
Plastics — Determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test

Plastiques — Évaluation du degré de désintégration de matériaux plastiques dans des conditions de compostage simulées lors d'un essai de laboratoire



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Foreword

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ISO 20200 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Introduction

The test method described in this International Standard determines the degree of disintegration of plastic materials when exposed to a composting environment. The method is simple and inexpensive, does not require special bioreactors and is scaled for use in any general-purpose laboratory. It requires the use of a standard and homogeneous synthetic solid waste. The synthetic waste components are dry, clean, safe products which can be stored in the laboratory without any odour or health problems. The synthetic waste is of constant composition and devoid of any undesired plastic material which could be erroneously identified as test material at the end of testing, altering the final evaluation. The bioreactors are small, as is the amount of synthetic waste to be composted (approximately 3 l). With the limited amount of test material, this method provides a simplified test procedure. This test method is not aimed at determining the biodegradability of plastic materials under composting conditions. Further testing will be necessary before being able to claim compostability.

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Plastics — Determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test

1 Scope

This International Standard specifies a method of determining the degree of disintegration of plastic materials when exposed to a laboratory-scale composting environment. The method is not applicable to the determination of the biodegradability of plastic materials under composting conditions. Further testing is necessary to be able to claim compostability.

2 Normative references

The following referenced documents are indispensable for the application 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 3310-1:2000, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

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

3.1

compost

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

3.2

compostability

ability of a material to be biodegraded in a composting process

NOTE To claim compostability it must have been demonstrated that a material can be biodegraded and disintegrated in a composting system (as can be shown by standard test methods) and completes its biodegradation during the end-use of the compost. The compost must meet the relevant quality criteria. Quality criteria are e.g. low heavy-metal content, no ecotoxicity, no obviously distinguishable residues.

3.3

composting

aerobic process designed to produce compost

3.4

disintegration

physical breakdown of a material into very small fragments

3.5

dry mass

mass of a sample measured after drying

NOTE Dry mass is expressed as a percentage of the mass of the wet sample.

3.6

mesophilic incubation period

incubation at 25 °C to allow the development of microorganisms growing at room temperature

3.7

thermophilic incubation period

incubation at 58 °C to allow the development of microorganisms growing at high temperature

3.8

total dry solids

amount of solids obtained by taking a known volume of test material or compost and drying at about 105 °C to constant mass

3.9

volatile solids

amount of solids obtained by subtracting the residue obtained from a known volume of test material or compost after incineration at about 550 °C from the total dry solids content of the same sample

NOTE The volatile-solids content is an indication of the amount of organic matter present.

4 Principle

The method determines the degree of disintegration of test materials on a laboratory scale under conditions simulating an intensive aerobic composting process. The solid matrix used consists of a synthetic solid waste inoculated with mature compost taken from a commercial composting plant. Pieces of the plastic test material are composted with this prepared solid matrix. The degree of disintegration is determined after a composting cycle, by sieving the final matrix through a 2 mm sieve in order to recover the non-disintegrated residues. The reduction in mass of the test sample is considered as disintegrated material and used to calculate the degree of disintegration.

5 Synthetic solid waste

The composition of the synthetic waste used in this method is described in Table 1.

Well aerated compost from a commercial aerobic composting plant shall be used as the inoculum. The compost inoculum shall be homogeneous and free from large inert objects such as glass, stones or pieces of metal. Remove any such objects manually and then sieve the compost on a screen of mesh aperture between 0,5 cm and 1 cm. It is recommended that compost from a plant composting the organic fraction of solid municipal waste be used in order to ensure sufficient diversity of microorganisms. If such a compost is not available, then compost from plants treating farmyard waste or mixtures of garden waste and solid municipal waste may be used. The compost shall not be older than 4 months.

Prepare the synthetic waste manually by mixing the different components listed in Table 1. The allowed tolerance on the mass measurements of the synthetic waste components, water included, is 5 %. Add chlorine-free tap water, or de-ionized or distilled water, to the mixture to adjust its final water content to 55 % in total. Perform this operation just before start-up. The synthetic waste shall have a carbon:nitrogen (C/N) ratio of between 20:1 and 40:1. The urea concentration can be changed to adjust the C/N ratio to the required range. In this case, the concentration of the other components shall be adjusted proportionately in order to bring the total dry mass of the solid waste to 100 %.

