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Geosynthetics — Screening test method for determining the resistance of geotextiles and geotextile-related products to oxidation

Géosynthétiques — Méthode de détermination de la résistance des géotextiles et produits apparentés à l'oxydation

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Foreword

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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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 221, *Geosynthetics*.

This second edition cancels and replaces the first edition (ISO 13438:2004), which has been technically revised. The main changes compared to the previous edition are as follows:

— procedural guidance regarding the use of exposure ovens has been added;

— procedural guidance regarding the use of autoclaves has been added.

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

In many civil engineering applications, geotextiles and geotextile-related products can come into contact with water or aqueous solutions present in the soil environment. At the same time, in specific parts of the construction, they can be exposed to oxygen, giving rise to oxidative degradation processes. These processes are usually very slow.

Polyolefin materials, such as polypropylene (PP) and polyethylene (PE), are inherently more sensitive to oxidation than those based on polyethylene terephthalate (PET). Other polymers, such as poly(vinyl alcohol) (PVAL according to ISO 1043-1), are also sensitive to oxidation in specific conditions (aqueous media with oxidizing agent). This behaviour can be improved very effectively by the use of appropriate stabilizing additives.

It is the purpose of this document to provide a method for screening the resistance to oxidation of geotextiles and geotextile-related products in service for 25, 50 and 100 years. In order to achieve the sufficiently short exposure times needed for screening tests, the oxidative degradation process is accelerated. This acceleration can be achieved either by raising the temperature or by increasing the concentration of the active reaction partner. Raising the temperature can lead to the oxidation rate being limited by oxygen diffusion, thus invalidating the acceleration. This applies particularly to materials with a low surface-to-volume ratio and less to nonwovens made from fine fibres. Two methods are therefore proposed.

Method A (which was Method B in the previous edition) uses temperature alone as the accelerating factor and is used for PE, PP, PA and AR.

Method B operates at moderately high temperatures and, at the same time, the oxygen concentration is increased by using pure oxygen at high pressure. Method B is used for PVAL.

Geosynthetics — Screening test method for determining the resistance of geotextiles and geotextile-related products to oxidation

1 Scope

This document specifies a screening test method for determining the resistance of geotextiles and geotextile-related products to oxidation. The test is applicable to products as follows:

— **Method A** for material consisting solely in polypropylene (PP), polyethylene (PE), polyamide (PA), aramide (AR);

— **Method B** for material consisting solely in polyvinyl alcohol (PVAL).

The data are suitable for screening purposes but not for deriving performance data such as lifetime, unless supported by further evidence.

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 3696, *Water for analytical laboratory use — Specification and test methods*

EN 12226, *Geotextiles and geotextile-related products — General tests for evaluation following durability testing*

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Method A

4.1 Principle

Test specimens are stored in water (Grade 3 or better according to ISO 3696) at 80 °C for 28 days before being exposed to an elevated temperature in air over a fixed time period, using a regulated laboratory oven as described in 4.3. Oven ageing shall be carried out at a temperature of (100 ± 1) °C.

NOTE In the previous edition, the only difference between Methods A and B was the temperature (100 °C and 110 °C). With this revision, this difference was deleted.

The test specimens shall hang freely in the oven space.

After the fixed time period of oven ageing, the exposed test specimens are submitted to a tensile test. The tensile strength and the strain at maximum load are measured for both the control specimens and

the exposed specimens. The tensile test shall be carried out in accordance with EN 12226. Both the machine and cross direction shall be tested, unless otherwise agreed (e.g. if the machine and cross machine direction use the same size, raw material, stabilizers). For nonwovens, only one direction has to be tested. There shall be at least five test specimens and five control specimens in each relevant direction, unless further specimens are required to assure statistical significance. More information on specimens is given in 4.2.

4.2 Specimens

Products shall be manufactured at least 24 h prior to testing.

Only specimens from the same raw material/recipe shall be stored in water for 28 days and later on in an oven at one time.

The specimens to be tested shall be in accordance with EN 12226.

It is recommended to expose additional specimens in case an extra mechanical test is required.

4.3 Apparatus for oven testing

For the tests, a thermostatically regulated oven with an internal volume of sufficient size, capable of exposing test specimens to a temperature of (100 ± 1) °C, shall be used.

The oven shall be provided with a ventilation opening which shall be adjusted such that the set temperature can be maintained in that part of the oven in which the specimens are to be suspended and the flow of air through the oven is not less than three and not more than ten air changes per hour.

A higher air flow than ten per hour is a positive deviation of the test method and is accepted. This shall be noted in the test report.

The specimens shall be suspended from glass or other chemically inert fixtures in the centre of the oven, spaced and not touching, the distance from each wall being at least 100 mm.

The temperature around the specimens shall be recorded, for instance, with the aid of suitable calibrated thermocouples and a data logger.

