KBBPPS

Knowledge Based Bio-based Products' Pre-Standardization

Work package 1

Project Management

Deliverable N° 1.2:

Final Public Project Report

Public

Version: 1

Delft, 25 September 2015
prepared by:

NEN

Ortwin Costenoble

Netherlands Standardization Institute
NEN Energy Resources
Vlinderweg 6, 2623 AX Delft, the Netherlands
Tel.: +31 (15) 2690 326
Fax: +31 (15) 2690 207
sEmail: energy@nen.nl
Partner website : www.nen.nl
Project website : www.KBBPPS.eu

Table of content

1 Introduction and project objectives 3
2 Work progress and achievements of the project 4
2.1 Work package 1: Project management 4
2.2 Work package 2: Dissemination 4
2.3 Work package 3: Bio-based carbon content 5
2.4 Work package 4: Biomass content 6
2.5 Work package 5: Functionality 7
2.6 Work package 6: Biodegradability 8
3 Deliverables from the project 10
4 Conclusions 12

1 Introduction and project objectives

The research project "Knowledge Based Bio-based Products' Pre-Standardization", is a project of six research institutes and organizations involved in the bio-based economy:

1. NEN, the Netherlands Standardization Institute (NL),
2. ECN, the Stichting Energy research Centre Netherlands (NL),
3. UoY, the Green Chemistry Centre of Excellence at the University of York (UK),
4. nova, the nova-Institut für politische und ökologische Innovation GmbH (DE),
5. OWS, Organic Waste Systems NV (BE),
6. DLO-FBR, the Stichting Dienst Landbouwkundig Onderzoek, Institute Food & Bio-based Research at Wageningen University (NL), and
7. AUA, the Agricultural University of Athens (GR).

The project aims at increasing the uptake speed of standards and certification systems for bio-based products. The application of standards and certification systems in the European bio-based product industry has positive long-term effects on the overall development of bio-based product markets. Trade barriers are reduced and the development of a pan-European market for bio-based products is promoted. Finally, public acceptance of bio-based products is increased through ensuring and verifying the sustainable sourcing of raw materials, the effective bio-content and clear indication of their (comparative) functionality in relation to the regular products. The KBBPPS project aims at increasing the uptake speed of standards and certification systems for bio-based products.

This project covers research and demonstration on bio-based carbon content determination, biomass content methods not solely dependent on 14C-analysis and biodegradability and eco-toxicity test schemes. Next, identification and resolution of functionality related bottlenecks with the view to developing, harmonising and validating test methodologies have been undertaken. The possibilities for improving sample preparation, fractionation and thermal treatments have been be studied in order to cover bio-based carbon and other bio-based elements determination. A comparison on what the optimum definition and determination of "bio-based" is, has been concluded. Next, practical solutions for stakeholders, lab and field tests on biodegradation or biological derived elements will be investigated. The goal in the end is that the results can be copied one-to-one into European standards.

Following Mandates by the European Commission (EC), the European Standardization Committee (CEN) initiated a Technical Committee, CEN/TC 411. on "Bio-based products". The KBBPPS has participated in and reported to many of the groups developing standards under this committee. The project has exchanged information ASTM and ISO and with organizations in the USA and Australasia; either directly or via one of its advisory partners. By doing pre- and co-normative research for them, KBBPPS has allowed the European stakeholders to progress with well-defined, sound test methods correlated to actual field behaviour and applicability in the lab.

2 Work progress and achievements of the project

2.1 Work package 1: Project management

A total of seven project meetings have been organised. One mid-term report has been presented to the EC which received minimal comments. The project manager, O. Costenoble of NEN, has had several exchanges with the EC, both DG RTD and DG Growth, on the progress of the research work and on how to disseminate this to the EC and beyond. Assistance with workshops and trainings has been organized.

2.2 Work package 2: Dissemination

Apart from a website and a project leaflet, dissemination was mostly supported by presentations of the project at different conferences. Sometimes these were dedicated to standardization and the KBBPPS activities, sometimes comprising broader topics, but always referring to the importance of reliable standards as a mechanism to improve the marketability of bio-based products. A total of 42 conferences or exhibitions have been visited by the partners for interaction with stakeholders and on 24 a lecture or poster was presented whereas on 10 a partner or the project had a booth where KBBPPS information was shared with the attendees.

