# Verification Report
# ETV01/2015

## Aerobic Biodegradation of Third generation Mater Bi under marine condition
## By NOVAMONT Spa


**Author: Piero Franz**
**Reviewed by: Sabrina Melandri**
**Approved by: Claudia Gistri**



**Version: Rev 4_20150804**

# Table of Contents

ANNEX 3. a & b. PIANO DI PROVA (Eulittoral & Sublittoral)
ANNEX 4. COVER OF THE RING TEST FINAL REPORT ( ROUND ROBIN )
ANNEX 5. a & b LABORATORY TEST REPORTS (Ris551 ed3 and Ris552 ed3)
ANNEX 6. a & b . REPORTS OF ISTITUTO ITALIANO PLASTICI
ANNEX 7. CHECK LIST ISO 17025
ANNEX 8. AUDIT REPORT
ANNEX 9. PRIMING EFFECT LITERATURE - PAPERS
ANNEX 10. QUALIFICATION OF NOVAMONT BIODEGRADATION LABORATORY

# 1. INTRODUCTION

Environmental technology verification (ETV) is an independent assessment of the performance of a technology or a product for a specified application, under defined conditions and quality assurance

## 1.1 Name of technology

Bio-based plastics (Mater Bi of Third generation) that are biodegradable under marine conditions.

## 1.2 Name and contact of Proposer

Novamont S.p.A., via Fauser 8, I-28100, Novara Italia
Contact person: Francesco Degli Innocenti
Telephone: +390321699607
Email: fdi@novamont.com

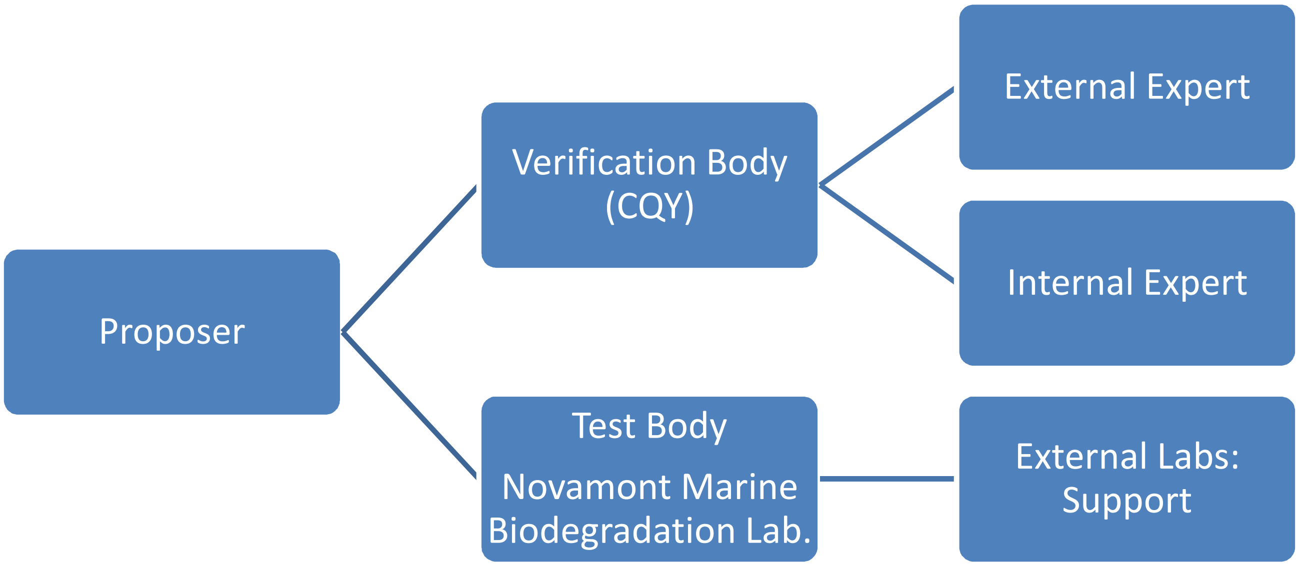
## 1.3 Name of verification body and responsible for verification