Table 1 — Composition of synthetic solid waste

Material	Dry mass %
Sawdust	40
Rabbit-feed	30
Ripe compost	10
Corn starch	10
Saccharose	5
Cornseed oil	4
Urea	1
Total	100
NOTE 1 Sawdust from untreated wood shall be used. It is preferable to use wood from deciduous trees. Sawdust shall be sieved through a 5 mm sieve before use.	
NOTE 2 The rabbit-feed shall be a commercial product based on alfalfa (lucerne) (<i>Medicago sativa</i>) and vegetable meal. If a product with a different composition is used, the composition shall be given in the test report. The protein content of the rabbit-feed shall be approximately 15 % and the cellulose content approximately 20 %.	

6 Composting reactor

The preferred composting reactor is a box made of polypropylene or other suitable material, having the following dimensions: 30 cm × 20 cm × 10 cm (l, w, h). The box shall be covered with a lid assuring a tight seal to avoid excessive evaporation. Additionally, any gap between box and lid may be sealed with adhesive tape. In the middle of the two 20 cm wide sides, a hole of 5 mm diameter shall be made approximately 6,5 cm from the bottom of the box. These two holes provide gas exchange between the inner atmosphere and the outside environment and shall not be blocked.

Other containers with a volume between 5 l and 20 l may also be used, provided that it can be verified that no unfavourable anaerobic conditions are generated. The container shall be closed in a way which avoids excessive drying-out of the contents. Again, openings shall be provided in order to allow gas exchange and ensure aerobic conditions throughout the composting phase.

7 Procedure

7.1 Test material preparation

Cut up test material to give pieces with the dimensions defined in Table 2, based on the thickness of the material.

Dry the pieces of test material in an oven at (40 ± 2) °C under vacuum for the length of time needed to reach constant mass. Prior to mixing the pieces of test material with the synthetic waste, immerse them in distilled water for no more than 30 s.

Table 2 — Dimensions of the pieces of test material used in the disintegration test

Thickness of test material	Dimensions of pieces mm
< 5 mm	25 × 25 × original thickness
> 5 mm	15 × 15 × thickness (from 5 mm to 15 mm)

7.2 Start-up of the test

Prepare a minimum of three reactors for each test material. Take between 5 g and 20 g of test material per reactor, depending on the volume occupied by the test material, and mix it with 1 kg of wet synthetic waste. The ratio of the mass of test material to the mass of wet synthetic waste shall be in the range from 0,5 % to 2 %. Place the mixture on the bottom of the reactor, forming a homogeneous layer. Do not compress the mixture, allowing efficient gas exchange with the interior of the bed. Record the mass of test material in each reactor.

7.3 Thermophilic incubation period (high temperature)

Close and weigh each reactor and place it in an air-circulation oven maintained at a constant temperature of (58 ± 2) °C for a minimum period of 45 days and a maximum of 90 days. Record the temperature of the oven throughout the test period or, alternatively, use a maximum-minimum thermometer, checking the temperature at least twice a week.

To ensure a good composting process, it is necessary to maintain suitable environmental conditions. Follow the procedure described in Table 3. This procedure aerates the composting matter whilst maintaining a sufficient water content. The gross mass of the reactor filled with the mixture is determined at the beginning of the composting process. At each scheduled point in time (see Table 3), the reactor is weighed and, if needed, the initial mass restored totally or in part by adding chlorine-free tap water, de-ionized water or distilled water as indicated in Table 3. It is important to note that the optimum water concentration is obtained when the composting matter is wet but no free water is present. This means that the maximum water-absorbing capacity has not been reached. The operator can determine this condition by squeezing the composting matter, which shall exude a small amount of water. The operator can then adjust the amount of water to be added indicated in Table 3 on the basis of this direct check.

Mixing of the composting matter can be performed with a laboratory spatula or a common spoon. This operation shall be carried out carefully, paying attention not to damage the pieces of test material in the composting matter. The purpose of mixing is to aerate the mass and remix the water, but it is important to avoid any mechanical degradation of the pieces of test material.