Exchange with CEN and especially CEN/TC 411 was ensured through regular participation of project members in working group meetings of the Committee. Several activities contributed to also reaching stakeholders beyond Europe, intending to have some standardization impact on a global level. This was of course much more limited than the European dissemination. As the standardization work developed within CEN and KBBPPS, cooperation with ASTM was also held up through regular communication via email and telephone by project members in order to discuss the evolving differences between the CEN and ASTM standards and how they could be harmonized. The communication resulted in potential changes of ASTM standard D6866 (currently being discussed) and an increased awareness of American stakeholders of the European view on bio-based measurements. See Deliverable D2.6 for more detail.

Four events were organised to reach effective exchange with players of the bio-based economy: one stakeholder workshop dedicated to WP5 and three general Advisory Workshops. All workshops had a mixed attendance in terms of number and background of participants. At each event discussion how best to utilise the research to make a positive impact in support of bio-based products (standardization), both within Europe and beyond, took place. Advisory partners were actively involved in two events, presenting their point of view on results. The harmonisation and global cooperation between US and EU standards was a much debated topic, as there are substantive differences as well as differences in terminology.

2.3 Work package 3: Bio-based carbon content

A review on bio-based carbon content/D3.1.on sampling and biogenic carbon standards on global level (D3.1) has been produced. It lists a detailed summary of connected international standards (EU, US, but also Chinese, Australian) on e.g. sampling of biofuels, mineral fuels, natural products and biomass parameters, with details on pre-treatment parameters. The work in the first months led to the conclusion that the 14C determination method can be performed by three techniques, which are considered to be equivalent:
1) AMS - accelerated mass spectrometry
2) LSC - liquid scintillation counting
3) BI - beta-ionization technique
This was the starting point for the standardization work laid down finally in a CEN Technical Specification (CEN/TS 16640, "Bio-based products — Determination of the bio based carbon content of products using the radiocarbon method") on the determination of bio-based carbon content in products, based on the 14C content measurement.

In order to finalize a standard from this technical specification, a number of verification steps had to be followed. All steps were discussed within CEN/TC 411/WG 3, which had to be completely connected to the complete process, so clarity of the process is maintained. The first step was a ruggedness test execute at ECN on a large number of different samples. A set of findings and sample preparation steps were defined. All findings of the ruggedness test were incorporated in CEN/TS 16640.

After the ruggedness test at ECN, the next step for verification of the method was an inter-laboratory assessment. This check test was performed on a product for which it was assume difficult to determine the total carbon and eventually bio-based carbon content using CEN/TS 16640. A water-based paint containing volatile components and a low amount of carbon was chosen. The relative standard deviation for the total carbon content measured among the five different laboratories is 15%. For the biogenic carbon content the relative standard deviations is 17%. Conclusion was that with a difficult product analysed with the present CEN/TS 16640 all laboratories obtained similar results. The next step in the verification process is a full inter-laboratory test with more labs, several products and a diversity of bio-based content in order to determine full precision and be able to develop the CEN/TS into a full European Standard.

The process for the biogenic carbon standard has been harmonized with CEN/TC 411. Discussions on the harmonization of CEN/TS 16640 with ASTM D 6866 have been entertained within the project and possible solutions are currently under review. The main difference is that ASTM determines the bio-based content on the basis of the total organic carbon. The KBBPPS project has defined additional sample preparation steps for products containing inorganic carbon as well as internal verification criteria to determine whether these have been successful. That allows the test laboratory to determine the total biogenic carbon content. At the final advisory workshop a discussion initiated by one of the advisory partners on the difference, resulted in the advice to CEN and ASTM to clarify in both standards the dif-

ferences in wording and terminology. This will have further complications in other standards and therefore will have to be discussed within CEN/TC 411. Also for the standard being developed for the bio-based content (WP 4), the standard regard-ing the bio-based carbon content is an essential part for prEN 16785: “Determination of the bio-based content using the radiocarbon analysis and elemental analysis”.

2.4 Work package 4: Biomass content

The first deliverable D4.1. on sample selection demonstrates comprehensive coverage of products and thermal treatment (combustion of organic samples). The results led to the recommendation that in instances where bio-based content analysis has produced a result of dubious accuracy, the combustion stage of sample preparation may be scrutinised to a greater degree with thermogravimetric analysis (TG). This provided a solid basis for an effective study of bio-content of the products in relation to the regular (non-bio-based) equivalents.