4.4 Test procedure

4.4.1 Leaching

4.4.1.1 General

The control specimens shall be exposed for 6 h in water (Grade 3 or better according to ISO 3696) at (80 ± 1) °C and then 6 h in an oven at (100 ± 1) °C. After exposure, they should be stored in a dark room at room temperature.

The test specimens shall be stored in water (Grade 3 or better according ISO 3696) at (80 ± 1) °C for 28 days.

4.4.1.2 Water temperature

Set the water temperature to (80 ± 1) °C.

4.4.1.3 Specimens subjected to leaching

Attach the specimens to the fixtures. Once the temperature has reached a steady value, place the specimens in the water. Suspend the specimens in the centre of the water bath, spaced, not touching each other and not touching the vessel wall.

4.4.1.4 Test conditions

Test specimens shall be exposed for 28 days at (80 ± 1) °C. The water has to be changed at least every 7 days and moved at least once per day.

4.4.2 Exposure in air

4.4.2.1 General

Test specimens shall be exposed in a temperature of (100 ± 1) °C in an oven.

4.4.2.2 Oven temperature

Set the oven temperature at (100 ± 1) °C.

4.4.2.3 Specimens subjected to heat ageing

Attach the specimens to the fixtures. Once the temperature has reached a steady value, place the specimens in the oven. Suspend the specimens in the centre of the oven, spaced, not touching each other, and so that the distance from each wall is at least 100 mm.

4.4.2.4 Duration of the oven test

Test specimens shall be exposed for the duration shown in Table 1.

This table shows the test duration for a service life in natural soil with 4 ≤ pH ≤ 9 and a soil temperature ≤25 °C.

Table 1 — Temperatures and durations

Method	Material	Application of material	Service life	First: Temperature and duration in water	Second: Temperature and duration in the oven
A	**Polypropylene (PP), Polyethylene (PE), Polyamide (PA) and aramid (AR)**	non-reinforcing and reinforcing	25 years	80 °C for 28 days	100 °C for 28 days
			50 years	80 °C for 28 days	100 °C for 56 days
			100 years	80 °C for 28 days	100 °C for 112 days

Practical experience has shown that the following considerations are important in order to achieve good reproducibility:

a) the specimens should be placed in the middle of the oven;

b) draught near the oven should be avoided if a reproducible fresh air change is to be maintained;

c) the oven and the fixtures should be cleaned of any remaining residues before each new test;

d) thermo-oxidative degradation of polymer material (e.g. polypropylene) can release substances which have a catalytic effect; therefore, polymers containing different stabilizers should not be tested at the same time in the same oven, with the exception of geotextile composites.

5 Method B for PVAL

5.1 Principle for first evaluation of service lives

Test specimens are exposed for a specified time to an aqueous test liquid enriched with oxygen due to an elevated oxygen pressure above the liquid and a specified elevated temperature. Method B specifies a duration of 28 days.

The properties of the specimens are tested after this exposure in accordance with EN 12226. The tensile strength and the strain at maximum load are measured for both the control and the test specimens.

5.2 Apparatus and reagents

5.2.1 Apparatus

5.2.1.1 A pressured vessel (autoclave), large enough for the test liquid (see 5.3.1) that shall cover the specimens completely during the test. The free space above the liquid should be at least 20 % of the liquid volume. The material of the vessel and equipment shall be resistant to the test liquid under the conditions used, e.g. high-grade stainless steels.

5.2.1.2 A pressure transducer, to measure the oxygen pressure above the test liquid, with an accuracy of ±1 % operating pressure.

5.2.1.3 A temperature sensor, to measure the temperature, with an accuracy of ±0,5 °C.

5.2.1.4 Specimen holders, to ensure correct placing of specimens (see 5.3.2).

5.2.1.5 Stirring device, to maintain the homogeneity of solvent, solutes and temperature, and to allow exchange of matter between specimens and solvent.

5.2.1.6 Valves, for filling the vessel with oxygen and for emptying the vessel.

5.2.1.7 Drain-off valve, to drain the solvent from the vessel after testing.

5.2.1.8 Heating device, to maintain the test liquid at a constant temperature.

5.2.1.9 Monitoring device, for regular monitoring (at least every 15 min) of the temperature and pressure inside the vessel.

5.2.1.10 Pressure release safety equipment, as appropriate.

5.2.2 Reagents

5.2.2.1 The test specimen shall be immersed in **diluted sulphuric acid with a pH of 3,0**.

5.2.2.2 Oxygen, at a purity of at least 99,5 % by volume.

5.2.2.3 Water, Grade 3 or higher quality in accordance with ISO 3696.

5.2.3 Specimens

Products shall be manufactured at least 72 h prior to testing. The specimens to be tested shall be in accordance with EN 12226. Both the machine and cross direction shall be tested, unless otherwise

agreed (e.g. if the machine and cross machine direction use the same size, raw material, stabilizers). For nonwovens, only one direction is required to be tested. The number of control specimens and test specimens shall be a minimum of 5 in each direction tested.

