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Standard testing methods & specifications for biodegradation of bio-based materials in soil – a comparative analysis

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Abstract

In this paper the currently available norms and standards on testing bio-based materials for biodegradation in soil are analysed with the aim to identify and clarify the constraints, gaps and limitations of existing relevant testing methods, and propose needed modifications. The main international standards for testing biodegradability of plastics in soil include ISO 17556, ASTM D5988, NF U52-001 and UNI 11462. These standards determine biodegradability of plastics in soil under normalized circumstances. Besides a biodegradation test method, also pass levels and a time frame need to be defined in order to determine that a bio-based product will biodegrade sufficiently under soil conditions. Currently no European or international specification exists that defines criteria for biodegradation of bio-based products in soil. Only in the national standards NF U52-001 and UNI 11462 specifications for the time frame and pass levels for biodegradation of biodegradable materials used in agriculture and horticulture are defined together with criteria for environmental safety. However, the evaluation of the biodegradability in soil is not obligatory in the French specification. In order to guarantee transparent communication, rules should be standardised to define when claims are justified.

Keywords: testing methods, specifications, biodegradation, soil, bio-based materials

1 Introduction

1.1 Plastics and bio-based plastics in the Agricultural sector

About 50% of plastics is used for single-use disposable applications, such as packaging, agricultural films, and disposable consumer items (Hopewell et al., 2009). Agricultural applications in Europe in 2012 have a share of 4.2% of plastics demand (Plastics Europe). Waste generated from agricultural plastics counts for 5-6% of the total plastic waste in Europe. In general, an estimated 2–3 million tons of plastics are used each year in agricultural applications world-wide (European Bioplastics). Almost half of this amount, dominated by the use of plastic made out of polyethylene, is used in protected cultivations (greenhouses, mulching, small tunnels, etc.). A major negative consequence of the expanding use of plastics for protected horticulture is related to handling the plastic wastes and the associated environmental impact as only a small percentage of the constantly rising amount of agricultural plastic waste is currently recycled (this varies, however, from region to region or country and depends on the plastic category, e.g. most of silage films and bale wraps are recycled) (European Bioplastics). The handling of agricultural plastic waste lies considerably behind that of municipal plastic waste as it requires special management schemes and infrastructure organized at the rural areas level (Briassoulis et al., 2010). A large portion of this waste is left on the fields, buried or burnt uncontrollably by the farmers releasing harmful substances with the associated negative consequences to the environment and possibly for the safety of the

food produced (including aesthetic pollution and landscape degradation, degradation of soil quality characteristics; release of harmful substances with negative consequences to the environment and to the human health and possible danger for the safety of the food produced) (European Bioplastics).

In 2004, 143,000 tons of plastic mulch were disposed of in the U.S., either in the landfill or burned on site (Shogren & Hochmuth, 2004). This amount of plastic mulch, assuming a typical mulching film 1.3 m wide and 25.4 µm thick, would wrap around the earth over 100 times. Analogous are the quantities of plastic mulching film waste generated in Europe (Briassoulis et al., 2013). On-site burning or burying of polyethylene mulch is illegal in both EU and the US (European Bioplastics, Washington State University Extension 2013) and can also have undesirable environmental and public health impacts, such as the release of the airborne toxic substances, including dioxins, and the irreversible soil contamination. The main reasons for the mismanagement of the plastic mulching (and other agricultural) film waste are the lack of cost efficient systematic disposal techniques and management systems available to the growers along with the high labour cost for the proper collection of the plastic films and other agricultural plastic wastes following the end of the cultivation (European Bioplastics, Briassoulis, 2007). Costs for polyethylene mulch disposal (not including labour) can be up to 250$/ha (Shogren & Hochmuth, 2004). In addition, recycling is not considered as a cost efficient and/or technically feasible solution, for thin, dirty mulching or low-tunnel films.