The next work on D4.2 on fractionation of formulations for biogenic measurement gives sections on biolubricants, -plastics, -solvents, -surfactants with innovative research data on fractionation techniques and comparative analysis of biodegradability for commercial utility products. In general, the fractionation approaches used were broadly successful, with some technical limitations reported. The fractionation methods demonstrated possible use in the verification of the total bio-based content of formulations. We found that simple preparative techniques such as freeze drying can assist conversion to carbon dioxide before radiocarbon analysis, and should be considered if combustion is not successful at first. In general fractionation can only be recommended if an independently determined total bio-based content of a formulation or composite is disputed by the supplier.

Broader aspects of sample preparation are reviewed in D4.3, which concludes that there are sufficient spectroscopic, drying, and particle size reducing procedures already established for the needs of total bio-based content determination.

The next work was to look to alternatives for the C14 isotope. The investigations have shown that the promising technique of stable isotope analysis is actually not suitable as a means of determining total bio-based content. Explanation of the different indirect methods and showing the different outcomes (which can be confusing on the market in B2B and B2C relations), has been provided in D4.5.

Final conclusion is that it proved unsuccessful to develop methods for direct measurement of the bio-based content of products (stable isotopes, markers). In CEN/TC 411/WG 3 two standards for the determination of the bio-based content of products are at an advanced stage of development: FprEN 16785-1 (Bio-based content - Part 1: Determination of the bio-based content using the radiocarbon analysis and elemental analysis) and prEN 16785-2 (Bio-based content - Part 2: Determination of the bio-based content using the material balance method). We propose on the basis of the KBBPPS work that the best method to determine the bio-based content of products is the mass balance described in Part 2. The bio-

based content can then be validated by using Part 1 when appropriate. Using this approach is the best way to communicate the (minimum) total bio-based content of a product. In D4.6 a satisfactory framework for utilising EN 16785-2 with validation made possible by EN 16785-1 has been constructed.

The recommended techniques for the determination of total bio-based content have been demonstrated to work together in a complementary way. The robustness and accuracy of these methods is to be further elucidated in the EU project Open-Bio.

2.5 Work package 5: Functionality

Bottlenecks and impacts on functionality tests have been defined and discussed at stakeholder events with key policy and industry stakeholders present. In the first half of the project, a list of 26 bio-based products was chosen as a basis for the research work (D5.2), after a stakeholder workshop was successfully held for validation of the list and its objectives (D5.1). The list has been proven to be of high value and impact as it was sighted on many occasions beyond the project.

The analysis of existing barriers hampering market entry for bio-based products (BBP) led to the Deliverable D5.3 “Market entry barriers”. Three categories of barriers stemming from norms and standards were identified:

1. Commonly used product specifications are not addressing favourable bio-based properties,
2. Commonly used product specifications cover properties that are not really necessary for or related to product functionality, but these are not fulfilled by bio-based products, and
3. Bio-applicability is missing due to “old thinking” in terms of conventional products.

These were used in the second step of the bottleneck research. Here, we conducted further expert interviews with the objective to confirm the identified barriers and to come up with solutions related to standardisation and testing. The results were presented in D5.4. For some products, however, some very concrete problems were found in standards and test methods:

Product group	**Barrier**	**Suggested resolution / next steps**
Packaging films	Overly ambitious barrier properties	Testing in Open-BIO
WPC	Thermosets excluded / no mention of bio-based / lack of modulus of elasticity	CEN working group on WPC revisiting EN15534 should address these issues
Natural fibre insulation	Heat decrement delay / acoustic performance not included; automatic moisture correction factor	Adapt the relevant standards EN ISO 10456, DIN 4108-4 and DIN 52612-2
Mulch films	No specific standard; no differentiation from conventional films	CEN working group should make exceptions for minimum thickness for bio-based, biodegradable mulch films
Adhesives	Adhesion strength on wet surfaces	Testing in Open-BIO

Another study executed was the analysis of green labels with a view on including bio-based content as a criterion yielded an overview report (D5.5). The report concluded that for the four most popular European Type I multi-criteria ecolabels it is technically possible to add a bio-based share of products as a criterion to existing or newly developed criteria catalogues. Especially the EU Ecolabel, the Nordic Swan and the Blue Angel offer good framework conditions for such a development.

2.6 Work package 6: Biodegradability

The first task executed was to develop D6.1, an overview on biodegradation and ecotoxicity standards. This is a detailed and technical review of the topic containing information on international (e.g. ISO, US, Australian, EU, etc.) standards in addition to the review of toxicity standards and a discussion on specifications of the (national) European labels. This document was used as basis of further work but also as reference work in a French standard for home compostable products.