Only specimens of the same chemical composition shall be stored in an autoclave at one time.

It is recommended to expose test specimens if additional tests can be required.

5.3 Test procedure

5.3.1 Quantity of test liquid and of gas phase

The mass of the test liquid shall be greater than 20 times the mass of the specimens. The free space above the liquid shall be at least 20 % of the volume of the liquid. The liquid shall cover all specimens completely during the whole test.

5.3.2 Positioning and installing the specimens

The specimens shall be held in place by a specimen holder made from an inert material. Because of the possible occurrence of shrinkage during the test, the control specimens shall be exposed for (24 ± 2) h, to the same conditions as in the test. If this is not sufficient, a fixing of the specimen without pre-strength is possible. Position the specimen holder in the vessel such that the mean distance between the specimens and the walls of the vessel, between the specimens and the surface of the liquid, and between one specimen and another, is at least 1 cm.

Fill the autoclave with the correct volume of liquid and apply the pressure for a minimum of 16 h while stirring the liquid and maintaining the temperature. Adjustment of the pressure is not necessary.

Slowly release the pressure and open the autoclave. Adjust the intensity of stirring the liquid such that no oxygen bubbles are introduced into the liquid and that the distance between the specimens and the liquid surface is maintained. Close the autoclave and increase the oxygen pressure steadily over approximately 5 min. The total time for depressurizing, loading and pressurizing should not exceed 30 min.

NOTE This process restores the test temperature, ensures enrichment of the liquid with oxygen and removes other gases dissolved in the liquid or present in the free space in the vessel.

5.3.3 Test conditions

For service lives up to 25 years, the test conditions are as follows:

— oxygen pressure: 3 000 kPa;

NOTE 1 The pressure observed at the initiation of the test is variable due to atmosphere consumption and associated ramp to equilibrium. This time is generally observed to be 24 h to 48 h. It is thus possible that equilibrium pressure control is not achieved until this time.

— after achievement of equilibrium, the oxygen pressure shall not vary more than ±100 kPa;

— test temperature: (70 ± 1) °C;

— test duration: 28 days.

For service lives of 50 years and 100 years, the test conditions are as follows:

— oxygen pressure: minimum 200 kPa;

NOTE 2 The pressure observed at the initiation of the test is variable due to atmosphere consumption and associated ramp to equilibrium. This time is generally observed to be 24 h to 48 h. It is thus possible that equilibrium pressure control is not achieved until this time.

— after achievement of equilibrium, the oxygen pressure shall not vary more than ±20 kPa;

— test temperatures of 50 °C, 60 °C, 70 °C (with an accuracy of ±1 °C) shall be used;

— an Arrhenius plot shall be used for the evaluation of the retained strength for the corresponding service life.

5.3.4 Procedure during exposure

During the first 24 h, maintain the pressure within ±4 % of set value inside the vessel. This can be achieved by several pressure adjustments, especially in the first hours following loading. After this period, maintain set pressure and temperature.

NOTE The changes in pressure are caused by the solution process of oxygen in the liquid and by possible leaks in the system, which can be detected and eliminated.

After the initial 24 h, keep the oxygen pressure, the temperature of the liquid and the intensity of stirring constant. Record the pressure and temperature regularly.

5.3.5 Removing the specimens

On completion of exposure, reduce the oxygen pressure gradually over a period of about 5 min, open the autoclave and remove the specimens from their holder.

Rinse the specimens in deionized water and condition them in accordance with EN 12226.

5.3.6 Control specimens

Expose the control specimens to the same liquid and temperature under atmospheric pressure for (24 ± 2) h and then rinse and condition them in the same way as the tested specimens.

After each test, the vessel and its equipment should be carefully cleaned of any remaining residues.

Because of the possible effect of such residues on the oxidative testing process, no products manufactured using different polymerization processes or containing different additives should be tested in the same vessel at the same time.

For safety reasons, it is essential that the specimens tested be completely covered by the test liquid during exposure to pressured oxygen.

5.4 Principle for follow up procedure

For follow-up evaluation, the following conditions are applied in order to confirm the service life chosen in the product type determination (PTD) long term durability:

— The test specimen shall be immersed in diluted sulphuric acid with a pH of 3,0;

— Oxygen pressure: the same as used for the PTD;

— Test temperature: (70 ± 1) °C;

— Test duration: depending on the activation energy determined in the PTD;

— Determination of mechanical properties.

6 Determination of mechanical properties

When the fixed time period of vessel ageing has elapsed, remove the specimens and test them in accordance with EN 12226. Determine both the tensile strength and the strain at maximum load for both the control specimens and the test specimens. Determine the ratios (as percentages) of the properties of the test specimens to those of the control specimens.