Based on the above, the biodegradation of agricultural plastics, especially mulching films, is a very important subject with major economical and environmental aspects. The possibility to use bio-based materials biodegradable in the soil appears as a very attractive alternative to the conventional (mostly polyethylene) agricultural plastics and a real challenge for enhancing sustainable and environmental friendly agricultural activities, especially in mulching and possibly low-tunnel cultivation applications (Briassoulis, 2007). The emphasis with regard to agricultural biodegradable films, during the last years, has been placed on developing thin bio-based biodegradable mulching films. Bio-based biodegradable low-tunnel and direct cover films have also been developed at the experimental stage as well as irrigation systems (Hiskakis et al., 2011). Most biodegradable plastic mulches that are currently available commercially are made mainly from plant starch. Agricultural films made of starch are prepared using conventional plastics processing technology. However, due to the poor mechanical properties of starch, including its brittleness, it must be blended with other polymers and/or plasticizers (Washington State University Extension 2013).

1.2 Bio-lubricants in Agriculture

According to Total, the use of bio-lubricants in forestry and agriculture is limited to small equipment (e.g. chainsaws, brush cutters) and machinery for logging (forwarders, log loaders), soil preparation and harvesting. The bar and chainsaw oil market is small, estimated at 2-3 million gallons per year in Europe (Bremmer and Plonsker, 2008). As the bar and chainsaw lubricant serves to externally lubricate the bar and chain of the saw, all expelled oil enters the environment. For such total loss applications it makes sense to replace the mineral oil products by bio-lubricants.

2 Biodegradation in soil

2.1 Standard testing methods for biodegradation in soil

Biodegradation in soil is a particularly relevant end-of-life option for bio-based materials used in agricultural applications. Products (plastics, lubricants, etc.) may be introduced into the soil both intentionally and accidentally because of various agricultural activities. Then they may, or may not, degrade biologically. Compostable bio-based materials are not necessarily biodegradable in soil. For bio-based materials which do degrade in soil, the rate of degradation can vary considerably, depending not only on the molecular structure of the material, but also on soil characteristics and soil conditions such as temperature, water and oxygen availability which influence microbial activity. There are a few norms and standards, or non-standardised

testing methods on testing plastics for biodegradation in soil. The most important standard test methods for assessing biodegradation of plastics and chemicals in soil (up to the year 2012) are presented in *Table **1***.

Table 1: Overview of Standard testing methods for determining biodegradability of materials in soil

Current versions	Title
American Society for Testing and Materials International (ASTM)	
ASTM D 5988-12	Standard test method for determining aerobic biodegradation of plastic materials in soil
International Standards	
ISO 17556-2012	Plastics - Determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved
ISO 11266-1994	Soil quality - Guidance on laboratory testing for biodegradation of organic chemicals in soil under aerobic conditions
French and Italian Normalisation Organisations (AFNOR, UNI)	
NF U52-001 February 2005	Biodegradable materials for use in agriculture and horticulture - Mulching products - Requirements and test methods
UNI 11462:2012	Plastic materials biodegradable in soil - Types, requirements and test methods
OECD Guidelines	
304A	Inherent Biodegradability in Soil
307	Aerobic & Anaerobic Transformation in Soil

A detailed presentation and a critical review of these standards are presented in (Briassoulis and Dejean, 2010). The standards are not harmonised and so the test results also depend of the standard test method used.
Test method ISO 11266 is designed for testing biodegradability of organic chemicals under aerobic soil conditions (pure compounds; chemical purity > 98 %). The OECD guidelines 304A and 307 also describe special test methods for the determination of the mineralisation rate of a ^{14}C-labelled compound in soil. There are no standard test methods or specifications for biodegradation of bio-based lubricants in soil available up-to-date.

