SLOVENSKI STANDARD
SIST EN 17417:2021

01-januar-2021

Ugotavljanje dokončne biorazgradnje plastičnih materialov v vodnem sistemu pri anoksičnih (denitrifikacijskih) pogojih - Metoda z meritvijo zviševanja tlaka

Determination of the ultimate biodegradation of plastics materials in an aqueous system under anoxic (denitrifying) conditions - Method by measurement of pressure increase

Bestimmung der vollständigen Bioabbaubarkeit von Kunststoff-Materialien in wässriger Phase unter anoxischen (denitrifizierenden) Bedingungen - Verfahren mittels Messung der Druckzunahme

Détermination de la biodégradation ultime des matériaux plastiques dans un système aqueux dans des conditions anoxiques (dénitrifiantes) - Méthode par mesure de l'augmentation de pression

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13.030.99	Drugi standardi v zvezi z odpadki	Other standards related to wastes
83.080.01	Polimerni materiali na splošno	Plastics in general

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English Version

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

European foreword

This document (EN 17417:2020) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2021, and conflicting national standards shall be withdrawn at the latest by May 2021.

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Introduction

Biodegradation of a chemical substance strongly depends on environmental conditions. The presence or the absence of oxygen is significant for the metabolic pathway on which the degradation by bacteria can take place. At present, several test methods for the investigation of biodegradability of polymers under aerobic conditions, but only a few test methods for the investigation of biodegradability under anaerobic conditions exist. However, degradation under anoxic (denitrifying) conditions has barely been considered yet. The concept “anoxic” has been created by engineers and designates conditions under which denitrification can take place. This means that either a little amount of oxygen or no oxygen at all (< 0,1 mg/l) but nitrate (> 0,1 mg/l NO_3^--N) is present. During heterotrophic denitrification, e.g. inside the denitrification tank of a wastewater treatment plant, nitrate is reduced to nitrogen and at the same time organic substrate is oxidized to CO_2. In nature, anoxic conditions can be present within the hypolimnion of eutrophic lakes or within the sediment at the transition zone between the aerobic and the anaerobic zone.

A way to use biodegradable polymers after intended service life would be their addition as additional carbon source to the denitrification unit of a wastewater treatment plant. In order to check if this way of disposing a polymer is possible, the biodegradability under anoxic (denitrifying) conditions shall be determined. Even if a substance shows good aerobic degradability, this does not necessarily apply under anoxic conditions.

Furthermore, a distinction shall be made between biodegradable polymers that are soluble in water and those not soluble in water.

Those biodegradable polymers that are soluble in water could be added systematically and continuously to the denitrification unit as a solid substrate, which is quickly converted and which can therefore replace the addition of an external liquid carbon source such as ethanol or acetic acid. Testing their aerobic degradability, their water solubility and, if necessary, their water dispersibility can be carried out in accordance with EN 14987 [1]. In addition to this, special testing regarding their use as a carbon source for denitrification is done according to this document. As long as these biodegradable polymers are present as a solid substance, it shall be ensured that they remain in the denitrification tank in order to prevent operational failure during other phases of the wastewater treatment plant.

Those biodegradable polymers that are not soluble in water are discontinuously introduced as a solid substance into a specially designed denitrification reactor, where they substantially remain because of an appropriate process control. Induced by bacterial activity, they continuously release carbon for the purpose of denitrification during a process of anoxic degradation, the duration of which depends on their dimensions (surface/volume ratio). Special testing regarding their use as a water insoluble carbon source for denitrification is described in this document.

1 Scope

This document specifies a method for the determination of the ultimate anoxic biodegradation of plastics made of organic compounds, where the amount of the produced nitrogen and carbon dioxide at the end of the test is measured.

The test substance is exposed to an inoculum stemming from the denitrification tank of a wastewater treatment plant. Testing is performed under defined laboratory conditions.

Claims of performance are limited to the numerical result obtained in the test and not used for making unqualified claims such as “disposable in waste water treatment plants” 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.

EN 872:2005, *Water quality — Determination of suspended solids — Method by filtration through glass fibre filters*

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:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
ultimate anoxic biodegradation
degradation of an organic compound into carbon dioxide, water and mineral salts of any of the present elements (mineralization) as well as new biomass by means of microorganisms in the presence of oxidized nitrogen compounds (nitrate, nitrite) and in the absence of oxygen

3.2
suspended solids
solids obtained by filtration under specified conditions

[SOURCE: EN 872:2005, 3.1]

3.3
dissolved inorganic carbon
DIC
part of the inorganic carbon in water which cannot be removed by specified phase separation

Note 1 to entry: Phase separation can be achieved for example by centrifugation at 40 000 m s^{-2} for 15 min or by membrane filtration using membranes with pores of 0,2 µm to 0,45 µm in diameter

[SOURCE: EN ISO 14852:2018, 3.4]

3.4
lag phase
time, measured in days, from the start of a test until adaptation and/or selection of the degrading microorganisms is achieved and the level of biodegradation of a chemical compound or organic matter has increased to about 10 % of the maximum level of biodegradation

[SOURCE: EN ISO 14855-1:2012, 3.7]

3.5
level of biodegradation related to the nitrogen production
measured level of biodegradation of a chemical compound or organic substance in a test, calculated from the amount of actually produced nitrogen divided by the theoretical maximum amount of nitrogen

Note 1 to entry: It is expressed as a percentage.

