

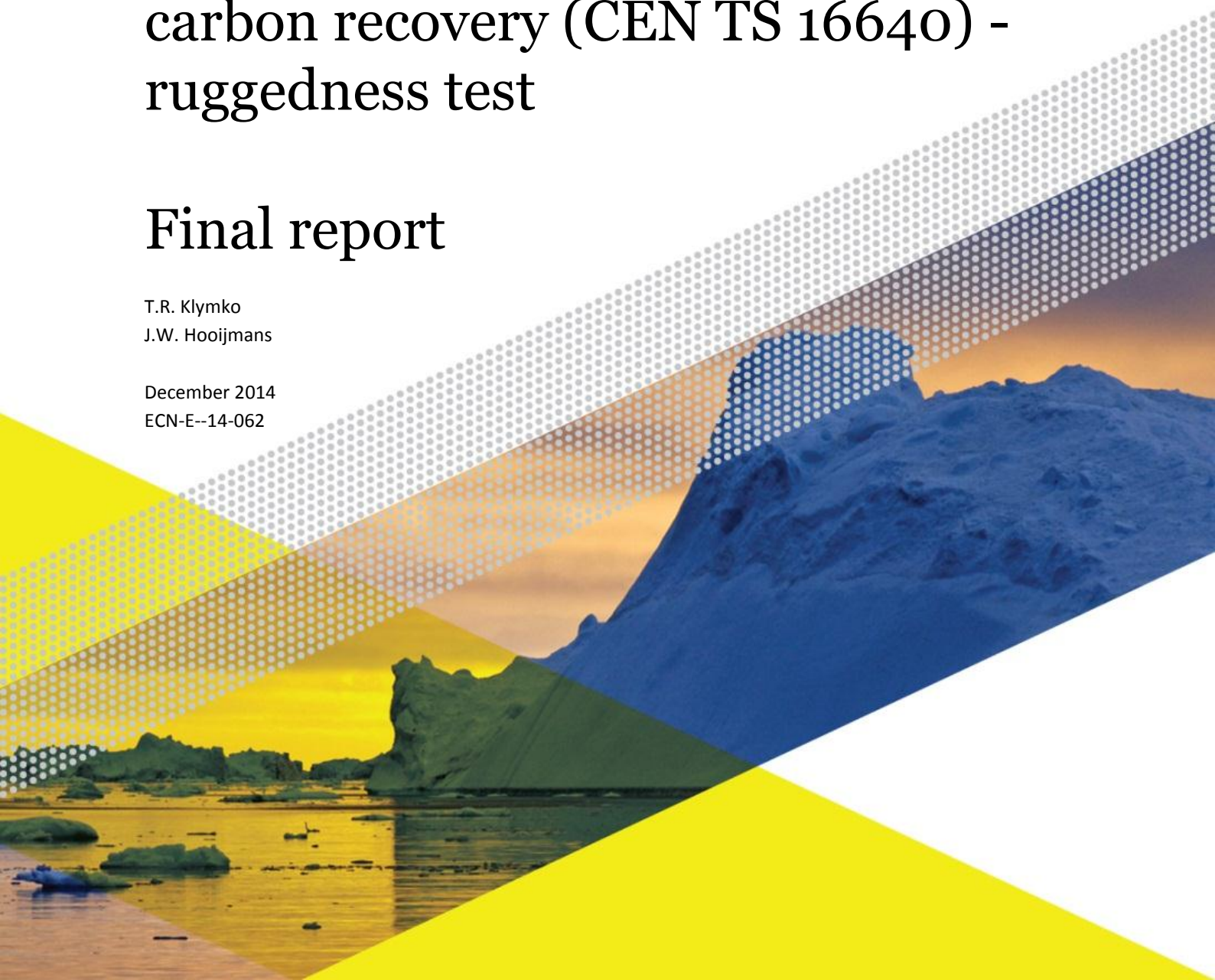
KBBPPS

Knowledge Based Bio-based Products'
Pre-Standardization -
Verification of method for total
carbon recovery (CEN TS 16640) -
ruggedness test

Final report

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KBBPPS

Knowledge Based Bio-based Products'

Pre-Standardization

Work package 3
Bio-based carbon content

Deliverable N° 3.4:

Verification of method for total carbon recovery (CEN TS 16640) - ruggedness test

Final report

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1. Publishable summary

Determination of the biogenic carbon of a finished product includes three steps:

- 1 – obtaining a representative sample,
- 2 – sample pretreatment and
- 3 – C14 analysis of the sample.

Our review on sampling standards was given in Deliverable 3.1. The method to determine total carbon and biobased carbon content was reviewed in Deliverable 3.2 that is converted into the technical specifications *CEN/TS 16640 "Bio-based products — Determination of the bio based carbon content of products using the radiocarbon method"*. Deliverable 3.3 presented the first stage of the verification of the method on the total carbon determination.

Deliverable 3.3 described the intermediate stage, approximately half of the challenging products were tested - volatile paints, volatile liquids, paint components, sun lotion and all its constituents.

Since the C14 determination techniques are very accurate and extensively tested, we verified only the total carbon recovery method which is based on complete combustion of a material. The results obtained on the total carbon fractions in each of the analysed materials were in good agreement with theoretical data (when known) and with data provided by products suppliers (when available). The results of our measurements indicated a very good accuracy.

Deliverable 3.4 of the European KBBPPS project presents the final results of evaluation of the method for the total carbon recovery in various products. Present document is a continuation of the intermediate report that was published as Deliverable 3.3. Current report includes the results of the ruggedness test on the next set of challenging products including bio-composites, bio-textile, boards made from wheat straw and gaseous materials.

The known C14 determination techniques are already standardized and exhibit a very high accuracy. Therefore a verification is needed only for the total carbon recovery method which is based on a complete combustion of a material. In relation to this, given report focuses exclusively on the ruggedness test that aims to check the method which is used to retrieve all available carbon from different materials and concerns only the treatment part and aims to check if it is possible to extract all available carbon (via conversion to CO₂) for different types of materials or products. Extration of available carbon can be evaluated via determining the combustion recovery, as it was explained in Deliverable 3.3 and will be repeated in Paragraphs 4 and 5 of this report.



For most tested materials, the CO₂ conversion results indicate a very high carbon recovery rate and thus prove the reliability of total carbon determination by the combustion and titration method. All performed measurements indicate a very good accuracy and a good agreement with data provided by sample suppliers (when available).

As a recommendation, special attention shall be paid to those materials or products which contain volatile components since omitting the carbon from the volatile part can lead to incorrect value for the total carbon content. When a special enhancer is used to ignite a product, it is of importance to know the carbon content that is added to the system from the enhancer. Inhomogeneity, as it was illustrated by one of the analyzed materials, can lead to several percents difference in the carbon content. In case a material is suspected to be inhomogeneous and a preparation of a representative sample is therefore of difficulty, it is better to analyze such material as a whole (when possible), or if possible, to analyze each component of it separately.



2. Introduction

CEN TC 411 concerns the bio-based products and a number of standards will be developed for the determination of the bio-based (carbon) content. The first standard to be developed is CEN TS 16640, devoted to the determination of the bio-based carbon content of products using the radiocarbon method. This procedure involves several steps: sampling, sample pre-treatment and ^{14}C determination. Each of these steps are described in CEN TS 16640. While sampling and ^{14}C determination are well tested and standardized, sample pretreatment is less studied with respect to various materials. The basis for sample preparation for ^{14}C analysis is to achieve complete carbon capture in the desired form recovered from the sample to be analyzed.

The method to be tested, will be used to liberate all carbon available in the sample, and is based on complete combustion of material. The method is applicable for those materials or products that are burnable.

In general, the material needs to be completely combusted in order to make all carbon free. This holds for solid, liquid, or gaseous materials. Some materials, as paints for instance, can be difficult to analyze due to the presence of volatile compounds which can cause problems by omitting some carbon which should be included. Ignition problems can also arise when combusting some materials. For such materials, the use of igniting enhancer is of necessity.

Several combustion options and the requirements are specified in Deliverable 3.2 of KBBPPS (TS 16640 "*Bio-based products — Determination of the bio based carbon content of products using the radiocarbon method*"). Typically combustion is performed in a calorimeter or in a tube furnace. An elemental analyser can also be used for this purpose. After combustion, all available carbon is collected as CO_2 in a suitable absorption medium: the CO_2 present in a representative stack gas sample is absorbed in an alkaline medium or transferred to a gas bag or lecture bottle. After sampling, the collected CO_2 is prepared for C^{14} analysis.

Practical difficulties can occur when sampling materials that are partially combustion resistant and therefore cannot be combusted completely (ceramics for example), or materials containing volatile components. Such materials are quite peculiar due to their inhomogeneity which cannot be removed by simple mechanical effects (crushing, shaking, centrifuging, mixing, etc.). Determination of their CO_2 content and then the C content demands a different approach which sometimes is not given in the Standards. If this is the case, then the method of sampling and testing has to be developed first on a laboratory scale.