2.2 Standard specifications for determining biodegradation in soil

Besides a biodegradation test method, also pass levels and a time frame need to be defined in order to determine that a bio-based product will biodegrade sufficiently under soil conditions. *Table 2* presents an overview of standard specifications for plastics, which are biodegradable in soil. Currently no European or international specification exists that defines criteria for biodegradation of bio-based products in soil. The French and Italian national standards offer specifications. An ASTM standard specification is under development.

Table 2: Overview of standard specifications for determining biodegradability in soil

American Society for Testing and Materials International (ASTM)	
ASTM WK29802 (under development)	Standard Specification for Aerobically Biodegradable Plastics in Soil Environment
French and Italian Normalisation Organisations (AFNOR, UNI)	
NF U52-001 February 2005	Biodegradable materials for use in agriculture and horticulture - Mulching products -Requirements and test methods
UNI 11462:2012	Plastic materials biodegradable in soil - Types, requirements & test methods
Other Specifications	
Belgium Royal decree (9/09/2008) effective in July 2009	Decree specifying the norms that products should meet to be compostable or biodegradable

2.3 Standards for measurement of ecotoxicity of chemicals in soil media

An overview of guidelines developed by OECD, international standards and American stan-

dard that evaluate ecotoxicity of chemicals in soil media is given is given in *Table 3*.

Table 3: Overview of OECD guidelines & standard tests with regard to ecotoxicity of chemicals in soil

OECD Guidelines	
Guidelines/Standards	**Description**
OECD 207 (4-4-1984)	Earthworm, Acute Toxicity Tests
OECD 208 (19-7-2006)	Terrestrial Plant Test: Seedling Emergence and Seedling Growth Test
OECD 222 (13-4-2004)	Earthworm Reproduction Test (*Eisenia fetida/Eisenia Andrei*)
OECD 317 (22-7-2010)	Bioaccumulation in Terrestrial Oligochaetes
International Organization for Standardization (ISO)	
ISO 11268-1:2012	Soil quality - Effects of pollutants on earthworms (*Eisenia fetida*) - Part 1: Determination of acute toxicity using artificial soil substrate
ISO 11269-2:2012	Soil quality - Determination of the effects of pollutants on soil flora - Part 2: Effects of chemicals on the emergence and growth of higher plants
ISO 22030:2005	Soil quality -- Biological methods - Chronic toxicity in higher plants
American Society for Testing and Materials International (ASTM)	
ASTM E 1676 – 04	Standard Guide for Conducting Laboratory Soil Toxicity or Bioaccumulation Tests with the Lumbricid Earthworm *Eisenia Fetida* and the Enchytraeid Potworm *Enchytraeus albidus*

These methods are designed to assess various levels of toxicity effects in short to long-term tests in terrestrial systems. The measurement of the ecotoxicity of chemicals in soil media aim at obtaining laboratory data to evaluate the adverse effects of contaminants (for example, chemicals or biomolecules) associated with soil to earthworms (Family Lumbricidae) and potworms (Family Enchytraeidae) or to higher plants seedlings.

2.4 Labelling

The Belgian certification institute *AIB-VINÇOTTE International S.A./N.V. has established a certificate for* awarding and use of the OK biodegradable SOIL conformity mark on products used in horticultural and agricultural applications. A number of normative references have been used and specific requirements set evaluating the biodegradation and the environmental safety of the products under test. The biodegradability testing requirements and criteria include 90% biodegradation (absolute or relative to a suitable reference substrate) of the complete product/material (or for each organic constituent present in more than 1% of the material) within a maximum period of 2 years measured by the test methods ISO 17556, ISO 11266 or ASTM D5988. The certification includes assessment of ecotoxicity.