3.6
level of biodegradation related to carbon
measured level of biodegradation of a chemical compound or organic substance in a test, calculated from the final products of mineralization of the carbon fraction (amount of carbon from carbon dioxide and biomass) divided by the carbon fraction of the amount of the test substance used

Note 1 to entry: It is expressed as a percentage.

3.7
maximum level of biodegradation
measured level of biodegradation of a chemical compound or organic substance in a test, above which no further biodegradation takes places during the test

Note 1 to entry: It is expressed as a percentage.

[SOURCE: EN ISO 14855-1:2012, 3.8, modified — the unit “percentage” has been included in the Note]

3.8
biodegradation phase
time from the end of the lag phase of a test until about 90 % of the maximum level of biodegradation has been reached

Note 1 to entry: It is expressed in days.

[SOURCE: EN ISO 14855-1:2012, 3.9, modified — the unit “days” has been included in the Note]

3.9
plateau phase
time from the end of the biodegradation phase until the end of a test

Note 1 to entry: It is expressed in days.

[SOURCE: EN ISO 14855-1:2012, 3.10, modified — the unit “days” has been included in the Note]

3.10
nitrogen recovery rate
sum of the mass concentrations of the nitrogen fractions of nitrate, nitrite, ammonium, protein and of elementary nitrogen at the end of the test divided by the sum of the corresponding mass concentrations at the beginning of the test

Note 1 to entry: It is expressed in percent.

3.11
carbon recovery rate
sum of the mass concentrations of the dissolved organic carbon (DOC), the carbon fractions of the test substance, of carbon dioxide and of biomass at the end of the test divided by the sum of the corresponding mass concentrations at the beginning of the test

Note 1 to entry: It is expressed in percent.

3.12
theoretical oxygen demand
ThOD
theoretical maximum amount of oxygen required to completely oxidize a chemical compound

Note 1 to entry: It is calculated from the molecular formula of this compound and expressed in milligram of oxygen uptake per milligram of the test compound.

3.13
theoretical nitrogen production
ThNP
theoretical maximum amount of nitrogen produced during biodegradation under denitrifying conditions

Note 1 to entry: It is calculated from the molecular formula of the test compound and the stoichiometry of the anoxic biodegradation of this compound (simplified, without biomass) and expressed in milligram of nitrogen produced per milligram of the test compound.

3.14
theoretical nitrate demand
ThND
theoretical maximum amount of nitrate-nitrogen that is reduced during biodegradation under denitrifying conditions

Note 1 to entry: It is calculated from the molecular formula of the test compound and the stoichiometry of the anoxic biodegradation of this compound (simplified, without biomass) and is expressed in milligram of nitrate-nitrogen produced per milligram of the test compound.

3.15
preadaptation
pre-incubation of an inoculum in the presence of the chemical compound or organic substance under test, with the aim of enhancing the ability of the inoculum to biodegrade the test substance by adaptation and/or selection of the microorganisms

4 Principle

In order to investigate anoxic degradation, the nitrogen production (N_2) is monitored by means of pressure measurement in a closed system.

A mineral salt medium, free of oxygen and containing nitrate, with the test substance being the only carbon source, is placed in a pressure-tight bottle and inoculated with the inoculum from the denitrification tank of a waste water treatment plant; The gas space of the bottle shall be gassed with argon or nitrogen in order to prevent oxygen from entering. Subsequently, the vessel is closed by means of a pressure measuring head. The use of KOH is favourable if the absorbed CO_2 is to be measured by means of titration in order to prepare a carbon balance.

NOTE KOH is used as its sorption capacity is higher than NaOH, especially if the solution cannot be stirred [4].

Therefore, the pressure increase inside the bottle is proportional to the nitrogen that is produced during denitrification. Pressure measurement values of a sample are captured and recorded by pressure sensors and can be transmitted to a personal computer any time. The analytical determination of the NO_3^-, NO_2^-, NH_4^+ and protein content at the beginning and the end of the test allows a nitrogen balance which provides information about the plausibility of results and, in particular, about the leak-tightness of the system. Based on experience, the resulting concentrations of the metabolites NO and N_2O will be very low, which is why they can be neglected.