The verification is performed by Certiquality S.r.L. Via G. Giardino 4, 20123 Milano
Phone: + 39028069171
The appointed verification expert is: Piero Franz ;
Phone +39 0280691727; +393385778565
Email: p.franz@certiquality.it; piero.franz@fastwebnet.it

## 1.4 Organization of verification, experts, verification process

As mentioned above the verification was managed by Certiquality (VB) according to the flow chart of the organizations involved in the process:

Figure 1: Organizations involved in the EU ETV pilot:



The Certiquality expert was supported by an external qualified person on laboratory quality.
Lab. quality expert: dr. Marina Mari
Certiquality auditor/expert: dr. Piero Franz
Since the technology is on the market the laboratory tests have already been carried out by the Novamont Marine biodegradation laboratory located in Novara, via Fauser 8.
Therefore the verification process followed the steps listed below with the relevant documentations:

Table 1: Main phases of the verification process:

| *Step* | *Responsible* | *Document* |
|---|---|---|
| *Preparation* | *Verification Body* | *Quick Scan*<br>*Contract with Proposer*<br>*Specific Verification Protocol* |
| *Testing* | *Test Body* | *Test Plan*<br>*Test Report* |
| *Verification* | *Verification Body* | *Verification Report*<br>*Statement of Verification* |

Quality Assurance is managed by the internal quality procedures audited by an external Qualification Body (IIP, Istituto Italiano Plastici) according to the ISO 17025 standard and finally reviewed by the Verification Body (Certiquality).
The statement of verification will be issued by Certiquality after completion of the verification process.
A detailed table in section 7 Quality assurance of verification process is provided for more explanation.

### 1.5 Changes vs the specific verification protocol

Since the verification was based on existing tests and data the specific protocol was designed accordingly and either the protocol or the proposed schedule were followed.

## 2. DESCRIPTION OF TECHNOLOGY AND APPLICATION

### 2.1 Summary description of the technology

The manufacturing of this innovative material consists of a combination (generally by means of intimate mixing) of the native corn starch with copolyesters and other natural and renewable raw materials. Possible plastic scraps from manufacturing process are recycled in the extrusion process directly.
The proposed technology tries to combine the mechanical and barrier properties of plastics with the biodegradability of cellulosic materials. This will fit a larger spectrum of requirements coming from the market.
Plastic litter in our oceans is a growing concern. Uncontrolled disposal of waste (e.g. litter) is a serious social problem that must be solved by increasing environmental and civic education, and people’s environmental awareness. This said, it is of interest to know the marine biodegradability of plastics, in case of uncontrolled release.

## 2.2 Scope and borders of application

Alternatives covering all the possible applications where marine biodegradability is a relevant characteristic do not exist.

Plastics that are biodegradable in liquid conditions are: poly(vinyl alcohol) (PVA) and Polyhydroxyalcanoates (PHA). However, PVA is soluble in water and this limits its suitability only to very specific applications. PHAs are bacteria-synthesized polymers that are known to be easily biodegradable in all environment, but whose massive commercial exploitation is still, after 40 years from the original development by ICI (UK), to be fully developed.

All the other plastic materials currently present in the market are not biodegradable or their marine biodegradability is unknown.

Products based on lignocellulose are known to be biodegradable (unless non-biodegradable constituents are added during conversion; e.g. plastic coatings, non-biodegradable additives, etc.). However, mechanical properties of paper, paperboard, etc. are generally lower in comparison with plastics and not suitable to meet the required specifications of some products. For some applications where marine biodegradability can be a relevant characteristic (e.g. shopping bags) cellulosic alternatives do exist and can satisfy the customers' requirements.

This technology makes it possible to manufacture products that, still possessing the main characteristics of plastics (mechanical properties, workability, barrier properties), are biodegradable similarly to the cellulosic products (paper). This is the prerequisite for biological recycling by means of composting. On top of that, this technology offers materials that are biodegradable under marine conditions.