3. List of materials and their characteristics

Table 1 represents a general list of materials and samples that were analyzed during the ruggedness test. The results of analysis of materials from suppliers A-D have already been reported in Deliverable 3.3 (intermediate report) and are repeated in this report in Table 2. The new results of analysis of Samples E-I will be given in Table 3 of this report. Note that the list includes both product constituents and finished products in different states (solid or liquid or gaseous or powder). According to the confidentiality agreement, product names and product suppliers are not mentioned in this report.

Table 1 List of materials for the ruggedness test

Supplier	Materials	Remark
A	Sun lotion and its constituents, 9 different samples in total	Reported in Deliverable 3.3, see Table 2 in this report
B	water-based matt paint, 5 cans of the same paint	Reported in Deliverable 3.3, see Table 2 in this report
C	2 different types of samples: bi- onaphta and biodiesel; 25 samples in total	Reported in Deliverable 3.3, see Table 2 in this report
D	3 types of samples: resins used to make paint; water based alkyd emulsion; water soluble linseed oil; 6 samples in total	Reported in Deliverable 3.3, see Table 2 in this report
E	3 different materails: wheat, maiz and rice straw panels	See Table 3 for the results
F	bio-composite board	See Table 3 for the results
G	bio-textiles	See Table 3 for the results
H	bio-gas	See paragraph 5 in this document
I	Bio-composite	See table 3 for the results
J	ceramic powder	n/a

This paragraph gives a brief description of Materials A-I that are mentioned in Table 1. Note that none of these materials demanded special laboratory storage conditions. Materials A-D have already been reported in Deliverable 3.3, but are also repeated in this report, in order to give a complete overview of the results and to make comparative conclusions on different types of materials.



3.1 Material A / Supplier A

Among materials A1-A10 there are the constituents of sun lotion and also the sun lotion itself as a final product.

Material A1

Glycerin EP/BP, 200 g

Materials A2

Yellow liquid, 1 glass bottle of 200 g, isopropyl isostearate

Material A3

Yellow liquid, 1 glass bottle of 200 g, Sorbitan Isostearate (and) Polyglyceryl-3Polyricinoleate

Material A4

Glass bottle of 200 g, yellowish liquid, 200 g, sorbitan sesquioleate

Material A5

1/3 of 200 ml bottle, white emulsion

Material A6

100 ml plastic box, white emulsion

Material A7

4 sample 50 g each in small metallic container, ethyl oleate

Material A8

4 sample 50 g each in small metallic container; Persea Gratissima

Material A9

Transparent liquid in glass bottle, 200 g, raw material, Squalane

Material A10

500 ml plastic bottle, white emulsion

3.2 Material B / Supplier B

Material B: the paint that is received is a water based matt paint.

5 cans of 500 ml each, 3.9 kg total weight;

The total percentage of binder is 4.03%, of which 2.9% acrylic binder and 1.13% renewable binder.

The volatile part in the paint is 34.6%.



3.3 Material C / Supplier C

25 samples all together that are divided in 5 groups.

- Group 1 contains C₅-C₁₀ isoparaffins and n-paraffins (bionaphta).
5 samples – referred to as C1 in Table 2 in this report.
- Group 2-Group 5 contain C₁₀-C₂₀ isoparaffins and n-paraffins (biodiesel).
Group 2 (5 samples), Group 3 (5 samples), Group 4 (5 samples) and Group 5 (5 samples) are referred to as materials C2 – C5 in Table 2 in given report.

All the samples are used as fuels or components in fuels and as solvents.

There is no detailed information about those specific samples for their carbon content. The supplier has analysed the carbon contents of similar samples. For similar sample as from Group 1 the carbon content was 83.9 wt% and for similar sample as from Groups 2-5 the carbon content was 84.6 wt%.

3.4 Material D / Supplier D

The samples are resins and emulsions that are used to make paint.

3 types of samples; 6 samples in total - labelled DA (2 samples), DB (2 samples) and DC (2 samples); 0.5 kg each.

Samples DA en DB are water based alkyd emulsion with a solids content around 45%.
Sample DC is a water soluble linseed oil with a solids content of 100% in the state as supplied.

All binders are for both interior and exterior application. The total carbon content is unknown.

3.5 Material E / Supplier E

Products received from Supplier E are panels made of rice straw (2 panels), maiz straw (2 panels) and wheat straw (2 panels). Each panel is approximately of 1x1 meter in size. The panels are made of approximately 97% bio-based material, and containg 3% of fossil resins.

The wheat panel that was analysed is mentioned as E in Table 3. Number 4 indicates that samples from area 4 from the wheat panel were taken for combustion and titration (see Annex B, Material E). Note that this is mentioned only for the informative reason since the composition of the board was proved to be homogeneous.



3.6 Material F / Supplier F

Bio-composite material was received as a bio-composite board. The supplier provided no information about the total carbon content of the material.

In Table 3 this material is referred to as F.

3.7 Material G / Supplier G

Bio-textile that was chosen for analysis was 100% hemp fibre of one color.

3.8 Material H / Supplier H

Bio-gas

Bio-gas was collected from a sewage sludge water treatment plant located in the Netherlands. The H_2S content of the biogas was 25 ppm. The bio-gas was sampled in special cylinders and pressurized to 2.5 bar that made it possible to transport and to store the gas for a longer time.

CH_4 content of the biogas was measured around 60%, CO_2 content is approximately 39%.

3.9 Material I / Supplier I

Material I is bio-based composite made of polypropylene and hemp fibre in equal proportions. According to the material supplier, the material is produced by compression molding. No additives nor bonding agents were used.

3.10 Material J / Supplier J

Bio-based ceramic material was unavailable at the time when the ruggedness test was performed



4. Sampling

The main principle of sampling is to have a homogeneous sample that is representative for the complete product.

When preparing a test sample, usually a bigger sample must be reduced to one or more test portions that are smaller than the original sample. The main principle that has to be hold for sample reduction is that the composition of the sample as taken on site shall not be changed during each stage of the sample preparation: each sub sample shall be representative of the original sample. Solid materials in most cases introduce no practical difficulties in obtaining a representative sample. Usually combustion method and CO₂ trapping is used to sample CO₂ and to determine the C content subsequently. Liquids should be either single phase or relatively homogeneous. The homogeneity in many cases can be achieved by stirring or sample centrifuging. Another approach is to separate the clear liquid and the sediment by centrifuging and then to examine them separately. In case of viscous liquids or materials with volatile components the transfer problems exaggerate weighing problems. Effort should be made to obtain the sample weight in the combustion tube, rather than transferring a previously weighed sample to the oxidation tube

Among analyzed materials A-I, only material F appeared to be inhomogeneous and demanded a special approach. This material, and material E for comparison, will be discussed here.

Material F is a bio-composite board shown in Figure 1. The panel was made by injection moulding. Similarly to the wheat panel, the composite board was tested on its homogeneity. Several samples were taken from various areas of the board, including the edges and inner and outer parts. Elemental composition of each sample showed a noticable variation in the carbon content thus indicating that we can not fullfil the requirement of having a representative sample when taking a sample from an arbitrary area of the board.

This is confirmed by the bio-based carbon results, showing also a large variability over the sub-samples taken.





	% C
Area 1	73.4
Area 2	75.3
Area 3	74.2
Area 4	72.0
Area 5	77.0
Area 6	71.7

Figure 1. Various areas of the bio-composite board that were chosen to make test samples in order to check the homogeneity of the board.

As can be seen from Table 3, the variation in carbon content between different areas of the material reaches even 6% (F-5 and F-6) that serves as evidence for the inhomogeneity of the material. Nevertheless, analyzing the test samples from each area individually, we obtain very high recovery rates. The material presented no difficulties with respect to combustion. It must be concluded that the injection moulding process resulted in an uneven distribution over the complete board of the adhesive and the wood particles.

In such cases, when carbon distribution over the material is not uniform, a combustion of the complete product would give the most reliable information about the (bio-based) carbon content.

Contrarily to the wheat panel that is described below, the composite board could not be used for the inter-laboratory test because of the inhomogeneity that makes a comparison of results from different laboratories impossible.

Among available materials from Supplier E, the wheat panel of approximately 1x1 meter size was chosen for the analysis. With this type of product, special attention should be paid to obtain a smaller representative sample that can be analyzed using an elemental analyzer or using a calorimeter. In order to ensure the homogeneity of the panel, sub-samples were taken from different areas of the panel (close to the edges and in the middle of the panel).



These samples were analyzed for their composition. The compositions of all samples were almost identical that proves the homogeneity of the panel. The maximum variation in carbon content that was observed between the different areas of the wheat panel, was 0.5%. It therefore allows us to take any arbitrary area of the panel for further analysis and the same time the requirement of the representativeness of the sampling is kept.

The wheat panel that was analyzed is shown as E in Table 3. Number 4 indicates that samples from area 4 from the wheat panel were taken for combustion and titration (see Annex B, Material E). Note that this is mentioned only for the informative reason since the composition of the board is homogeneous. The material indicated no difficulties when analyzing and shows a high recovery rate.