7.4 Mesophilic incubation period (at room temperature)

If at the end of the thermophilic incubation period the test material has not sufficiently disintegrated, it is possible to extend the test, using the following procedure. Add 25 g of fertile earth to each reactor. Mix the compost and the earth gently to avoid any mechanical damage to the residual pieces of test material. Close each reactor and leave it in an air-circulation oven at (25 ± 2) °C for a maximum period of 90 days. Record the temperature of the oven throughout the test period or, alternatively, use a maximum-minimum thermometer, checking the temperature at least twice a week. Check the mass once a week and add water, if needed, to restore the mass to 70 % of the mass measured at the beginning of the test (see 7.3). Do not mix the composting mass during this period.

If the test is extended in this way, this shall be mentioned in the test report.

Table 3 — Composting procedure (thermophilic incubation period)

Time from start days	Operation
0	Record initial mass of reactor
1, 2, 3, 4, 7, 9, 11, 14	Weigh reactor and add water to restore the initial mass, if needed. Mix the composting matter.
8, 10, 16, 18, 21, 23, 25, 28	Weigh reactor and add water to restore the initial mass, if needed. Do not mix the composting matter.
30, 45	Weigh reactor and add water to restore the mass to 80 % of the initial mass, if needed. Mix the composting matter.
From 30 till 60, twice a week	Weigh reactor and add water to restore the mass to 80 % of the initial mass, if needed. Do not mix the composting matter.
From 60 onwards, twice a week	Weigh reactor and add water to restore the mass to 70 % of the initial mass, if needed. Do not mix the composting matter.

8 Monitoring the composting process

During the test, the synthetic waste shall become compost, i.e. the composting process shall occur. The composting reaction is monitored by inspecting the composting matter when mixing and adding water. The diagnostic parameters to be taken into account are both subjective and objective and are described in Clause 9.

9 Diagnostic parameters

9.1 Odour

During the composting process, it is possible to detect a precise succession of specific odours. Within the first two or three days, the synthetic waste has an acidic odour, which gradually decays into an ammoniacal odour from day 5 to day 10, lasting approximately 10 days. Finally, no particular odour, or an earth-like one, is detected. Record in the test report any departure from this scheme.

9.2 Visual appearance

The visual appearance of the composting matter changes during the first two weeks. Mycelia which grow on the composting matter will usually be visible during the first week. The colour of the synthetic waste, which is initially light yellow because of the high sawdust concentration, turns brown within 10 days. Record in the test report any departure from this scheme.

9.3 Chemical analysis

Analyse samples of synthetic waste used at the beginning of the test and samples of the compost obtained at the end of the composting process, after sieving, in order to determine the initial and final values of the following parameters: total carbon/total nitrogen (C/N ratio) and pH. Measure these parameters using standard test methods, and record them.

NOTE The total carbon content used to determine the C/N ratio can be taken as the volatile-solids content divided by a factor of 2.

9.4 Determination of dry mass and volatile solids

After sieving, determine the dry mass and volatile-solids content of the original synthetic waste and of the final compost obtained at the end of the composting process (see Clause 10). Determine the dry mass DM of the sample by drying in an oven at 105 °C to constant mass. Express the dry mass as a percentage of the total mass of the sample. Determine the volatile-solids content by calcining, at 550 °C for 6 h to 8 h, the sample previously dried at 105 °C to determine the dry mass. Weigh the sample, and repeat the calcination and weighing procedure until constant mass is reached. The mass loss from calcination corresponds to the volatile-solids content of the sample. Express the volatile solids VS as a percentage of the DM of the sample.

10 Termination of the test and measurement of the degree of disintegration

Remove the lid of each reactor and place the reactors in an air-circulation oven at (58 ± 2) °C to dry the contents. Gently break up any lumps of compost, paying attention not to damage any residual pieces of test material trapped inside them. Terminate this drying process when constant mass is reached.

Sieve the compost from each reactor independently, using standard sieves (see ISO 3310-1), starting with a 10 mm sieve. Examine the fraction not passing through this sieve. Gently break up the lumps of compost, paying attention not to damage any residual pieces of test material trapped inside them. Allow the particles to pass through the screen to join the fraction of particle size less than 10 mm. Collect and store any pieces of test material which do not pass through the 10 mm sieve. Subsequently screen the compost with a 5 mm sieve and, afterwards, with a 2 mm sieve, using the same procedure as for the 10 mm sieve. Pool the test material collected at the various sieving phases, clean them of compost and, if appropriate, wash them by dipping in water. It is essential that the cleaning and washing procedure be performed with great care in order to avoid any accidental loss of test material. Finally, dry the test material in an oven at (40 ± 2) °C under vacuum to constant mass. Record the final mass.