The biodegradability criterion here applied is that a material exposed to an natural environment (such as soil or marine environment etc.) shall show a minimum biodegradation of 90% absolute or relative to cellulose in less than 2 years.

Mater-Bi AF03A0 and Mater-Bi AF05S0 have been both certified as biodegradable and compostable by the Belgian Certifier Vinçotte in accordance to the European standard EN 13432:2000 Packaging. Requirements for packaging recoverable through composting and biodegradation. The standard involves, among other requirements, the demonstration of biodegradability (> 90%, absolute or relative to the reference material, in 6 months).

## 2.3 Environmental benefits

The technology currently uses resources both of fossil origin and of natural origin (bio-based). The fossil constituents are made by the conventional petrochemical industry, while the "bio-based" constituents are extracted by plants (starch) or are obtained starting from vegetable oils by means of innovative chemical processes.

According to the proposer the utilization of vegetable constituents is a positive aspect because it decreases the dependence from non-renewable fossil feedstock. This bio-based feedstock is obtained in a sustainable way. No deforested or natural virgin soils are exploited for the production of renewable raw materials used in the technology. Starch, a substance which for decades had a significant use as an industrial additive in many products e.g. paper, is produced from non-genetically-modified maize cultivated in Europe following the current agronomical practices applied by the European farms.

Vegetable oils are derived from non-genetically-modified crops cultivated in Europe. Neither palm oil nor soybean oil are used in the technology. Non-food dedicated plant-oils are now going to be used with the completion of a bio-refinery located in Europe.

The current land requirements per metric ton of the product are approximately: Maize: 0.03 ha (European average); Vegetable-oil: 0.14; Other natural substances (additives): 0.01 ha Irrigation depends on the geographical location of the farmland. It is estimated that 15-30 liters of irrigation water are needed to grow the renewable raw materials needed to produce one kg of the product. The specific electricity consumption is 0.4 kWh (Environmental Product Declaration ver. 3.3 (SP-00222) Mater-Bi CF05S, November 2012)

No relevant emissions to air or water or specific impacts are caused by the production on the local area.

# 3. EXISTING DATA

## 3.1 Acceptance of existing data

The existing data, as reported in next chapters, were already evaluated and judged reliable by the Istituto Italiano Plastici (Annexes 6a and 6b) ; anyway both the methodology and the test results have been reviewed again by Certiquality as part of the planned auditing activity described in next chapters.

The Certiquality audit report shows a general acceptance of the existing data considering the level of confidence of the test methods which are still in draft.

The test methods followed during the tests performed by the proposer are:

- A test method under development for determining Aerobic Biodegradation of Plastics Buried in Sandy Marine Sediment under Controlled Laboratory Conditions (for Eulittoral condition, see Novamont operating procedure SOP20a)
- ISO/DIS 19679 "Test method for determining aerobic biodegradation of plastic materials sunk at the sea water/sandy sediment interface" (In Draft and reported in Novamont operating procedure SOP20b for Sublittoral conditions ).

As a general concept plastics can end up also in several habitats in the marine environment. In this ETV the two habitats that are most sensitive for the human activities and environment have been considered: the shoreline (e.g. sandy beaches) and the coastal sea bottom. Currents, waves and tides tend to accumulate plastic debris in these areas that are very sensitive for human activities. In addition the density of biodegradable plastics is higher than 1, so they tend to sink. This has been considered as a priority for this ETV.

The two above zones are called: Eulittoral and Sublittoral.

### *3.1.1 Eulittoral zone*

The tidal zone, i.e. the part of the coast affected by the tides and movement of the waves, is the borderline between sea and land, frequently a sandy area that is kept constantly damp by the lapping of the waves (definition from the procedure SOP2a).

The substrate used for the trials was a natural sediment withdrawn at Porto di Marina di Campo (LI) from Hydra laboratory personnel .The plastic films were manufactured at Novamont plant in Novara.

### *3.1.2 Sublittoral zone*

This zone is the interface water/sand where the material that sinks finally reach the sea floor, in a benthic zone where sunlight reaches the ocean floor (photic zone) that, in marine science is called sublittoral zone (definition from the standard draft).

