volume of syringe).

c) Record the concentration measurement C (mg/kg or µg/kg) indicated by gas chromatograph spectrum.

d) Calculate the amount of CO_2 in the sampling volume according to Formula (1):

$$V(m^3) = C \times V_0 \quad (1)$$

e) Calculate the mole of CO_2 in the sampling volume according to Formula (2):

$$N(mol) = V/V_m \quad (2)$$

where V_m is a molar volume of an ideal gas at 1 atmosphere of pressure.

f) Calculate the mass of CO_2 in the sampling volume according to Formula (3):

$$m_s(g) = N \times 44\ g/mol \quad (3)$$

g) Find the volume of air flowing through the system during the sampling interval, using Formula (4):

$$V_{air}(m^3) = Q \times \Delta t \quad (4)$$

where Δt (s) is the time interval of sampling.

h) Calculate the ratio of sampling volume to volume of air according to Formula (5):

$$R = V_0/V_{air} \quad (5)$$

where R is also the ratio of CO_2 sampled to total CO_2 emitted during the time interval.

i) Calculate total mass of CO_2 emitted during sampling interval, $m(g) = m_s/R$

Therefore, the total mass of CO_2 emitted during sampling interval should be described as shown in Formula (6):

$$m = [(Q \cdot \Delta t)/V_m] \times [(C_1 + C_2)/2] \times 44 \quad (6)$$

where

m is the total mass of CO_2 emitted during sampling interval (g);

Q is the flow rate of air (m^3/s);

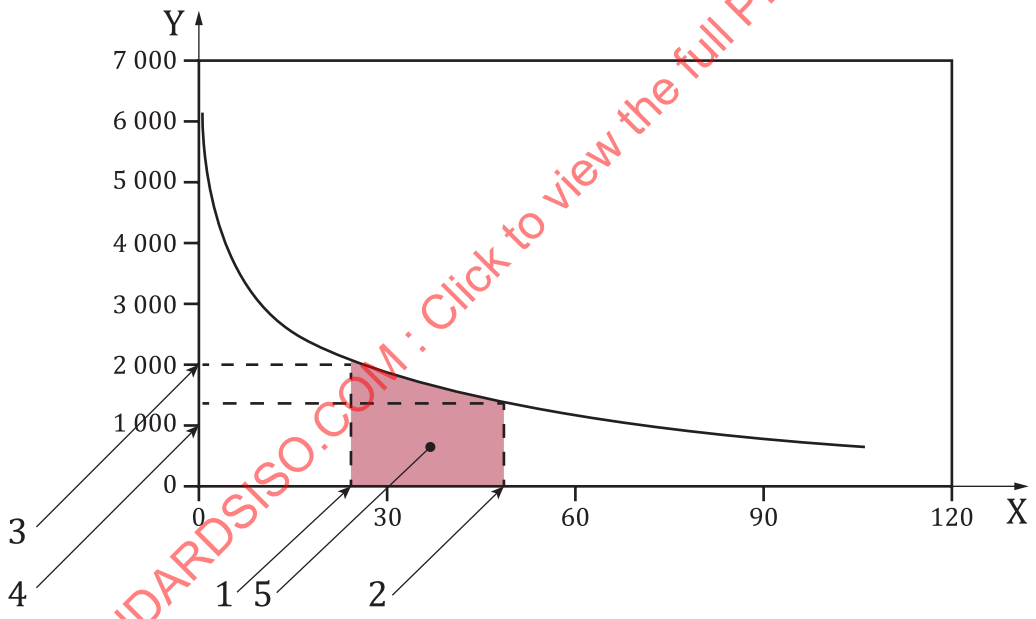
Δt is the interval of sampling (= $t_2 - t_1$) (s);

V_m is the molar volume (m^3/mol);

C_1, C_2 are the concentration measured by gas chromatograph at t_1 and t_2 (mg/kg);

44 is the molecular mass of carbon dioxide (g/mol).

NOTE Figure 1 shows a conceptual diagram to calculate the accumulative concentration measured by gas chromatograph at t_1 and t_2 (mg/kg).



Key

X time (day)
Y concentration of CO_2 (mg/kg)
1 t_1
2 t_2
3 C_1
4 C_2
5 accumulative concentration of CO_2 (mg/kg)

Figure 1 — Calculation of the accumulative concentration

8.2 Percentage of biodegradation

According to ISO 14855-1, the degree of biodegradation of the test material is calculated from the cumulative amounts of carbon dioxide released. In this case, the biodegradability between different test results cannot be compared directly, due to the biodegradability of the reference materials are different for each test. Therefore, calculate the relative biodegradability from the ratio of biodegradation of test material and reference material using Formula (7).

$$D_{rel} = \left(D_t \ / \ D_{ref} \right) \times 100 \tag{7}$$

where

D_{rel} is the relative biodegradability (%);

D_t is the degree of biodegradation of the test material (%);

D_{ref} is the degree of biodegradation of the reference material (%).

8.3 Validity of results

The test is considered as valid if:

a) the degree of biodegradability of the reference material is more than 70 % after 45 days;

b) the difference between the percentage biodegradation of the reference material in the different vessels is less than 20 % at the end of the test;

c) the inoculum in the blank has produced more than 50 mg but less than 150 mg of carbon dioxide per gram of volatile solids (mean value) after 10 days of incubation.

9 Test report

The test report shall include the following information:

a) a reference this document, i.e. ISO 21701:2019;

b) identification of the sample;

c) for accelerated hydrolysis tests:

— the set points of temperature and humidity;

— the total exposure time to which the specimen has been subjected; and

— the weight-average molecular weight or intrinsic viscosity at terminated time.

d) for biodegradation tests:

— the average flow rate of air Q (m^3/s) and interval of sampling Δt (s);

— the incubation period;

— the degree of biodegradation of test material and reference material; and

— the relative biodegradability.

e) any deviation from the specified procedure in this document.

Annex A
(informative)

Example of apparatus

A.1 Apparatus for thermal desorption, having the following characteristics:

— a primary desorption oven with adjustable desorption temperature up to 250 °C and adjustable desorption time;

— a cold trap/secondary desorption oven;

— a connecting tube to the gas chromatograph, with adjustable heating up to 150 °C;

— carrier gas flow rate adjustable up to 40 ml/min.

NOTE Instruments for thermal desorption are commercially available.

A.2 Example of the analysis condition of gas chromatograph.

Detector	Flame ionization detector (FID)
Column	length 6 ft, internal diameter 2 mm
Injector	temperature 200 °C, flow 15 ml/min
Oven temperature	40 °C during 10 min
Detector temperature	250 °C
Carrier gas	Air
H_2 flow	30 ml/min
Air flow	300 ml/min
Make-up (N_2)	1,2 ml/min
Run time	22,5 min