5. Carbon determination

As it was already described in Deliverable 3.3, the determination of total carbon and eventually the C14 content, the carbon that is present in the sample has to be converted to CO₂. The conversion is done by combustion in an oxygen rich environment. If necessary, a combustion aid can be used to ensure complete oxidation of the C to CO₂. Following the technical specification *CEN/TS 16640*, different preparation techniques of samples are required, depending on the method that is used for the C14 determination. When LSC (liquid scintillation counting) method is used, then CO₂ shall be collected in a cooled mixture of carbamate solution and a suitable scintillation liquid. When BI (beta ionisation) or AMS (accelerated mass spectrometry) methods are used, then the CO₂ shall be collected in 4M NaOH solution or on a suitable solid absorber. More details on the requirements are given in earlier mentioned technical specifications.

Two different approaches have been used to define the total carbon content in the products that were tested.

1. Determination of total C fraction using an elemental analyser.
2. Combustion in a calorimeter with subsequent CO₂ collecting in NaOH and determination of the total C fraction by titration.

These methods are briefly described in the next sub-paragraphs.

5.1 Elemental analysis

Elemental CHN-O analyser determines the percentages of carbon (the analyser also determines how much hydrogen and nitrogen is present). The key components of CHN-O analyser are auto-sampler, combustion reactors, chromatographic column, and thermal conductivity detector (TCD). The working principle is based on complete combustion of a sample in a tin capsule. To determine the oxygen content, high-temperature pyrolysis is used. To measure C, N and H, a sample is burned in a small excess of oxygen at approximately 1700°C. At this temperature the sample will be converted to oxides, salts and metal - the combustion products are separated by a chromatographic column which converts the compound in the form of N₂, CO₂, and H₂O that are detected by the thermal conductivity detector. The latter gives an output signal proportional to the concentration of the individual components of the mixture. The instrument is calibrated with the analysis of standard compounds. This method has greatest utility in finding out percentages of C, H, N in compounds which are generally combustible at 1700°C.

When using the CHN-O analyser, the following rules must be kept:

- ✓ Sample must be pure and all contaminants must be removed.
- ✓ Sample weight should be in between 1-2mg.
- ✓ Liquid samples should have constant weight.



5.2 Combustion and titration

For combustion in the calorimeter at least 0.5g of material is needed. After combustion the subsequent CO₂ gathering in NaOH and determination of the total C fraction by titration. For combustion of the sample in a calorimetric bomb, any test method such as ISO 1716, ISO 1928 or EN 15400 can be used. After the complete combustion, the combustion gas is collected in a 200mL wash bottle, containing 1M NaOH and the titration of the solution is done with 0.1M HCl.

Practical difficulties can occur when sampling materials that are partially combustion resistant and therefore cannot be combusted completely (ceramics for example or materials containing a large water content), or materials containing volatile components. Such materials are quite peculiar due to their inhomogeneity which cannot be removed by simple mechanical effects (crushing, shaking, centrifuging, mixing, etc.) Determination of their CO₂ content and then the C content demands a different approach which sometimes is not given in the Standards. If this is the case, then a method of sampling and testing has to be developed first on a laboratory scale.

In this paragraph, the most challenging materials with respect to carbon recovery, will be reviewed. These are paints, materials with low carbon content and a high water content, and materials with a volatile component. Since combustion of gases is not trivial and the methods described above are not applicable, in this section the approach and the experimental set-up that has been used at ECN is also presented. Treatment of ceramic materials accordingly to ISO 21068 is describe as well.

5.2.1 Paints, materials with low carbon amount and materials with a volatile component

For combustion of materials A6 (sun lotion), B (matt paint) , DA and DB (resins and emulsions used to make paint) it was necessary to use the combustion enhancer (material was put in the polyethylene bags with known carbon content) in order to achieve better combustion in a calorimeter. Alternatively, benzoic acid can also facilitate the complete burning of these materials.

Material B is matt paint with the volatile fraction of 34.6%. Material B indicated difficulties with its ignition and therefore polyethylene were used in order to ensure the complete combustion of Material B in a calorimeter. A low recovery rate of only 50% was determined for material B. It can be explained by low carbon content and combustion difficulties.

For material A6 (sun lotion), the carbon recovery rate is lower, due to lower amount of carbon in that material. For A6, an enhancer was needed to improve its combustion efficiency. At ECN, polyethylene bags with known high carbon content (at least 80%) were used to enhance the combustion.



For Material DA and DB (resins used to make paints), the use of polyethylene bags with known carbon content was necessary in order to achieve a good recovery rates. For Materials DC, no combustion enhancer was necessary to reach good carbon recovery rates.

5.2.2 Biogas combustion

Sampling gas at the customer site

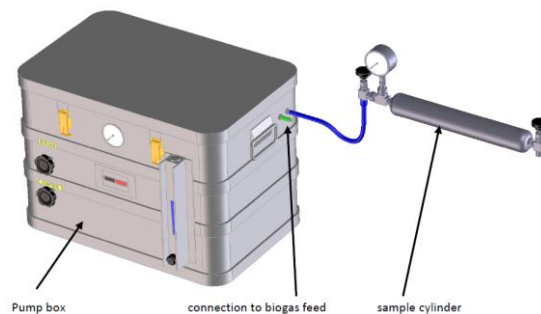


Figure 2. Schematic drawing of a gas sampling installation

The pump box is connected to the biogas source. The pump box is needed to increase the pressure of the biogas source, typical 0.1 bar gauge, to 3 bar gauge. Both valves of the sample cylinder are opened to shortly flush the cylinder with biogas. Then the downstream valve is closed and the cylinder is filled to approximately 3 bar. The upstream valve and the biogas feed valve are closed, and the pump box disconnected from the biogas feed. The sample cylinder is disconnected from the pump box and the sample cylinder is transported for further processing.

Processing the gas sample

The process is burning the biogas with an excess of air and capturing all carbon on an ascarite column (NaOH on a solid absorber). To check if the conversion is complete a gas analyser is installed at the exit. The reactor is filled with 6mm ball of alumina to create more length (time) for the gas to pass through the hot zone. This will ensure complete combustion. Schematically the installation is shown in Figure 3.



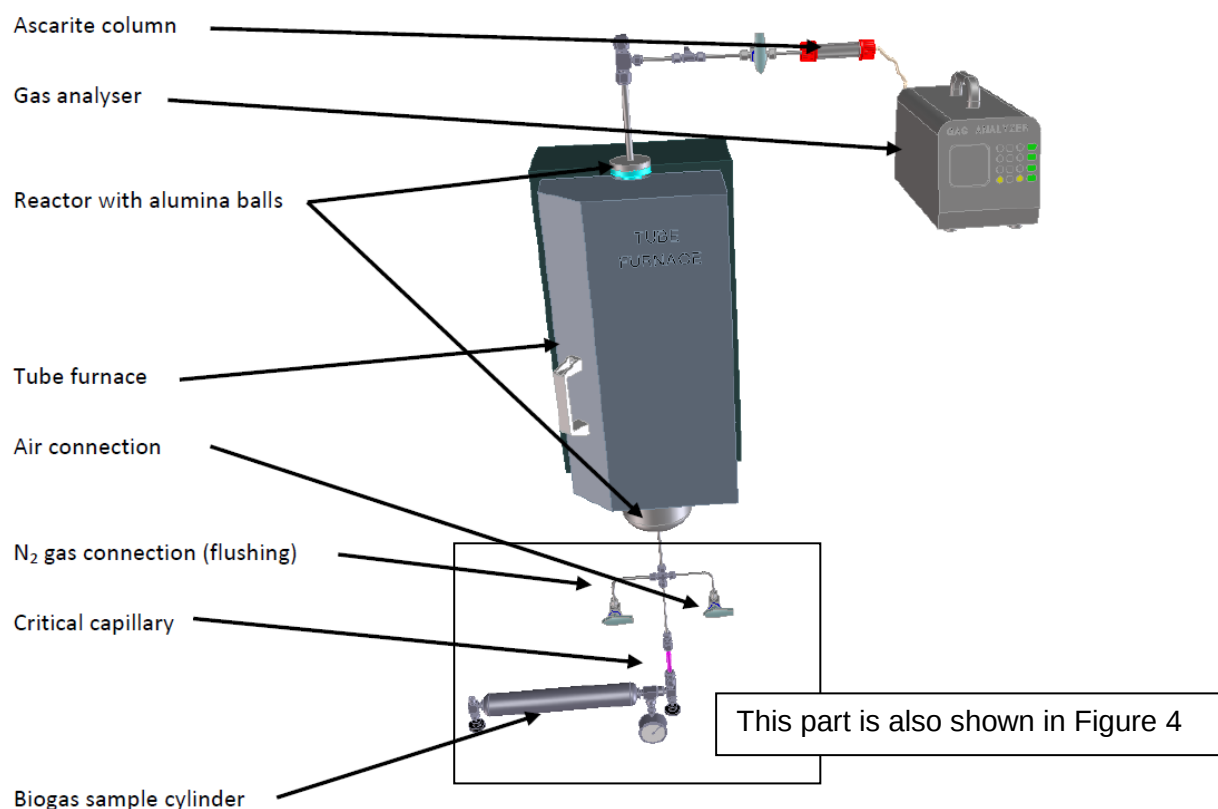


Figure 3. Gas processing installation

Procedure and details

The tube furnace is heated to 850°C with a N₂ flow of 1.5 l/min. When the temperature is reached the sample cylinder is connected. The N₂ flow is switched off and the air flow is switched on, the air flow is also 1.5 l/min. After a minute the valve of the sample cylinder is opened and the biogas will enter the system with a flow rate of 0.17 l/hr. The biogas flow rate is regulated by the critical capillary. The manometer is used to check when the sample cylinder is empty and the process can be stopped (see Figure 4).