The substrate and the tested material were originated as described above.

# 4. EVALUATION

## 4.1 Evaluation of existing tests

The test methods are designed to determine the % of biodegradation of a plastic material in film form when exposed to a sediment kept wet with salt-water in a reactor, to simulate the littoral zone (the tidal zone and at the interface sea water/sea sediment).

Biodegradation is determined by measuring the carbon dioxide evolved by the plastic material.

The selected materials to be tested are, in both above conditions, listed in the table 2:

Table 2: tested materials (extracted by test results in Italian)

| Denominazione | Lotti interni | Forma fisica e Spessore | % Carbonio |
|---|---|---|---|
| *RIC1511c2* | *2303/4525* | *Film 25 µm* | *56,65* |
| *RIC1532* | *IP1932/1* | *Film 25 µm* | *58,85* |
| *Carta da filtro in cellulosa controllo positivo* | *Whatman® n. 42 Cat No 1442 125* | *Disco 200 µm* | *44,44* |
| *PHB Ecoman (polyhydroxybutyrate) Controllo positivo* | *RIC1536* | *Film 60 µm* | *55,65* |

The tested materials were provided to the Novamont Biodegradation Laboratory with the sampling methodology described in the Internal Quality System Procedure (PG17) which has been verified by the Certiquality auditor. The test materials (denominated RIC1511c2 and RIC1532) were produced and filmed in Novamont plant in Novara. According to the internal procedure PG17, the film samples were obtained (under the control of Lab Manager) by selecting the reel during the central phase of production i.e. a routine operation after the cleaning of the extruder is completed; three meters of film, at least, were discarded and the film was cut with a clean razor blade, and then it was folded and stored in a polyethylene bag.

Sand and sea water were provided by the HYDRA Laboratory, applying the condition set up by PG17, in particular, the sampling took place near the tourist port of Marina di Campo (Elba), a few meters from the shore with a plastic container directly below the water line. With the solid sediment, was taken also the water for keeping it moist. All samples were transferred in a closed container to be transported to the laboratory. The sediment was placed in the Lab refrigerator at about 4°C and was used within 15 days from sampling.

Percentage of Carbon of the samples was analyzed by external labs reported on both “PIANO DI PROVA” as well as their own accreditation/certification references.

The concentration of $CO_2$ is measured on all 10 lab reactors, (2 for RIC1511c2 material, 2 RIC1532 material, 2 for Filter Paper material as a positive control sample, 2 for PHB Ecoman (polyhydroxybutyrate) as a positive control sample and 2 for blank) by titration roughly every week for the first 3 weeks, every two

weeks up to the third month and once a month until the end of the test. See also (Annex 3a&b PIANO DI PROVA and Annex 5a&b LABORATORY TESTS REPORTS).
Round Robin test has been explained in detail in the Verification specific protocol (Annex 2) as well as the criteria of selection of the reference standard and method (draft) for the Novamont Laboratory tests .
AS reported in the Verification Protocol the Round Robin test was carried out mainly for confirming the reliability of the methodology from the reference Standards and method drafts.
All the RRT have been checked by Certiquality and conclusions are accepted.
See also section 3.1 for methods selection and 4.1.4.
All testing approaches applied at worldwide level are based on respirometry, following the biodegradation standard tests developed by OECD in 1981 for chemicals. The laboratory test methods currently developed for marine sediments have been already used for other matrices.
For instance:

- ISO 14855 Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions -- Method by analysis of evolved carbon dioxide -- Part 1: General method
- ISO 17556 Plastics -- Determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved
- ISO 14852 Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium -- Method by analysis of evolved carbon dioxide.