3 Results

3.1 Differences between standard testing methods

The standard testing methods determine biodegradability of plastics in soil under normalized circumstances. Discrepancies identified between the normalized conditions of the main standard testing methods for biodegradation in soil are related to:

a) Soil medium:

- ASTM D 5988: Laboratory mixture made of equal parts of natural and fertile soil samples collected from the surface layers of fields and forests obtained from at least 3 diverse locations or a mixture of natural soil and mature compost
- ISO 17566: Use of a “standard soil” as an alternative to natural soil that constitutes of industrial quartz sand, clay, natural soil and mature compost.
- NF U52-001: Natural soil with organic C < 2%.

b) Test sample:

- ASTM D 5988: Sample quantity of 200-1000 mg C for 500 g soil

- ISO 17566: Sample quantity of 100-300 mg to 100-300 g of soil; test samples may be reduced in size by means of cryogenic milling. When the CO_2 production is measured, higher test material quantities can be used (e.g. 2500 mg for 200 g of soil).
- NF U52-001: Sample quantity of 200-1000 mg of organic carbon in 500 g of soil substrate; test samples film cut to pieces 1-2 cm or powder.

c) Soil pH:

- ASTM D 5988: Soil pH should be 6-8. No suggestion about possible adjustment
- ISO 17566: Soil pH should be 6-8; pH should be adjusted to meet this requirement.
- NF U52-001: Soil pH should be 6-8; pH should be adjusted to meet this requirement.

d) Water content of soil:

- ASTM D 5988: Water content 80-100% of moisture-holding capacity (MHC) (by D425) or 50 to 70 % (by D2980)
- ISO 17566: Water content 40-60% of the total water holding capacity of the soil; specific salts may be added in the soil preferably when adjusting the water content
- NF U52-001: Water content 80% of the total water holding capacity of the soil.

e) Ratio C/N:

- ASTM D 5988: C:N ratio adjusted to a value between 10:1 and 20:1 by weight to the added carbon in the test specimen (note defined explicitly; presumably for the C:N of test sample; needs clarification though).
- ISO 17566: The ratio C:N is at least 40:1 for the sample organic C to the soil N.
- NF U52-001: C:N ratio adjusted to a value between 10:1 and 20:1 with respect to the mass of carbon contained in the sample and total N contained in the soil. The same amount of nitrogen is added in the reactors containing the soil blanks.

3.2 Differences in standard specifications

a) Biodegradability in soil testing requirements & criteria:

- NF U52-001: Only in the French specification (NF U52-001) (and the Italian UNI 11462; not presented here) for biodegradable materials used in agriculture / horticulture, time frame and pass levels for biodegradation are defined together with environmental safety criteria:
  - The tests can be done in three media: water, soil, compost
  - Minimum biodegradation percentage (%): 90 (water), 60 (soil), 90 (compost)
  - Time (months): 6 (water), 12 (soil), 6 (compost)
  - Minimum biodegradation percentage should be reached at a minimum for two of the three media for validation of the biodegradability of the mulching film. It is not required though that the soil medium is necessarily one of the two media to be tested for the validation of its biodegradability in soil.
- ASTM WK29802 (under development): The properties in the draft ASTM WK29802 specification are those required to determine if products (including packaging) will biodegrade to predetermined acceptable limits under controlled test conditions.
- Belgian Royal Decree for Acceptance of Compostable and Biodegradable Plastic Materials: The materials conform to the French specification NF U 52-001 for biodegradability of agricultural films in soil are also conform the royal decree. The biodegradation should be at minimum 90 % absolute or relative (reference material: microcrystalline cellulose) within 24 months.

b) Environmental safety testing requirements & criteria:

- NF U52-001: Criteria for environmental safety:
  - Threshold limits in heavy metals, fluorine, PCB (polychlorinated biphenyl) and PAH (polycyclic aromatic hydrocarbon) contents
  - Ecotoxicity tests: emergence and growth of 1 mono and 1 dicotyledonous plant species (ISO 11269-2); acute earthworm toxicity test (FD X 31-251); growth inhibition test with Pseudokirchneriella subcapitata (NF T 90-375).