Subsequently, questionnaires were sent to lubricant and solvent industry in order to investigate needs and problems related to biodegradability, environmental safety and labelling. For lubricants, it could be concluded that the existing OECD methods are well established and there is no need to write completely new biodegradation testing methods or ecotoxicity testing methods. Focus was thus placed on improvement of biodegradation testing methods (addition methods for poorly water-soluble test items, reproducibility, variation in the inoculum, etc.) in coordination with CEN/TC 19/WG 33, *Bio-lubricants*. Biodegradation test methodologies in freshwater and in soil were developed for bio-lubricants (deliverable D6.2). For solvents, the responses showed that no further work on biodegradation and toxicity seemed necessary. This was confirmed in a meeting with CEN/TC 411/WG 2, *Bio-solvents*.

In the final phase of the project the reproducibility of the developed test methodologies was evaluated by means of two interlaboratory tests (D6.4, Biodegradability method validation and D6.5, Biodegradability standards assessment report). Two different addition methods have been investigated and the optimum was included in the final test description. The results of the first ruggedness study for fresh water biodegradation by the project were presented by OWS during a WG 33 meeting in August 2014. The objective of the second part of the interlaboratory test in freshwater was the evaluation of the reproducibility between the laboratories (as requested by WG 33). The results of the freshwater biodegradation test after 28 days were characterised by a maximum standard deviation around 10%. The results were forwarded to WG 33 “TF Biodegradation” in May 2015 in order to determine the reproducibility of the developed freshwater methodologies.

The fresh water results led WG 33 to submit to CEN/TC 19 two proposals for new work, based on carbon-dioxide production or oxygen consumption. NEN, OWS and AUA assisted with writing these proposal, which are balloted between August and October 2015.

The samples received from WG 33 for the testing in freshwater were also used for the ruggedness study in soil, which was performed by AUA, OWS and advisory partner SCION. These results were characterised by standard deviations < 7%. Such standard deviations are comparable/lower than standard deviations reported in the Round Robin test that was executed in ISO 17556:2012 on microcrystalline cellulose reference material and starch/poly(butylene adipate-co-butylene terephthalate) blend test material. and to develop a proposal for new work. Finally, the validation of the laboratory tests by a field test was executed by AUA. Biodegradability in soil under laboratory conditions was determined by measuring the CO2 production, while in the field the evolution of the organic carbon content in the soil was determined to estimate qualitatively the fate of the lubricant. Although the field test took place under uncontrolled conditions, it can be concluded that the biodegradation of the samples roughly followed the same pattern as observed in the laboratory tests.

Besides the development and validation of biodegradation testing methods, acceptance criteria for bio-based products have been developed and discussed amongst the KBBPPS partners. From these internal discussions in 2014 (also with advisory partners), the idea of the horizontal biodegradation standards was developed and introduced to the CEN/TC 411 meeting of 25 June 2015. CEN/TC 411 felt that it was not the suitable group in order to start such work, as people could get confused by making a link between the terms “biodegradable” and “bio-based”. Developing ideas around a new standardization group in CEN or ISO on environmental claims and characterization will be part of the Open-BIO project.

3 Deliverables from the project

The following table shows all the deliverables from each work package, them being reports (R) or objectives reached within the project (O) being established via a milestone or in a project meeting. The level of dissemination varies between confidential within the project (CO) or to researchers only (PP), restricted to groups defined by the project partners (RE, mostly CEN) or publicly available at the KBBPPS website (PU).

Deliverable no	Deliverable name	WP	Nature	Dissemination level
D 1.1	Intermediate project report	WP1	R	CO
D 1.2	Publishable final report	WP1	R	PU
D 1.3	Final project report to the EC	WP1	R	CO
D 2.1	Website	WP2	O	PU
D 2.2	First workshop participant list and report	WP2	R	PU
D 2.3	Halfway workshop: participant list and report	WP2	R	PU
D 2.4	Final event participant list and report	WP2	R	PU
D 2.5	(Non-) scientific publications and press releases	WP2	R	PU
D 2.6	General overview report on publications and outreach to non-European organizations	WP2	R	PU
D 2.7	Status report to CEN/TC 411	WP2	R	RE
D 2.8	Final report of work to CEN	WP2	R	RE
D 3.1	Overview of current relevant sampling and biogenic carbon standards on global level	WP3	R	PU
D 3.2	Generic description of a horizontal method	WP3	O	RE
D 3.3	Interim report on the verification of the method	WP3	R	RE
D 3.4	Final report on the verification of the method	WP3	R	RE
D 3.5	Report on the interlaboratory test concerning a difficult product	WP3	R	RE
D 3.6	Public report on horizontal standard for biogenic carbon determination	WP3	R	PU
D 4.1	Assessment study on novel thermal treatments for biogenic measurement	WP4	R	CO
D 4.2	Assessment study on fractionation of formulations pre biogenic measurement	WP4	R	CO
D 4.3	Report on sample preparation techniques for total biomass content determination	WP4	R	PU
D 4.4	Assessment of analytical techniques for measurement of the biomass content including elements other than carbon	WP4	R	RE
D 4.5	Assessment of indirect declaration techniques to determine the total biomass content	WP4	R	PU
D 4.6	Draft test methodology and testing scheme for determination of biomass content	WP4	O	RE
D 5.1	Stakeholder workshop report	WP5	R	PU