### *4.1.1 Control data*

All data are managed according to the Novamont Biodegradation laboratory Manual. The Manual is the main reference for all the Lab procedures, operating instructions, analytical standards. In particular Section 6.6 of Manual defines the management of data. The manual and relating procedures have been verified by IIP – Istituto Italiano dei Plastici as well as the test equipment, samples traceability, etc. The IIP in his report of September 26$^{th}$ 2014 declares that, for the reference tests, works are done according to the ISO 17025 standard; this is not a real accreditation but a significant support to the Verification Body decision of just planning a two days audit (one on site and one off site) to the test Laboratory of Novamont in order to confirm its own acceptable quality standard. (ref. ISO 17025 check list and final Audit Report from the Certiquality Laboratory expert, Annexes 7 and 8)

### *4.1.2 Analytical laboratory evaluation*

An audit with reference to the ISO 17025 standard has been carried out by a qualified quality auditor from Certiquality (Verification Body ) with a large expertise on laboratory practices.
The audit took 2 days one on site and one off with a double objective:

- Verify and confirm that all the laboratory activities are run according to the good laboratory practices and to the ISO 17025 as reported by the proposer and by the Istituto Italiano Plastici.
- Check how the tests have been conducted, the relevant conditions , the result reliability etc; the preliminary evaluation of technical documentation raised some questions that were analyzed during the audit ( Annex 7 and 8).

The audit report did not raised non conformances but just a few recommendations that were accepted by the proposer and are being implemented. The report clarified the above questions and confirmed the Lab is following the GLP as required.

#### *4.1.3 Test run conditions evaluation*

Two campaigns of tests have been carried out by the Novamont Biodegradation Laboratory in 2014 according to the standards and method mentioned at paragraph 3.5 of attached verification protocol (Annex 2) and on the samples shown on the table 2 at paragraph 4.1.

Some questions raised during the evaluation of the test methodology were investigated.

Temperature and shape/size of material samples are defined in the reference standard draft.

The proposed test method aims at measuring inherent biodegradation of plastic materials when exposed to a marine microbial population, independently by any specific application, shape, thickness. In fact the selected standards reports: “Test material should possibly have the form of a film, or a sheet. Cut samples of test material in the shape of a disk. Disks shall have a radius lower than the glass flasks’ radius so that the disks can be easily laid on the bottom of the glass flask.” The choice of film versus sheet is simply due to the most cost effective criterion; the plastic sheet would have a much longer test time without any real benefit in terms of reliability of results.

The reference standard sets a test temperature between 15 and 28°C max. Again the border temperature value has been chosen in order to shorten as much as possible the test duration.

The Certiquality expert audit and a comprehensive evaluation of the documentation provided by the proposer let’s assess that the tests were complying with the reference methodologies.

#### *4.1.4 Test results quality evaluation*

The whole process quality has been evaluated by IIP and is part of the revision from Certiquality during its audit. In addition the test reliability was investigated with the Round Robin Test whose summarized results are reported in Annex 2.

As far as the Novamont test results are concerned the full set of data is reported in the ANNEXES 5a&b (LABORATORY TEST REPORTS) as well as the test conditions, comments etc.

Regarding the Round Robin Test it has been evaluated by Certiquality and it reports in detail the whole set of data from all 9 attending laboratories. The report cannot be attached because is property of ISO and cannot be displayed without ISO formal approval.

ANNEX 4 is the cover of the mentioned report

#### *4.1.5 Existing data results*

Full reports of the Novamont Biodegradation Laboratory are in ANNEXES 5a&b

The results of these tests are summarized herein below.

*Test of eulittoral zone simulation*

Table 3: full data for all samples in eulittoral simulation (biodegradation %).