Figure 4. Part of gas processing installation (see Figure 2 for a complete schematic overview)



After leaving the furnace section the gas passes through an ascarite column which binds all the carbon in the form of a carbonate. To check if the process works the exit gas is analysed. No CO_2 must be detected at this point. The exhaust is connected to a vent system (not shown).

There is a sample point in front of the ascarite column, this enables taking a sample of the burnt gas before the carbon is captured (see Figure 5).

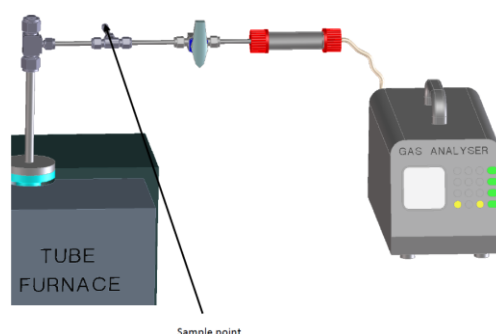


Figure 5. Position of sampling point (ascarite column is in red)

The ascarite column is brought to the ECN lab for analysis.

At the lab the ascarite will be flushed with double demineralised water into a polyethylene vessel. After this the alkaline solution will be titrated with 0.1M HCL in order to determine the amount of trapped CO_2 in the form of carbonate. The alkaline solution is used for the ^{14}C isotope determination.

5.2.2.1 Treatment of ceramic materials

Since when performing the ruggedness test, bio-based ceramic materials were unavailable and tested, in this paragraph we refer to ISO 21068 that describes methods and experimental set-up and that can be used for determining the total carbon content in ceramic materials. The same analysis shall be also possible for bio-ceramic materials.

ISO 21068: Chemical analysis of silicon carbide containing raw materials and refractory products. Part 1: general information and sample preparation. Part 2: Determination of loss on ignition, total carbon, free carbon and silicon carbide, total and free silica and total and free silicon.

In particular, the second part of ISO 21068 covers the full range of analysis from pure silicon carbide to oxidic refractory composition with a low content of silicon carbide and/or nitrides. This part of ISO 21068 provides methods to distinguish between different carbon-bound types like total carbon (C_{total}) and free carbon (C_{free}) and derives from these two the silicon carbide content. The method for chemical analysis of SiO_2 , total Si, oxygen and nitrogen and other oxidic-bound metals which typically occur in the materials is also described within the Standard.



Accordingly to the referred standard, the total carbon content can be determined by the following combustion methods and detection techniques:

By combustion method using :

resistance furnace

in oxygen and lead borate as decomposing agent

in oxygen and tin powder as decomposing agent

induction furnace

in oxygen and metallic powder as decomposing agent

By detection method using:

- coulometry
- gravimetry using resistance furnace and using high-frequency induction furnace
- conductometry
- infrared absorption method and resistance furnace combustion
- thermal conductivity method and induction furnace combustion

The standard also describes the combustion and detection techniques and the reagents to use in a resistance and induction furnaces. More technical details can be found in the standard.

5.3 Carbon recovery

Carbon recovery percentage is determined as a ratio between the carbon fraction obtained by these two approaches: from titration and from CHNO analyser.

Special attention should be given to the use of combustion aids. For the carbon recovery percentage, the total carbon amount from the combustion aid should be subtracted from the measured amount of carbon by titration. This should also be done when measuring the C14 content, and it is essential to measure the C14 content of the combustion aid separately, to make the bio-based carbon content determination accurate.

5.4 Data analysis

For each analysed sample we perform multiple measurements (see Annex A and Annex B) to determine its carbon (also hydrogen, nitrogen and oxygen) content. Measurement results that are presented in the next paragraph for each material are the averaged numbers from multiple measurements.



Based on the number of measurements, a relative standard deviation due to a random error is calculated for each analysed material. Besides a random error, also a systematic error (in literature sometimes referred to also as percent error) is determined as a relative difference between measured and theoretical accepted value. If the theoretical accepted value for some compound is unknown, then the systematic error remains un-estimated. Note that for most materials their theoretical formula was unknown and therefore the systematic error remained unestimated. Also, for all materials mentioned in Table 3 the suppliers did not provide data on carbon content that makes it impossible to compare stated and measured carbon values for these materials.

Relative standard deviation RSD is calculated as:

$$RSD = \frac{SD}{\bar{X}} 100\%$$

$$SD = \sqrt{\frac{\sum_{i=1}^N (X_i - \bar{X})^2}{N-1}}$$

where $\bar{X}$ is averaged experimentally measured quantity,

SD is standard deviation

N is number of measurements.



6. Results

The overall summarizing results obtained by two methods (EA and combustion and titration) that are described above are given in Table 2 and Table 3. Table 2 represents the results of Materials A-D that were already reported in Deliverable 3.3 at the intermediate stage of the process. Table 3 represents new materials that are reported here for the first time. More details are available in Appendix A for the results obtained by using the CHNO analyser and in Appendix B for CO₂ conversion and titration results. Due to the confidentiality agreement with product suppliers, product names are mentioned in this report in anonymous way. A systematic error given in Table 2, represents a relative deviation of measured and theoretically known values for the total carbon content of each of analyzed materials. For materials where the theoretical carbon content is unknown, the systematic error remains unestimated (n/a in Table 2).



Table 2 Carbon content as estimated by a supplier, measured and theoretical values (Materials A-D)

Material	Carbon content from supplier*	Biobased content from supplier	Carbon content measured average (CHNO)	SD	Systematic error (CNHO)	Carbon content theoretical	Carbon content measured average (titration)	SD	Systematic error (titration)	Carbon recovery percentage
A1	39%	100%	38.9%	0.2%	-0.72%	39.1%	37.1%	1.8%	-5.3%	95%
A2	85%	65%	77.2%	0.7%	-0.13%	77.3%	74.5%	1.1%	-3.7%	96%
A3	65%	100%	73.1%	0.3%	n/a	unknown	74.5%	2.2%	n/a	100%
A4	75%	100%	71.6%	0.2%	6.03%	67.5%	69.0%	1.0%	2.3%	96%
A5	45%	87%	42.6%	0.2%	n/a	unknown	39.9%	1.3%	n/a	94%
A6	24%	-	24.8%	0.2%	n/a	unknown	19.5%	0.1%	n/a	79%
A7	78%	90%	76.9%	0.4%	-0.63%	77.4%	73.9%	1.3%	-4.6%	96%
A8	76%	100%	77.2%	0.4%	n/a	unknown	75.2%	1.9%	n/a	97%
A9	85%	100%	85.8%	0.5%	0.62%	85.3%	82.7%	1.6%	-3.1%	97%
A10	45%	100%	42.4%	0.1%	n/a	unknown	39.8%	0.8%	n/a	94%
B	-	-	10.5%	0.1%	n/a	unknown	5% max	-	n/a	50% max
C1	~ 83.9%	-	83.8%	0.7%	n/a	unknown	74.2%	0.3%	n/a	89%
C2	~ 84.6%	-	84.2%	0.7%	n/a	unknown	79.4%	0.7%	n/a	94%
C3	~ 84.6%	-	84.0%	0.5%	n/a	unknown	80.3%	0.9%	n/a	96%
C4	~ 84.6%	-	84.7%	0.2%	n/a	unknown	81.2%	1.2%	n/a	96%
C5	~ 84.6%	-	84.4%	0.2%	n/a	unknown	83.4%	0.2%	n/a	99%
D-A1	-	-	44.7%	0.3%	n/a	unknown	34.3%	0.4%	n/a	77%
D-A2	-	-	44.0%	0.4%	n/a	unknown	35.7%	0.6%	n/a	81%
D-B1	-	-	40.2%	0.4%	n/a	unknown	33.0%	0.3%	n/a	82%
D-B2	-	-	40.4%	0.2%	n/a	unknown	32.0%	0.2%	n/a	80%
D-C1	-	-	70.5%	0.2%	n/a	unknown	64.3%	1.1%	n/a	91%
D-C2	-	-	70.6%	0.3%	n/a	unknown	63.5%	0.4%	n/a	90%

*Total carbon content as estimated by a supplier ($\pm 5\%$ for A1-A9 samples; approximate values for C1-C5 samples).