The level of biodegradation under anoxic conditions is expressed as a percentage and determined by comparison of the actual and the theoretical nitrogen production. The test result is the maximum level of biodegradation determined from the plateau phase of the biodegradation curve. Optionally, a carbon balance can be calculated in order to obtain additional information regarding biodegradation and to improve the evaluation of biodegradation (see C.2).

5 Equipment and materials

5.1 Pressure measurement system

The following example represents the principle of a system for measuring the produced nitrogen by means of pressure measurement.

The pressure measurement system [1)] (see Figure 1) consists of:

— pressure measuring heads, a combination of a screw cap with integrated pressure sensor;

— bottles capable of being sealed pressure-tight for being used as reaction vessels, nominal volume: 500 ml (or 1 000 ml) with side necks made of glass also capable of being sealed pressure-tight;

— sorption vessel for the purpose of CO_2 sorption in the reaction vessels.

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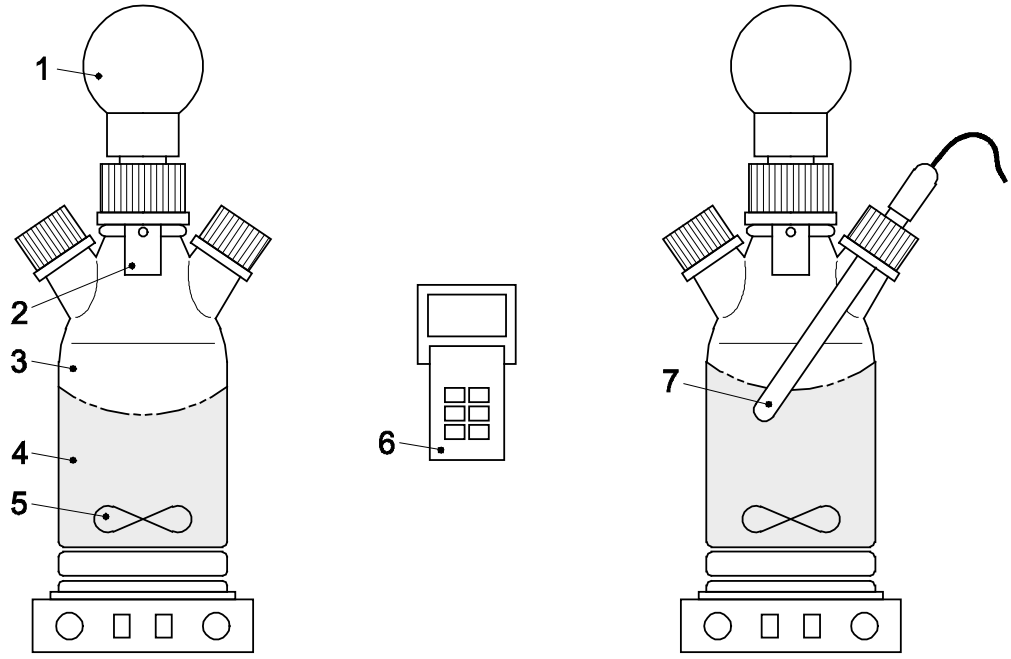
1) The OxiTop® Control system of WTW, Weilheim, Germany, consisting of "OxiTop®-C" pressure measuring heads, sorption vessels, adequate bottles and accessories, is an example of a suitable product available commercially. For this system, pressure measurement values of a sample are continuously recorded by the OxiTop® heads. At the start of the test, the value is automatically set to ambient pressure (relative zero). The heads are controlled (e.g. mode, start, measurement duration, GLP monitoring, etc.) by means of a controller (OxiTop® OC110) using an infrared interface. The relevant pressure measurement values can be retrieved, displayed and further processed by a software programme any time. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of the product named.



Key

1 pressure measuring head with IR interface
2 sorption vessel for CO_2 sorption
3 volume of the gas space (V_{gas})
4 reaction vessel containing the test mixture with medium, test substance and inoculum
5 magnetic stirrer bar
6 controller with IR interface
7 pH electrode (optional version with pH measurement)

Figure 1 — Pressure measurement system for measuring biodegradation under anoxic conditions (example)

5.2 Stirring platform or single magnetic stirrers

Stirring platforms or magnetic stirrers [2)] with a heat emission as low as possible should be used. The temperature inside the test vessels should largely remain constant because temperature variations strongly influence the measurement results.

5.3 Room or incubator with a constant temperature of (20 ± 2) °C

The incubator [3)] should maintain the temperature setpoint as closely as possible because temperature variations strongly influence the measurement results.

5.4 Argon for the elimination of oxygen from the medium and the gas space

Argon 5.0 with a purity of > 99,999 % shall be used.

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2) The stirring platform IS 6-Var of WTW, Weilheim, Germany, for 6 test vessels is an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of the product named.

3) The Thermostat Cabinets of WTW, Weilheim, Germany, are an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of the product named.