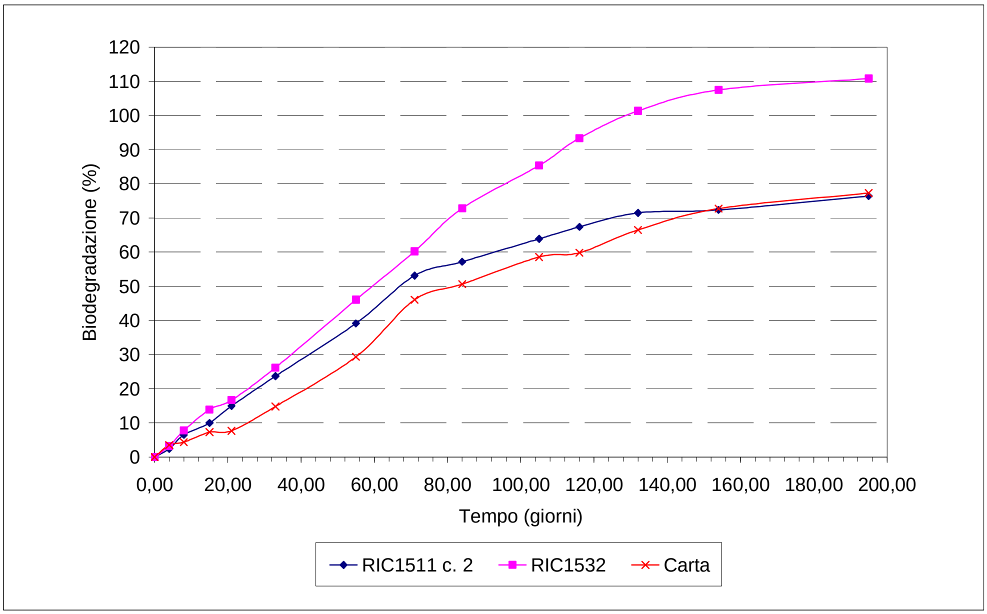
| Time days | R3 %Bio RIC1511 | R4 %Bio RIC1511 | R5 %Bio RIC1532 | R6 %Bio RIC1532 | R7 %Bio PHB | R8 %Bio PHB | R9 %Bio Cellulose | R10 %Bio Cellulose |
|---|---|---|---|---|---|---|---|---|
| 0,00 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 4,00 | 1,31 | 3,53 | 3,06 | 3,37 | 4,15 | 1,93 | 6,41 | 0,63 |
| 8,00 | 5,38 | 7,61 | 8,40 | 7,14 | 10,91 | 10,15 | 5,58 | 3,15 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 15,00 | 8,87 | 11,11 | 13,15 | 14,69 | 18,89 | 18,38 | 8,06 | 6,50 |
| 21,00 | 16,15 | 13,74 | 15,82 | 17,52 | 27,19 | 25,65 | 8,47 | 6,92 |
| 33,00 | 26,04 | 21,32 | 23,53 | 28,85 | 36,40 | 32,61 | 14,26 | 15,31 |
| 55,00 | 40,88 | 37,36 | 42,83 | 49,29 | 58,83 | 38,62 | 30,37 | 28,32 |
| 71,00 | 54,26 | 51,94 | 63,60 | 56,84 | 74,19 | 50,00 | 43,60 | 48,46 |
| 84,00 | 57,17 | 57,19 | 74,28 | 71,31 | 94,46 | 56,96 | 47,73 | 53,50 |
| 105,00 | 60,67 | 67,10 | 85,56 | 85,15 | 117,20 | 68,98 | 54,34 | 62,73 |
| 116,00 | 63,87 | 70,89 | 91,79 | 94,91 | 132,86 | 83,84 | 53,93 | 65,66 |
| 132,00 | 67,94 | 74,98 | 98,92 | 103,71 | 150,68 | 93,96 | 59,72 | 73,22 |
| 154,00 | 68,81 | 75,85 | 103,96 | 110,95 | 163,89 | 108,83 | 66,74 | 78,67 |
| 195,00 | 71,43 | 81,39 | 108,41 | 113,15 | 174,64 | 152,16 | 70,46 | 84,12 |

Table 4: average data for all samples in eulittoral simulation (performance parameters).

| Material | Biodegradation (%) average | Deviation (%) |
|---|---|---|
| RIC1511c2 | 76,41 | 6,52 |
| RIC1532 | 110,78 | 2,14 |
| PHB | 163,40 | 6,88 |
| Filter paper positive control | 77,29 | 8,84 |

Figure 2: average data for all samples in eulittoral simulation.