SD – standard deviation



Table 3 Carbon content for Materials E-G, from CHNO analyzer and from combustion and titration

Material	Carbon content measured average (CHNO)	Carbon content measured average (titration)	Carbon recovery rate
E	42.9% $\pm$ 0.3%	40.1% $\pm$ 0.7%	93%
F-1	73.4%	67.9% $\pm$ 0.3%	93%
F-2	75.3%	70.0% $\pm$ 0.4%	93%
F-3	74.2%	73.7% $\pm$ 1.6%	99%
F-4	72.0%	72.3% $\pm$ 0.3%	100%
F-5	77.0%	72.0% $\pm$ 0.4%	94%
F-6	71.7%	70.5% $\pm$ 0.6%	98%
G	41.7% $\pm$ 0.2%	38.9% $\pm$ 0.8%	93%
I	64.8 $\pm$ 0.8, %	56.6 $\pm$ 0.4, %	87%

Remark: no data on carbon content from product suppliers and no theoretical values are available for materials E-I given in Table 3.

For materials A, C, D, E, F, G and I their CO₂ conversion results indicate a very high carbon recovery rate (see Table 2 and 3) and thus prove the reliability of total carbon recovery by combustion and titration method. All performed measurements indicate a very good repeatability and a good agreement with data provided by sample suppliers (when available). Material B indicated comparably low recovery rate. It is related to a low carbon content and a high water content, indicating that these types of materials have combustion difficulties.

Materials A were sun lotion and its components. All analysed materials A1-A10 (except A6) presented no difficulties with respect to combustion and indicate very high recovery rates. For material A6, the carbon recovery rate is lower, due to lower amount of carbon in that material. For A6, a combustion enhancer was needed to improve its combustion efficiency. At ECN, polyethylene bags with known high carbon content (at least 80%) were used to enhance the combustion.



Material B is matt paint with the volatile fraction of 34.6%. Material B showed difficulties with its ignition and therefore polyethylene was used in order to ensure the complete combustion of Material B in a calorimeter. Even though, a low recovery rate of only 50% was determined for material B.

Material C presented no difficulties when analyzing and show a very good recovery rates.

For Material DA and DB, the use of polyethylene bags with known carbon content was necessary in order to achieve good recovery rates. For Materials DC, no combustion enhancer was necessary to reach good carbon recovery rates.

Material G is textile, with no combustion difficulties and a high recovery rate.

For Material I, the recovery rate was 87.4% when no combustion enhancer was used. Combustion efficiency can be improved by using benzoic acid or polyethylene bags.

Gaseous samples can be challenging for combustion and the CO₂ trapping. An experimental set-up that has been used at ECN is described in this report and can be used as an example.

Performing the ruggedness test, no bio-ceramic material was available. Since ceramic materials possess very high resistance to combustion, they also require a special treatment. Part 1 and 2 of ISO 21068 are referred to and recommended to use for analysis of ceramic materials: ISO 21068 - Chemical analysis of silicon carbide containing raw materials and refractory products. Part 1: general information and sample preparation. Part 2: Determination of loss on ignition, total carbon, free carbon and silicon carbide, total and free silica and total and free silicon.



7. Discussions and recommendations

Various materials in different states (solids, liquids (including volatile components), emulsions with dispersed particles, textiles, composites and gases) were analyzed in the framework of the performed ruggedness test.

For materials with known theoretical values for their carbon content, there is a good agreement with measured and theoretical values. Note that for most materials that have been analysed there is no information on their theoretical carbon fraction. For such materials the systematic error remains un-estimated. Systematic error with a “-” sign in Table 2 indicates that measured average value is smaller than theoretical accepted value for a given compound.

It is observed that for the same materials, the systematic error when the carbon content was determined directly by the CHNO analyzer, is smaller (in absolute values) than the systematic error for carbon content obtained by titration after combustion. This can indicate that combustion in CHNO analyzer is more complete, but the difference between the carbon content obtained by combustion and titration, and directly from the CHNO analyzer, is negligible for all analyzed materials. In order to have a satisfactory result on the total carbon content, a recovery percentage of at least 95% is recommended. However, it is observed that for a number of samples a recovery of at least 90% is achieved. For these materials conversion aids should probably be used, or more homogeneous samples should be obtained.

As recommendation, special attention shall be paid to those materials or products which contain volatile components since omitting the carbon from the volatile part can lead to a wrong value for the total carbon content and especially to the bio-based carbon content. When a special enhancer is used to ignite a product, it is of important to know the carbon content (and the bio-based carbon content) that is added to the system from the enhancer. Inhomogeneity, as it was illustrated by Material F, can lead to several percents difference in carbon content. In case a material is suspected to be inhomogeneous and preparation of a representative sample is therefore difficult, it is better to analyze the part of such a material as a whole when possible, or if known, to analyze each component of it.



8. Next steps

Round robin assessment is initiated and involves 9 different laboratories that will perform the total carbon analysis, the C14 analysis and combustion. Each laboratory received 6 identical samples that include liquids, solids, gaseous, paint and emulsion samples.

Two separate reports are being prepared:

- Report on the interlaboratory test concerning a difficult product – the results of the proficiency test (Deliverable 3.5 of KBBPPS).
- Performance characteristics for horizontal bio-based carbon content standard - the results of the round robin test (Deliverable 3.1 of Open-Bio).



Annex A Results from CHN-O analyser

Material A1Fout! Ongeldige koppeling.

Material A2Fout! Ongeldige koppeling.

Material A3Fout! Ongeldige koppeling.
Fout! Ongeldige koppeling.

Material A4Fout! Ongeldige koppeling.

Material A5Fout! Ongeldige koppeling.

Material A6

Sun lotion - ready product					
Measurement	%N	%C	%H	%O	Sum
1	0.02	24.44	10.20	Not measured	
2	0.03	24.90	10.50	Not measured	
3	0.03	24.81	10.60	Not measured	
4	0.03	24.82	10.40	Not measured	
5	0.03	25.04	10.22	Not measured	
6					
Measured average	0.03	24.80	10.38	-	35.22
Standard deviation	0.00	0.22	0.17		
%RSD	14.19	0.89	1.66		
No of measurements	5	5	5		
Theoretical value	-	-	-	-	



Material A7[Fout! Ongeldige koppeling.](#)

Material A8[Fout! Ongeldige koppeling.](#)

Material A9[Fout! Ongeldige koppeling.](#)



Material A10

sun lotion component					
Measurement	%N	%C	%H	%O	Sum
1	0.03	42.21	6.95	not measured	
2	0.04	42.55	6.73	not measured	
3	0.03	42.42	6.92	not measured	
4	0.04	42.39	7.00	not measured	
5	0.04	42.50	6.95	not measured	
6	0.04	42.41	6.93	not measured	
7	0.03	42.50	7.02	not measured	
Measured average	0.04	42.42	6.93		49.39
Standard deviation	0.01	0.11	0.09		
%RSD	14.07	0.26	1.36		
No of measurements	7	7	7		
Theoretical value	-	-	-	-	

Material B Fout! Ongeldige koppeling.

Material C1 Fout! Ongeldige koppeling.



Material C2[Fout! Ongeldige koppeling.](#)

Material C3[Fout! Ongeldige koppeling.](#)

Material C4[Fout! Ongeldige koppeling.](#)



Material C5 Fout! Ongeldige koppeling.**Material DA1**

sample DA1 (alkyd emulsion)					
Measurement	%N	%C	%H	%O	Sum
1	2.29	45.05	10.48	not measured	
2	1.73	45.03	10.58	not measured	
3	0.91	44.97	10.62	not measured	
4	0.67	44.70	10.50	not measured	
5	1.82	44.71	10.53	not measured	
6	1.84	44.29	10.64	not measured	
7	1.03	44.22	10.62	not measured	
Measured average	1.47	44.71	10.57		56.75
Standard deviation	0.60	0.34	0.06		
%RSD	40.69	0.77	0.61		
No of measurements	7	7	7		
Theoretical value	-	-	-	-	

Material DA2

sample DA2 (alkyd emulsion)					
Measurement	%N	%C	%H	%O	Sum
1	3.10	44.18	10.53	not measured	
2	1.86	43.60	10.51	not measured	
3	1.29	43.68	10.56	not measured	
4	0.99	43.75	10.71	not measured	
5	0.99	44.06	10.61	not measured	
6	0.98	44.77	10.50	not measured	
Measured average	1.53	44.01	10.57		56.11
Standard deviation	0.84	0.44	0.08		
%RSD	54.67	1.00	0.78		
No of measurements	6	6	6		
Theoretical value	-	-	-	-	

Material DB1

sample DB1 (alkyd emulsion)					
Measurement	%N	%C	%H	%O	Sum
1	1.72	40.61	10.86	not measured	
2	2.78	40.53	10.85	not measured	
3	2.30	40.08	10.99	not measured	
4	1.54	39.67	11.05	not measured	
5	3.00	40.08	10.85	not measured	
6	3.60	40.02	10.49	not measured	
Measured average	2.49	40.16	10.85		53.50
Standard deviation	0.79	0.35	0.19		
%RSD	31.59	0.87	1.78		