- ASTM WK29802 (under development): The properties in the specification are required to assure that biodegradation of the materials will not diminish the value or utility of the soil resulting from the biodegradation process (= environmental safety).
- Belgian Royal Decree for Acceptance of Compostable and Biodegradable Plastic Materials: OECD 208 test in soil or conform plant toxicity requirements of EN 13432 norm.

c) Specific requirements for mulching films:

- NF U52-001: When permitted by the manufacturing process, mulching products shall bear the product name (commercial name or its reference). Otherwise, this marking shall appear in the tube supporting the mulching product.

4 Discussions & critical review

4.1 Critical review of standard testing methods for bio-based polymers

Soil medium: The ISO 17566 "standard soil" procedure evaluates biodegradability in a controlled process in an artificial soil that ensures repeatability of test results. ISO 11266 and NF U52-001 standards lead to biodegradation results closer to the targeted specific real soil conditions, which however cannot be generalised as they may still be very different from other categories of soil types and real soil conditions. The ASTM D5988 provisions are closer to ISO 11266, NF U52-001 except when mixture of natural soil and mature compost is used.

Test sample: As the ISO 17566 standard allows for the test specimens in the form of films, pieces, fragments, powder or formed articles, and NF U52-001 in the form of pieces or powder, biodegradability of the same material will be affected by the test specimens' form.

Ratio C/N: Average C:N ratios of soils vary from region to region depending on the predominant soil type and the prevailing conditions (e.g. farming, climate, etc.). However, as a C:N ratio value between 8 and 17 is typical, the recommendation of the ASTM standard is in better agreement with the real soil conditions in that respect provided that the sample organic C to N (if this provision is interpreted correctly) remains in the same range and so it will not affect significantly this ratio. The NF U52-001 provision defines this ratio in the same range but for organic C over the total sample-soil N. On the other hand, the ISO 17566 provision for a ratio of the organic C of the sample to soil N of 40:1 "*so as to ensure good biodegradation*" follows the general direction of this method for enhancing artificially biodegradation in soil. It may also be noticed that N is necessary for the biodegradation of C. Therefore high C:N may not ensure good biodegradation. It appears that the C:N ratio requirement represents an ambiguous biodegradation testing parameter not clearly defined by the various standards.

4.2 Critical review of standard specifications for bio-based polymers

Despite that several different international and national norms and standards have been developed for testing the biodegradability of plastic materials or plastic products, only a few of them are applicable to testing biodegradability of plastics in soil and only the national standards NF U52-001 and UNI 11462 specify requirements with regard to biodegradability of agricultural plastic films in soil. According to the few norms, specifications, certification schemes identified for soil medium the biodegradability level of the plastic material or product should be:

- 60 to 90 %.
- obtained in a range of temperatures from 20°C to 28°C
- in a time period from 6 to 24 months

Biodegradability is quantified by the evolved carbon dioxide. Biodegradability in a standardized lab test can deviate from the behaviour of biodegradable plastics under real soil conditions. As stated in the scope of the draft ASTM WK29802 standard, although results may indicate that the tested plastic material will biodegrade under the after-mentioned test conditions at a certain rate, it is cautioned that the results of any laboratory exposure in this specification cannot be directly extrapolated to soil environments at the actual site of use or dis-

posal since soil properties, local temperatures and humidity ranges shall be considered as they vary widely with geography.

Further, as the evaluation of the biodegradability in soil is not obligatory in the French specification (NF U52-001 (evaluation of biodegradability in freshwater and compost is also considered sufficient)), the achievement of the long term biodegradation in soil cannot be guaranteed by this standard. As pointed out in (Briassoulis and Dejean, 2010), it should be required that the soil medium is necessarily one of the two media to be tested for the validation of the biodegradability of a biodegradable bio-based product.

