Open-BIO

Opening bio-based markets via standards, labelling and procurement

Work package 5

In situ biodegradation

Deliverable N° 5.1:

Interlaboratory test on soil biodegradation

Deliverable N° 5.3:

Biodegradability in soil test

Public summary

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List of abbreviations, acronyms and used terms

ASTM	American Society for Testing and Materials
CEN	European committee for standardisation (Comité Européen de Normalisation)
DM	Dry Matter
EC	Electric conductivity
ISO	International Organization for Standardization
KBBPPS	Knowledge Based Bio-based Products' Pre-Standardization
LDPE	Low Density PolyEthylene
NF	Association française de normalisation
PBSe	PolyButylene Sebacate
PBSeT	PolyButylene Sebacate-co-butylenTerephtalate
PHB	Poly(3-HydroxyButyrate)
TC	Total Carbon
TOC	Total Organic Carbon
UNI	Italian Organization for Standardization
USDA	U.S. Department of Agriculture
VS	Volatile solids
WC	Water capacity
WHC	Water holding capacity

1 Public summary

The goal of Open-Bio, a research project funded by the European Commission within FP7 (*7th Framework Programme for Research and Technological Development*) (www.open-bio.eu), was to investigate how bio-based products can be integrated into the market and Bio-based Economy, using standardisation, labelling and procurement. Open-Bio included research on biodegradability test methods for bio-based products in several natural environments: soil, freshwater and marine environment. Concerning biodegradation in soil and freshwater, the Open-Bio work is a follow-up of the work carried out earlier in the European project KBBPPS, in which the biodegradation of bio-based lubricants in soil and freshwater was studied and test methods were developed. The test method for biodegradation of lubricants in soil was based on existing relevant standard testing methods for biodegradation of plastics in soil (KBBPPS Deliverable N° 6.1: Report on current relevant biodegradation and ecotoxicity standards) and led to an optimized methodology for testing biodegradation of bio-based lubricants in soil.

One of the objectives of the Open-Bio project was to extend the test method for biodegradation of bio-based lubricants in soil to include also biodegradation of solid products (e.g. plastics). The horizontal soil biodegradation testing scheme developed by Open-Bio addresses a broad range of bio-based products including solids (e.g. plastics) and liquids (e.g. lubricants). The developed horizontal test method was refined by organizing a series of laboratory tests by several laboratories and the development of additional biodegradation test data.

The validity of the optimised horizontal test method for measuring biodegradation of plastics in soil was confirmed through an interlaboratory test. Due to the fact that the duration of the test is rather long (1-2 years) and requires large testing facilities and a significant amount of work, only three Open-Bio partners (Agricultural University of Athens, Novamont and OWS) participated to the interlaboratory testing.

The test methodology used in the Open-Bio interlaboratory test was based on carbon dioxide production in closed flask bioreactors, even though oxygen consumption methods and continuously aerated systems are also suitable for measuring biodegradation in soil. The horizontal testing method was based on the adaptation of the international standard 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*. Alternative provisions of the American standard ASTM D5988-12 *Standard Test Method for Determining Aerobic Biodegradation of Plastic Materials in Soil*, the French standard NF U52-001 *Biodegradable materials for use in agriculture and horticulture - Mulching products - Requirements and test methods* and the Italian standard UNI 11462 *Plastic Materials Biodegradable In Soil - Types, Requirements And Test Methods* were also taken into account. The horizontal soil test methodology not only included the special addition methods suitable for lubricants, which were in the KBBPPS project, but it also included provisions for improving the reproducibility of the test results. In particular the method proposed contains refinements with respect to sample quantity, addition of nutrients, and selection of soil, which improved the quality of the measurements.

The horizontal testing method used in Open-Bio concerns both bio-based and fossil-based materials. Similar to the biodegradation interlaboratory tests which were performed in freshwater and in the marine environments, polymers LDPE (Low Density Polyethylene; negative reference material), PHB copolymer (Poly(3-HydroxyButyrate)), PBSe (PolyButylene Sebacate) and PBSeT (PolyButylene Sebacate co butylene Terephthalate) were used for the interlaboratory test in soil.

The results of the interlaboratory test show that the reproducibility of the testing method is satisfactory. All participating laboratories found similar biodegradation for the four test materials despite the different inocula, namely natural soils of various compositions and sources. Identical biodegradation behaviour was found for the tested polymers in all laboratories. The following common trend was observed regarding the biodegradation rate of the tested materials in soil: cellulose ≈ PHB > PBSe > PBSeT > LDPE. The difference in biodegradation between the different laboratories is the lowest for the easily biodegradable polymers. In the case of materials which exhibit slower biodegradation, such as PBSeT, the variability of the results increases. This effect is indicated not only by the higher standard deviation between the replicates of the same laboratory but also by the variations found among different laboratories. Similar behaviour was observed for this material in the freshwater and in marine environments.

Despite the success of the current interlaboratory biodegradation tests, a few minor questions remain open concerning testing of biodegradation in soil. The interlaboratory test that took place in the framework of Open-Bio does not include for example composite materials having a part that it is not or very slowly biodegradable, while the rest of the item biodegrades. Only lubricants which were mixtures of compounds with different biodegradation behaviour were tested in the framework of KBBPPS. Therefore, the sensitivity of the test for such samples is not sufficiently investigated so far. If the tests concern composite materials of unknown synthesis or construction, the interpretation of the biodegradation test results of the products could be uncertain. Determining partial biodegradation may be proven to be difficult. Likewise, the behaviour of the two polyester materials PBSe and PBSeT especially that of PBSeT, was in several cases unpredictable without any profound explanation. Such behaviour may lead to non-reproducible test results and confusing conclusions. Future research should focus on investigating the complicated biodegradation mechanisms of materials and products exhibiting unpredictable or difficult to measure biodegradation in soil behaviour.

The results of the performed interlaboratory soil biodegradation tests could be useful input for standardisation groups working on soil biodegradation of (bio-based) polymers. Specifically, this deliverable may support the work of the ISO and CEN working groups which are responsible for the development of ISO 17556 and EN ISO 17556 (Soil biodegradation) (namely: ISO/TC 61/SC 5, and CEN/TC 249).

Website: www.open-bio.eu