No of measurements	6	6	6		
Theoretical value	-	-	-	-	

Material DB2

sample DB2 (alkyd emulsion)					
Measurement	%N	%C	%H	%O	Sum
1	3.71	40.25	10.68	not measured	
2	2.67	40.23	10.76	not measured	
3	1.56	40.26	10.76	not measured	
4	2.37	40.57	10.63	not measured	
5	2.32	40.44	10.61	not measured	
Measured average	2.53	40.35	10.69		53.56
Standard deviation	0.78	0.15	0.07		
%RSD	30.75	0.37	0.64		
No of measurements	5	5	5		
Theoretical value	-	-	-	-	

Material DC1

sample DC1 (linseed oil)					
Measurement	%N	%C	%H	%O	Sum
1	0.05	70.53	10.49	not measured	
2	0.04	70.78	10.50	not measured	
3	0.04	70.56	10.48	not measured	
4	0.04	70.61	10.57	not measured	
5	0.05	70.30	10.44	not measured	
6	0.05	70.49	10.63	not measured	
Measured average	0.05	70.54	10.52		81.11
Standard deviation	0.00	0.16	0.07		
%RSD	5.43	0.22	0.67		
No of measurements	6	6	6		
Theoretical value	-	-	-	-	



Material DC2

sample DC2 (linseed oil)					
Measurement	%N	%C	%H	%O	Sum
1	0.08	70.62	10.05	not measured	
2	0.05	70.34	10.50	not measured	
3	0.09	70.45	10.26	not measured	
4	0.07	70.45	10.23	not measured	
5	0.07	70.95	10.21	not measured	
6	0.06	70.95	10.37	not measured	
Measured average	0.07	70.63	10.27		80.96
Standard deviation	0.01	0.27	0.15		
%RSD	19.28	0.38	1.47		
No of measurements	6	6	6		
Theoretical value	-	-	-	-	

Material E

Wheat panel					
Measurement	%N	%C	%H	%O	Sum
1-1	1.02	42.83	5.75	39.43	
1-2	0.99	42.16	5.56	40.85	
2-1	1.09	42.98	5.79	40.86	
2-2	1.11	43.13	5.82	41.57	
3-1	1.04	42.83	5.67	42.51	
3-2	1.04	43.04	5.69	42.12	
4-1	1.05	42.97	5.75	41.66	
4-2	1.08	42.86	5.64	41.56	
5-1	1.09	43.10	5.65	42.10	
5-2	1.10	43.06	5.76	41.52	
Measured average	1.06	42.90	5.71	41.42	91.08
Standard deviation	0.04	0.28	0.08	0.87	
%RSD	3.70	0.65	1.38	2.11	
No of measurements	10	10	10	10	
Theoretical value	-	-	-	-	



Material F

Composite board					
Measurement	%N	%C	%H	%O	Sum
1	0.11	73.43	11.89	not measured	
2	0.08	75.33	12.26	not measured	
3	0.08	74.23	12.05	not measured	
4	0.08	71.99	11.53	not measured	
5	0.11	76.99	0.00	not measured	
6	0.00	71.72	11.44	not measured	
Measured average	0.08	73.95	9.86		83.89
Standard deviation	0.04	2.02	4.84		
%RSD	52.61	2.73	49.09		
No of measurements	6	6	6		
Theoretical value	-	-	-	-	

Material G

Textile					
Measurement	%N	%C	%H	%O	Total
Textile-1	0.00	41.60	6.22	not measured	
Textile-2	0.00	41.98	6.27	not measured	
Textile-3	0.00	41.42	6.15	not measured	
Textile-4	0.00	41.93	6.24	not measured	
Textile-5	0.00	41.89	6.22	not measured	
Measured average	0.00	41.76	6.22		47.98
Standard deviation	0.00	0.24	0.04		
%RSD		0.58	0.69		
No of measurements	5	5	5		
Theoretical value	-	-	-	-	

Material I

Hemp-polypropylene composite					
Measurement	%N	%C	%H	%O	Total
1	0.28	64.88	10.54	not measured	
2	0.25	64.50	10.52	not measured	
3	0.24	64.32	10.41	not measured	
4	0.24	66.19	10.81	not measured	
5	0.22	64.00	10.39	not measured	
6	0.23	65.13	9.86	not measured	
Measured average	0.24	64.84	10.42		75.50
Standard deviation	0.02	0.78	0.32		
%RSD	8.26	1.20	3.04		
No of measurements	6	6	6		
Theoretical value	-	-	-	-	



Annex B Results from conversion to CO₂ and titration

				titration				total	total	weight	C	C%	Recovery
A1	volume ml	volume	s.g.(24°C)	weight	1ste e.p.	2de e.p.	Δ	mg CO ₂	g Co2	sample		from CHN	respectively to CHN
	1.0	196.45	1.04	1.04	1.23	2.08	0.85	750	0.8	0.53	38.60%	38.850	99%
	1.0	196.45	1.02	1.02	1.66	2.51	0.84	742	0.7		38.19%	38.850	98%
	1.0	196.45	1.03	1.03	0.79	1.62	0.83	734	0.7		37.79%	38.850	97%
	1.0	200.72	1.03	1.03	0.87	2.28	1.41	1271	1.3	1.01	34.31%	38.850	88%
	1.0	200.72	1.04	1.04	0.99	2.54	1.54	1391	1.4		37.57%	38.850	97%
	1.0	200.72	1.04	1.04	1.07	2.56	1.49	1338	1.3		36.13%	38.850	93%
	1.0	200.72	1.04	1.04	1.00	2.51	1.50	1355	1.4		36.59%	38.850	94%
	1.0	200.72	1.03	1.03	1.01	2.54	1.53	1382	1.4		37.31%	38.850	96%
											37.06%		95%
A2													
	1.0	198.62	1.04	1.04	1.33	2.79	1.46	1300	1.3	0.48	73.85%	77.200	96%
	1.0	198.62	1.04	1.04	1.22	2.67	1.45	1293	1.3		73.48%	77.200	95%
	1.0	198.62	1.04	1.04	0.96	2.40	1.44	1287	1.3		73.14%	77.200	95%
	1.0	198.9	1.04	1.04	0.88	2.58	1.71	1523	1.5	0.55	75.50%	77.200	98%
	1.0	198.9	1.04	1.04	0.96	2.68	1.71	1529	1.5		75.82%	77.200	98%
	1.0	198.9	1.04	1.04	0.88	2.58	1.69	1511	1.5		74.95%	77.200	97%
											74.46%		96%
A3													
	1.0	202.95	1.02	1.02	1.03	2.63	1.60	1459	1.5	0.52	76.53%	73.060	105%
	1.0	202.95	1.02	1.02	0.99	2.60	1.61	1466	1.5		76.86%	73.060	105%
	1.0	202.95	1.02	1.02	0.58	2.17	1.59	1449	1.4		75.99%	73.060	104%
	1.0	199.33	1.05	1.05	0.75	2.26	1.51	1348	1.3	0.506	72.65%	73.060	99%
	1.0	199.33	1.05	1.05	0.66	2.16	1.50	1343	1.3		72.41%	73.060	99%
	1.0	199.33	1.05	1.05	1.15	2.65	1.50	1346	1.3		72.53%	73.060	99%
											74.49%		102%
A4													
	1.0	205.23	1.04	1.04	1.26	2.67	1.41	1299	1.3	0.503	70.45%	71.570	98%
	1.0	205.23	1.04	1.04	1.30	2.67	1.37	1260	1.3		68.32%	71.570	95%
	1.0	205.23	1.04	1.04	0.95	2.36	1.41	1294	1.3		70.18%	71.570	98%
	1.0	207.03	1.03	1.03	1.11	2.50	1.39	1291	1.3	0.515	68.39%	71.570	96%
	1.0	207.03	1.04	1.04	1.13	2.53	1.39	1295	1.3		68.59%	71.570	96%
	1.0	207.03	1.04	1.04	0.98	2.37	1.39	1289	1.3		68.25%	71.570	95%
											69.03%		96%
A7													
	1.0	204.84	1.04	1.04	1.04	2.38	1.33	1226	1.2	0.444	75.32%	76.930	98%
	1.0	204.84	1.04	1.04	0.90	2.24	1.33	1224	1.2		75.21%	76.930	98%
	1.0	204.84	1.02	1.02	0.63	1.94	1.32	1209	1.2		74.28%	76.930	97%
	1.0	204.23	1.04	1.04	0.81	2.31	1.50	1379	1.4	0.515	73.04%	76.930	95%
	1.0	204.23	1.04	1.04	1.00	2.51	1.51	1383	1.4		73.23%	76.930	95%
	1.0	204.23	1.04	1.04	1.25	2.74	1.49	1364	1.4		72.22%	76.930	94%
											73.88%		96%
A8													
	1.0	205.19	1.06	1.06	0.68	2.25	1.56	1440	1.4	0.504	77.90%	77.220	101%
	1.0	205.19	1.04	1.04	1.14	2.63	1.49	1370	1.4		74.13%	77.220	96%
	1.0	205.19	1.04	1.04	0.80	2.34	1.54	1418	1.4		76.71%	77.220	99%
	1.0	205.19	1.04	1.04	0.70	2.25	1.55	1427	1.4		77.21%	77.220	100%
	1.0	206.13	1.05	1.05	0.63	2.12	1.49	1377	1.4	0.502	74.82%	77.220	97%
	1.0	206.13	1.04	1.04	0.70	2.15	1.44	1336	1.3		72.56%	77.220	94%
	1.0	206.13	1.04	1.04	0.85	2.32	1.48	1367	1.4		74.27%	77.220	96%
	1.0	206.13	1.04	1.04	0.62	2.09	1.47	1356	1.4		73.70%	77.220	95%
											75.16%		97%
A9													
	1.0	205.88	1.03	1.03	0.78	2.45	1.67	1544	1.5	0.502	83.86%	84.840	99%
	1.0	205.88	1.04	1.04	0.93	2.61	1.67	1546	1.5		83.98%	84.840	99%
	1.0	205.88	1.05	1.05	0.77	2.45	1.68	1554	1.6		84.42%	84.840	100%
	1.0	203.48	1.04	1.04	1.00	2.63	1.63	1488	1.5	0.496	81.82%	84.840	96%
	1.0	203.48	1.04	1.04	0.90	2.49	1.59	1454	1.5		79.97%	84.840	94%
	1.0	203.48	1.05	1.05	0.59	2.25	1.66	1514	1.5		83.25%	84.840	98%
	1.0	203.48	1.04	1.04	1.00	2.62	1.62	1480	1.5		81.40%	84.840	96%
											82.67%		97%
	volume ml	volume	M NaOH	s.g.(24°C)	weight	1ste e.p.	2de e.p.	Δ	total mg CO ₂	total g CO2	Weight Sample	C	Recovery respectively to CHN
A5													
	1.0	184.5	1	1.03	1.03	1.57	3.31	1.74	1380	1.4	0.99	38.00%	89.2%
	1.0	184.5	1	1.05	1.05	2.57	4.35	1.78	1407	1.4		38.76%	91.0%
	1.0	184.5	1	1.05	1.05	1.18	2.93	1.75	1389	1.4		38.27%	89.8%
	1.0	202.39	1	1.03	1.03	0.99	2.61	1.63	1477	1.5	0.99	40.69%	95.5%
	1.0	202.39	1	1.05	1.05	1.01	2.63	1.62	1470	1.5		40.50%	95.1%
	1.0	202.39	1	1.04	1.04	0.98	2.58	1.60	1457	1.5		40.13%	94.2%
	1.0	201.2	1	1.04	1.04	0.89	2.72	1.83	1656	1.7	1.09	41.44%	97.3%
	1.0	201.2	1	1.04	1.04	0.56	2.38	1.82	1642	1.6		41.10%	96.5%
												39.86%	93.6%