Deliverable no	Deliverable name	WP	Nature	Dissemination level
D 5.2	Selection of bio-based materials, intermediates and products	WP5	O	PP
D 5.3	Identification of main market entry barriers	WP5	R	PU
D 5.4	Resolution action plan regarding functionality test methods for selected product streams	WP5	R	RE
D 5.5	Report on green and natural labels and need for action	WP5	R	PU
D 6.1	Report on current relevant biodegradation and ecotoxicity standards	WP6	R	PU
D 6.2	Draft standard(s) on biodegradability and environmental safety	WP6	O	RE
D 6.3	Evaluation study on eco-toxicological impact and labelling possibilities	WP6	R	CO
D 6.4	Validation report on standard(s) including advice on acceptance criteria	WP6	R	RE
D 6.5	Biodegradability standards assessment	WP6	R	PU

4 Conclusions

It proved relatively difficult to achieve dissemination impact beyond Europe, which was of course partly due to limited capacities and other priorities, such as the exchange with CEN. Aside from singular interactions that remained on a more general level, especially the communication with ASTM proved to be fruitful in the sense that the project not only initiated discussion in the two standardization committees but also exchanges between them.

The fact that advisory partners from outside Europe have been connected to the project was a mutual benefit to them and all partners, not to forget the project deliverables. However, it has not (yet) resulted in a more broader use of the KBBPPS results as far as we can see it at this stage. Maybe via the follow-up project Open-Bio more outreach results may become visible.

The work on bio-based carbon content has, in interaction with CEN/TC 411/WG 3 resulted in a draft test technique that has been published by CEN as CEN/TS 16640 "Bio-based products — Determination of the bio based carbon content of products using the radiocarbon method". Current tests will continue with a complete round robin in order to obtain the performance characteristics of CEN/TS 16640. For finalizing the standard a complete round robin will be performed within the Open-Bio project. The bio-lubricants and bio-solvents groups will wait for this standard in order to refrain from requiring ASTM D6866 in their documents.

When looking to sample preparation techniques for total bio-based content, fractionation is the most promising, but can only be recommended if an independently determined total bio-based content of a formulation or composite is disputed by the supplier. The recommended techniques for the determination of total bio-based content have been demonstrated to work together in a complementary way. Those methods that were not harmonious were discounted (e.g. stable isotope analysis, mass balance). How to apply the acceptable methods in such a manner to produce equivalent results was presented to CEN as D4.6.

The bio-based market has been brought back to a list of 26 product categories that make checking barriers and opportunities much easier. Extensive desk research and an industry survey served to investigated the most important barriers and bottlenecks impeding the market uptake of the 26 bio-based product categories. Possible solutions were also developed and a resolution action plan was conceptualized.

An extensive literature review on biodegradation and toxicity test methodologies in freshwater, marine environment, anaerobic conditions, soil and composting was published (publicly available on the KBBPPS website). This document was used as reference work in a French standard for home compostable products (NF T 51-800). Biodegradation test methodologies in freshwater and soil for bio-based lubricants have been developed. The freshwater biodeg-

radation methodologies were developed in cooperation with CEN/TC 19/WG 33 TF Biodegradation (with input of KBBPPS partners), while the test methodology in soil was developed by the project partners. The results of the tests were used by CEN in order to evaluate the reproducibility of the biodegradation test methodology in freshwater. This input has led to two proposals for new work for further development of a European test standard. In addition, the KBBPPS partners have developed generic acceptance criteria for bio-based products in terms of biodegradation and a proposal for a generic technical standardization committee for biodegradation and ecotoxicity work.