According to De Wilde B. (2002), as far as acceptance of biodegradable plastics in soil is concerned, two main categories of testing requirements are defined:

1) Biodegradation: 90% with duration in function of the application

2) Soil quality: no ecotoxicological effects and material may not contain heavy metals

These proposed specifications for biodegradation in soil are analogous, to some extent, with those of the French Norm NF U52-001 (apart for the optional use of the soil medium by NF U52-001), extended over a longer period to determine long-term effects. As mentioned by Briassoulis and Dejean (2010) analogous provision has been adopted by SP Technical Research Institute of Sweden concerning the requirements and associated test methods to certify polymeric materials and products: the ultimate aerobic biodegradability in soil (by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved) is determined by the requirement of biodegradation ≥ 90 % within 24 months. The time frame (≥ 60 % in 12 months) and biodegradation rate requirements of NF U52-001 do not guarantee the long term biodegradation in soil as proposed by De Wilde B. (2002).

4.3 Critical review of standard testing methods for bio-based lubricants

Testing methods based on the determination of the mineralisation rate of ^{14}C-labelled compounds (ISO 11266, OECD 304A and OECD 307) are not suitable for bio-based lubricants as lubricants are complex mixtures of different chemical substances and consequently it is impossible to select an appropriate site for the ^{14}C-label. In addition, these guidelines are very specialized and not easily applicable as they require highly specialised laboratories and the use of specially produced radiolabeled samples of the substances to be tested. Test methods that follow the CO_2 production (ISO 17556 and ASTM D 5988) can be suitable for bio-based lubricants provided that additional guidance is given towards the sample and soil media related testing procedures and conditions.

5 Conclusions

The main international standards for testing biodegradability of plastics in soil include ISO 17556, ASTM D5988, NF U52-001 and UNI 11462. These standards determine biodegradability of plastics in soil under normalized circumstances. Differences between the normalized conditions of these standards are related to a) soil medium (natural soils from specified locations or laboratory mixture of soils or mixture of natural soil and mature compost or “standard soil”); b) test sample (intact, cut in pieces or pulverised); c) soil pH (natural or adjusted); d) ratio C/N (natural or adjusted to 10:1-20:1 with respect to the mass of carbon contained in the sample or at least to 40:1 for sample organic C to soil N).

Biodegradability measured in a standardized lab test represents an inherent material characteristic of a product under standardized conditions. The standard testing procedures may enhance biodegradation of polymers, leading to an optimal controlled biodegradation process that may be not representative for biodegradation of the specific bio-based materials/products under specific soil conditions.

Test method ISO 11266 is designed for testing biodegradability of organic chemicals under aerobic soil conditions (pure compounds; chemical purity > 98 %). The OECD guidelines 304A and 307 also describe special test methods for the determination of the mineralisation rate of a ^{14}C-labelled compound in soil. There are no standard test methods or specifications for biodegradation of bio-based lubricants in soil available up-to-date. Testing methods based on the determination of the mineralisation rate of ^{14}C-labelled compounds (ISO 11266,

OECD 304A, OECD 307) are not suitable for bio-based lubricants as lubricants are complex mixtures of different chemical substances and consequently it is impossible to select an appropriate site for the ^{14}C-label. Test methods that follow the CO_2 production (ISO 17556, ASTM D5988, NF U52-001) can be suitable for bio-based lubricants provided that additional guidance is given towards the sample and soil media related testing procedures and conditions.

Besides a biodegradation test method, also pass levels and a time frame need to be defined in order to determine that a bio-based product will biodegrade sufficiently under soil conditions. Currently no European or international specification exists that defines criteria for biodegradation of bio-based products in soil. Only in NF U52-001 and UNI 11462 specifications for the time frame and pass levels for biodegradation of biodegradable materials used in agriculture and horticulture are defined together with criteria for environmental safety. However, the evaluation of the biodegradability in soil is not obligatory in the French specification.

Concerning bio-based lubricants, there is a need for an appropriate testing method that should be based on proper adaptation of testing methods for biodegradation of bio-based polymers in soil, combined with specifications and labeling analogous to those already available for biodegradable in soil plastics. Work in this direction is in progress in the framework of the KBBPPS project.

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