SLOVENSKI STANDARD
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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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ICS:

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

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

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

Bestimmung des endgültigen biologischen Abbaus von Kunststoffmaterialien in einem wässrigen System unter anoxischen (denitrifizierenden) Bedingungen - Verfahren durch Messung des Druckanstiegs

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 249.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 17417:2019) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

This document is currently submitted to the CEN Enquiry.

Introduction

Biodegradation of chemical substance strongly depends on environmental conditions. The presence or the absence of oxygen is significant for the metabolic pathway on which the degradation of 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. In addition to this, special testing regarding their use as a carbon source for denitrification is done according to this standard. 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 standard.

1 Scope

This document specifies a method for the determination of the ultimate anoxic biodegradability 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.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 http://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 removed by filtration under specified conditions

[SOURCE: EN 872:2005, 3.1]

3.3
dissolved inorganic carbon
DIC
that part of the inorganic carbon in water which cannot be removed by specified phase separation, 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 14852:2004, 3.4] [1]

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 14855-1:2013, 3.7] [2]

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 14855-1:2013, 3.8, modified — the unit "percentage" has been included in the Note] [2]

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 14855-1:2013, 3.9, modified — the unit "days" has been included in the Note] [2]

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 14855-1:2013, 3.10, modified — the unit "days" has been included in the Note] [2]

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 or NaOH is favourable if the absorbed CO_2 is to be measured by means of titration in order to prepare a carbon balance.

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