A10													
	volume ml	volume	s.g.(24°C)	titration weight	1ste e.p.	2de e.p.	Δ	totaal mg CO ₂	totaal g Co ₂	weight sample	C	C% from CHN	Recovery with respect to CHN
	1.0	202.34	1.037	1.037	1071.1	2709.2	1.64	1488	1.5	1.004	40%	42.42	95%
	1.0	202.34	1.042	1.042	1075.5	2708.1	1.63	1483	1.5		40%	42.42	95%
	1.0	202.34	1.041	1.041	1258.1	2909.7	1.65	1500	1.5		41%	42.42	96%
	1.0	190.21	1.045	1.045	1047.4	2880.1	1.83	1565	1.6	1.086	39%	42.42	93%
	1.0	190.21	1.041	1.041	1057.8	2868.4	1.81	1546	1.5		39%	42.42	91%
	1.0	190.21	1.042	1.042	1142.9	2961.0	1.82	1552	1.6		39%	42.42	92%
											39.75%		94%
	volume ml	volume	s.g.(24°C)	titration weight	1ste e.p.	2de e.p.	Δ	totaal mg CO ₂	totaal g Co ₂	weight sample	C	C% from CHN	Recovery respectively to CHN
C2-1													
	1.0	204.9	1.04	1.04	505.6	1725.0	1.22	1121	1.1	0.38	79.56%	84.18	95%
	1.0	204.9	1.036	1.036	353.5	1580.3	1.23	1128	1.1		80.04%	84.18	95%
	1.0	204.9	1.031	1.031	428.4	1635.0	1.21	1110	1.1		78.72%	84.18	94%
											79.44%		94%
C3-1													
	1.0	201.38	1.033	1.033	483.0	1716.5	1.23	1115	1.1	0.38	79.26%	84.01	94%
	1.0	201.38	1.034	1.034	866.8	2127.7	1.26	1140	1.1		81.02%	84.01	96%
	1.0	201.38	1.031	1.031	435.9	1690.7	1.25	1134	1.1		80.63%	84.01	96%
											80.30%		96%
C4-1													
	1.0	205.87	1.035	1.035	988.1	2242.7	1.25	1159	1.2	0.39	81.78%	84.73	97%
	1.0	205.87	1.035	1.035	669.9	1927.8	1.26	1162	1.2		82.00%	84.73	97%
	1.0	205.87	1.041	1.041	279.1	1502.5	1.22	1130	1.1		79.74%	84.73	94%
											81.17%		96%
C5-1													
	1.0	201.44	1.052	1.052	1031.8	2313.2	1.28	1158	1.2	0.38	83.58%	84.35	99%
	1.0	201.44	1.042	1.042	1157.2	2434.0	1.28	1154	1.2		83.28%	84.35	99%
	1.0	201.44	1.04	1.04	1139.1	2417.4	1.28	1156	1.2		83.39%	84.35	99%
											83.42%		99%
C1-1													
	1.0	200.66	1.026	1.026	708.9	1706.1	1.00	898	0.9	0.33	74.45%	83.81	89%
	1.0	200.66	1.026	1.026	823.9	1818.4	0.99	896	0.9		74.24%	83.81	89%
	1.0	200.66	1.029	1.029	795.1	1784.6	0.99	891	0.9		73.87%	83.81	88%
											74.19%		89%

Materials A6, DA1, DA2, DB1, DB2, DC1, DC2

	volume ml	volume	s.g.(24°C)	titration weight	1ste e.p.	2de e.p.	Δ	total mg CO ₂	total g Co ₂	weight bag	Corrected for bag g CO ₂	weight sample	% C	from CHN	Recovery to CHN
Solaveil CT-300 C10097 (A6)														6.5%	62%
Solaveil CT-300 C10	1.0	204.5	1.04	1.04	684.1	2563.1	1.88	1572	1.6	0.27	0.72	1.01	19%	24.80	78%
Solaveil CT-300 C10	1.0	204.5	1.05	1.05	1100.7	3009.8	1.91	1581	1.6	0.27	0.73	1.01	20%	24.80	79%
Solaveil CT-300 C10	1.0	204.5	1.05	1.05	593.4	2492.1	1.90	1572	1.6	0.27	0.72	1.01	19%	24.80	78%
													19.5%		79%
Biobased Drywood A1															
Drywood A1-1	1.0	203.32	1.05	1.05	1481.1	4215.1	2.73	2251	2.3	0.27	1.41	1.11	35%	44.71	78%
Drywood A1-2	1.0	203.32	1.05	1.05	1732.5	4436.5	2.70	2222	2.2	0.27	1.38	1.11	34%	44.71	76%
Drywood A1-3	1.0	203.32	1.05	1.05	1695.1	4402.6	2.71	2227	2.2	0.27	1.39	1.11	34%	44.71	76%
													34.3%		77%
Biobased Drywood A2															
Drywood A2-1	1.0	205.58	1.04	1.04	1963.8	4644.8	2.68	2242	2.2	0.28	1.37	1.07	35%	44.01	80%
Drywood A2-2	1.0	205.58	1.05	1.05	1687.1	4404.4	2.72	2264	2.3	0.28	1.39	1.07	36%	44.01	81%
Drywood A2-3	1.0	205.58	1.06	1.06	1962.3	4738.8	2.78	2291	2.3	0.28	1.42	1.07	36%	44.01	83%
													35.7%		81%
Biobased Drywood B1															
Drywood B1-1	1.0	204.22	1.06	1.06	1486.8	4192.4	2.71	2220	2.2	0.29	1.32	1.10	33%	40.16	81%
Drywood B1-2	1.0	204.22	1.05	1.05	2072.7	4788.8	2.72	2237	2.2	0.29	1.33	1.10	33%	40.16	82%
Drywood B1-3	1.0	204.22	1.05	1.05	1310.5	4028.0	2.72	2241	2.2	0.29	1.34	1.10	33%	40.16	82%
													32.9%		82%
Biobased Drywood B2															
Drywood B2-1	1.0	203.99	1.05	1.05	1905.8	4383.0	2.48	2036	2.0	0.26	1.21	1.03	32%	40.35	80%
Drywood B2-2	1.0	203.99	1.05	1.05	1718.3	4190.1	2.47	2036	2.0	0.26	1.21	1.03	32%	40.35	80%
Drywood B2-3	1.0	203.99	1.05	1.05	1668.0	4145.9	2.48	2049	2.0	0.26	1.22	1.03	32%	40.35	80%
													32.2%		80%
Without P bag															
Biobased Drywood C1															
Drywood C1-1	1.0	202.52	1.06	1.06	1464.0	4327.0	2.86	2334	2.3			1.01	63%	70.54	89%
Drywood C1-2	1.0	202.52	1.05	1.05	1598.1	4539.9	2.94	2410	2.4			1.01	65%	70.54	92%
Drywood C1-3	1.0	202.52	1.05	1.05	1512.8	4446.1	2.93	2394	2.4			1.01	65%	70.54	92%
													64.3%		91%
Biobased Drywood C2															
Drywood C2-1	1.0	203.68	1.05	1.05	1088.3	4141.7	3.05	2525	2.5			1.08	64%	70.63	91%
Drywood C2-2	1.0	203.68	1.05	1.05	1241.6	4284.4	3.04	2500	2.5			1.08	63%	70.63	90%
Drywood C2-3	1.0	203.68	1.06	1.06	1223.9	4288.1	3.06	2491	2.5			1.08	63%	70.63	89%
													63.5%		90%