11 Calculation of degree of disintegration

The plastic material recovered from the sieving procedure (see Clause 10) is considered to be non-disintegrated material. The material which passed through the sieves is considered to have disintegrated. The degree of disintegration D is calculated, in percent, using the equation:

$$D = \frac{m_i - m_r}{m_i} \times 100$$

where

m_i is the initial dry mass of the test material;

m_r is the dry mass of the residual test material recovered by sieving.

Calculate the degree of disintegration independently for each reactor.

12 Expression of results

For the purposes of this International Standard, the degree of disintegration is considered as the average of the degrees of disintegration obtained for the three replicates.

13 Validity of the test

The test is considered valid if the following requirements are satisfied:

a) Decrease in volatile-solids content

In each reactor, the decrease R in total volatile-solids content between the initial synthetic waste and the compost obtained at the end of the test shall be greater than or equal to 30 %, R being calculated using the following equation:

$$R = \frac{[m_i \times (DM)_i \times (VS)_i] - [m_f \times (DM)_f \times (VS)_f]}{[m_i \times (DM)_i \times (VS)_i]} \times 100$$

where

m_i is the initial mass of the wet synthetic waste introduced into the reactor;

$(DM)_i$ is the initial dry mass of the synthetic waste, expressed as a percentage divided by 100;

$(VS)_i$ is the initial volatile-solids content of the synthetic waste, expressed as a percentage divided by 100;

m_f is the final mass of the compost;

$(DM)_f$ is the final dry mass of the compost, expressed as a percentage divided by 100;

$(VS)_f$ is the final volatile-solids content of the compost, expressed as a percentage divided by 100.

EXAMPLE In a typical test, the initial wet mass of synthetic waste is 1 000 g. The percentage dry mass is 44,63 % and the percentage volatile-solids content is 91,15 % (relative to the dry mass). At the end of the test, the final wet compost mass is 511 g, the percentage dry mass is 54,83 % and the percentage volatile-solids content is 83,80 %. Therefore

$$R = \frac{(1\,000 \times 0,446\,3 \times 0,911\,5) - (511 \times 0,548\,3 \times 0,838\,0)}{1\,000 \times 0,446\,3 \times 0,911\,5} \times 100 = 42,8\%$$

which is > 30 %.

b) Variability of the results

The degrees of disintegration for the three replicates shall not differ by more than 10 %.

14 Test report

The test report shall include the following information:

- a) a reference to this International Standard (i.e. ISO 20200);
- b) all information needed to identify and describe the test material, including a description of its physical form, its thickness, and the dimensions of the pieces cut from it;
- c) a description of the synthetic waste used, i.e. the components used to prepare it and the amount of each, the C/N ratio of the synthetic waste, its dry mass (expressed as a percentage of the wet mass), its volatile-solids content (expressed as a percentage of the dry mass) and its pH;
- d) a description of the equipment used, i.e. the bioreactor and its dimensions, the standard sieves;

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- e) a table showing, for each reactor, the following information: the serial number of the reactor, the designation of the test material, the amount of synthetic waste introduced, the total amount of mixture (synthetic waste plus test material) and the initial mass of the reactor (gross mass);
- f) a table showing, for each reactor, the number of the reactor and the following characteristics of the compost, determined after sieving: the total mass, the dry mass (expressed as a percentage of the total mass), the volatile-solids content (expressed as a percentage of the dry mass), the C/N ratio and the pH;
- g) a table showing, for each reactor, the following information: the number of the reactor and the decrease R in the total volatile-solids content, calculated as indicated in Clause 13, item a);
- h) a table showing, for each reactor, details of the water-addition and mixing operations performed (day, operation performed, amount of water added, mass of reactor and any observations);
- i) a table showing, for each reactor, the initial amount of test material, the amount of test material recovered at the end of the test and the degree of disintegration D , calculated as indicated in Clause 11;
- j) information on the compost inoculum used, such as source, age, date of collection, storage, handling, stabilization, total dry mass, volatile-solids content, pH-value of a suspension (i.e. 1 part of compost with 5 parts of de-ionized water), and specific odours and visual appearance of the compost, if any.

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