Material E

Sample: Wheat panel sample 4

	volume ml	volume	s.g.(24°C)	titration weight	1st e.p.	2nd e.p.	Δ	total mg CO ₂	total g Co2	weight sample	% C	from CHN	Recovery to CHN
S4 1-1	1.0	201.57	1.06	1.06	529.7	2322.9	1.79	1448	1.4	1.00	39.31%	42.90	91.63%
S4 1-3	1.0	201.57	1.06	1.06	1349	3151.0	1.80	1458	1.5	1.00	39.57%	42.90	92.25%
S4 1-4	1.0	201.57	1.04	1.04	720.3	2473.5	1.75	1446	1.4	1.00	39.24%	42.90	91.48%
											39.37%	42.90	91.78%
S4 2-1	1.0	203.93	1.06	1.06	235.8	2087.0	1.85	1516	1.5	1.04	39.65%	42.90	92.44%
S4 2-2	1.0	203.93	1.05	1.05	659.5	2520.7	1.86	1532	1.5	1.04	40.10%	42.90	93.46%
S4 2-3	1.0	203.93	1.04	1.04	527.9	2360.9	1.83	1521	1.5	1.04	39.79%	42.90	92.75%
											39.85%	42.90	92.88%
S4 3-1	1.0	202.79	1.03	1.03	911.5	2779.2	1.87	1556	1.6	1.03	41.22%	42.90	96.08%
S4 3-2	1.0	202.79	1.03	1.03	344.3	2192.3	1.85	1542	1.5	1.03	40.86%	42.90	95.25%
S4 3-3	1.0	202.79	1.03	1.03	316.0	2157.5	1.84	1537	1.5	1.03	40.72%	42.90	94.91%
											40.93%	42.90	95.41%
											40.05%	42.90	93.36%

Material F

	volume ml	volume	s.g.(24°C)	tiration weight	1st e.p.	2nd e.p.	Δ	total mg CO ₂	total g Co2	weight sample	% C	from CHN	Recovery
F-1-1	1.0	199	1.04	1.04	2523.8	5494.0	2.97	2653	2.7	1.0627	68.08%	73.40	92.75%
F-1-2	1.0	199	1.04	1.04	1744.0	4714.0	2.97	2653	2.7	1.0627	68.08%		92.75%
F-1-3	1.0	199	1.03	1.03	2224.0	5174.7	2.95	2635	2.6	1.0627	67.63%		92.14%
											67.93%	73.40	92.54%
F- 2-1	1.0	198.48	1.03	1.03	1128.0	2651.9	1.52	1357	1.4	0.53	70.07%	75.30	93.06%
F- 2-2	1.0	198.48	1.04	1.04	1268.5	2782.2	1.51	1348	1.3	0.53	69.61%		92.44%
F- 2-3	1.0	198.48	1.04	1.04	1563.3	3094.3	1.53	1364	1.4	0.53	70.40%		93.49%
											70.03%	75.30	93.00%
F- 3-1	1.0	211.71	1.04	1.04	1150.8	3019.9	1.87	1776	1.8	0.65	74.02%	74.20	99.75%
F- 3-2	1.0	211.71	1.04	1.04	1214.0	3030.8	1.82	1726	1.7	0.65	71.95%		96.96%
F- 3-3	1.0	211.71	1.05	1.05	716.6	2613.4	1.90	1802	1.8	0.65	75.11%		101.23%
											73.69%	74.20	99.31%
F- 4-1	1.0	201.09	1.05	1.05	1105.4	2849.2	1.74	1574	1.6	0.59	72.61%	72.00	100.85%
F- 4-2	1.0	201.09	1.05	1.05	1398.0	3135.4	1.74	1568	1.6	0.59	72.34%		100.48%
F- 4-3	1.0	201.09	1.05	1.05	1271.4	2999.8	1.73	1560	1.6	0.59	71.97%		99.96%
											72.31%	72.00	100.43%
F- 5-1	1.0	208.98	1.04	1.04	1240.6	3001.8	1.76	1652	1.7	0.62	72.16%	77.00	93.72%
F- 5-2	1.0	208.98	1.04	1.04	1257.7	3005.3	1.75	1639	1.6	0.62	71.60%		92.99%
F- 5-3	1.0	208.98	1.04	1.04	1265.6	3029.4	1.76	1654	1.7	0.62	72.27%		93.86%
											72.01%	77.00	93.52%
F- 6-1	1.0	202.66	1.04	1.04	1205.0	2930.1	1.73	1569	1.6	0.61	70.64%	71.70	98.52%
F- 6-2	1.0	202.66	1.04	1.04	1310.8	3019.1	1.71	1554	1.6	0.61	69.95%		97.55%
F- 6-3	1.0	202.66	1.04	1.04	1312.8	3047.6	1.73	1578	1.6	0.61	71.03%		99.07%
											70.54%	71.70	98.38%

Average

73.93



KBBPPS

Work Package 3: bio-based carbon content

Deliverable 3.4: verification of the method for total carbon determination (ruggedness test)

Material G

Sample: Textile

	volume ml	volume	s.g.(24°C)	titration weight	1st e.p.	2nd e.p.	Δ	total mg CO ₂	total g Co2	weight sample	% C	from CHN	Recovery to CHN
tex 1-1	1.0	206.42	1.06	1.06	2515.7	4059.0	1.54	1283	1.3	0.93	37.48%	41.76	90%
tex 1-2	1.0	206.42	1.05	1.05	2670.0	4262.8	1.59	1330	1.3	0.93	38.86%	41.76	93%
tex 1-3	1.0	206.42	1.04	1.04	1341.6	2887.5	1.55	1306	1.3	0.93	38.16%	41.76	91%
tex 2-1	1.0	204.64	1.05	1.05	1784.2	3518.7	1.73	1434	1.4	1.00	39.00%	41.76	93%
tex 2-2	1.0	204.64	1.05	1.05	1527.4	3297.7	1.77	1463	1.5	1.00	39.77%	41.76	95%
tex 2-3	1.0	204.64	1.06	1.06	1833.6	3570.0	1.74	1430	1.4	1.00	38.90%	41.76	93%
tex 3-1	1.0	207.06	1.06	1.06	716.4	2572.4	1.86	1543	1.5	1.07	39.48%	41.76	95%
tex 3-2	1.0	207.06	1.05	1.05	909.2	2730.0	1.82	1521	1.5	1.07	38.91%	41.76	93%
tex 3-4	1.0	207.06	1.02	1.02	1615.7	3427.3	1.81	1560	1.6	1.07	39.93%	41.76	96%
											38.94%	41.76	93.25%

Material I

Sample: Hemp
Frisbee 17851-2

	volume ml	volume	s.g.(24°C)	Titration weight	1ste e.p.	2de e.p.	Δ	total mg CO ₂	total g Co2	weight sample	% C	from CHN	Recovery towards CHN
17851-2 1.1	1.0	204.87	1.05	1.05	1637.9	4221.5	2.58	2135	2.1	1.04	56%	64.84	86.4%
17851-2 1.3	1.0	204.87	1.04	1.04	1816.9	4384.2	2.57	2154	2.2	1.04	57%	64.84	87.2%
17851-2 1.4	1.0	204.87	1.05	1.05	1794.2	4393.5	2.60	2154	2.2	1.04	57%	64.84	87.1%
17851-2 2.1	1.0	205.23	1.05	1.05	1888.9	4364.2	2.48	2063	2.1	0.99	57%	64.84	87.9%
17851-2 2.3	1.0	205.23	1.05	1.05	1733.4	4206.1	2.47	2057	2.1	0.99	57%	64.84	87.7%
17851-2 2.5	1.0	205.23	1.05	1.05	1209.6	3691.4	2.48	2060	2.1	0.99	57%	64.84	87.8%
											56.6%	64.84	87.4%
											St. dev	0.4%	0.6%



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