The samples of MaterBi of third generation identified as RIC1511c2 e RIC1532 (identified respectively by alphanumeric code: Mater Bi AF03A0 and Mater-Bi AF05S0), reached a level of biodegradation respectively of 76,4% and of 110,8%. Test was run for 195 days of test in conditions that simulates the “beach” i.e in contact with natural sediment according to the internal standard method Sop 20a[1]; the temperature during the whole test was kept at 28°C in aerobic conditions. (Annex 5a). The reference materials showed a biodegradation of 163.4% (polyhydroxybutyrate) and 77.3% (cellulose filter paper) under the same conditions.

The 110% value depends on an effect that is well known in biodegradation testing, i.e "priming effect ". The addition of a fast biodegradable matter to a natural matrix (it can be compost, soil, or as in this case, marine sediment) can cause the stimulation of the biodegradation of the matrix itself. This in turn causes an extra respiration that adds to the test material value. The draft standard requires a preliminary oxidation phase to decrease the organic matter content of the matrix (that can cause the priming effect) to a minimum. This was done in all tests carried out by Novamont Biodegradation Laboratory. See also conclusion at chapter 6.2 and literature references can be found on Annexes 9 and 10 .

*Test of interface sand/water, sublittoral zone simulation:*

Table 5: full data for all samples in sublittoral simulation (biodegradation %) .

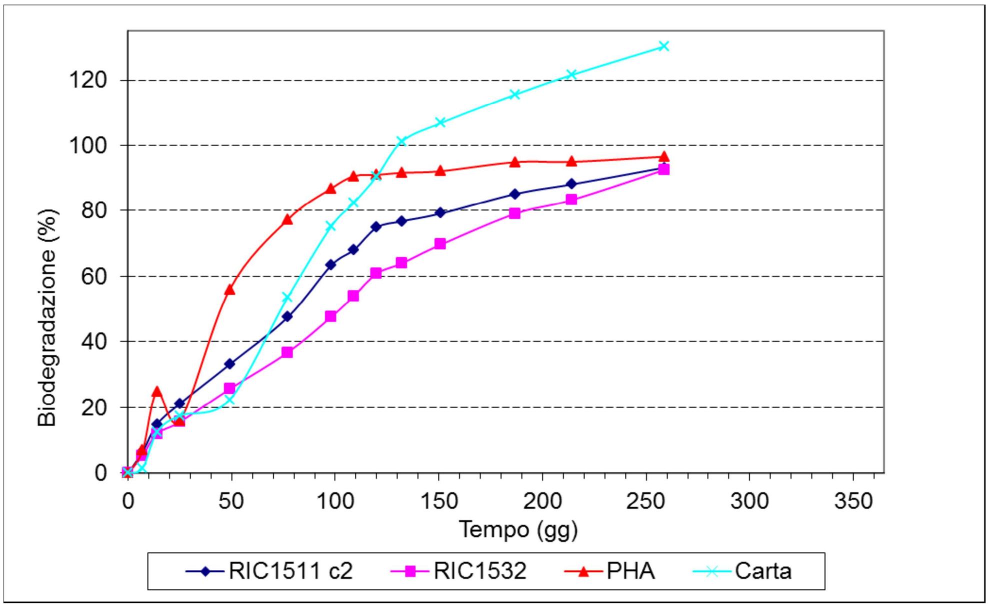
| Time | %Bio | %Bio | %Bio | %Bio | %Bio | %Bio | %Bio | %Bio |
|---|---|---|---|---|---|---|---|---|
| days | RIC1511 c2 | RIC1511 c2 | RIC1532 | RIC1532 | PHB | PHB | Cell | cell |
| 0,00 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 7,00 | 6,25 | 6,78 | 3,88 | 6,93 | 6,71 | 7,10 | 0,63 | 2,43 |
| 14,00 | 13,56 | 16,16 | 10,39 | 13,71 | 23,85 | 25,79 | 9,84 | 15,23 |
| 25,00 | 19,65 | 22,76 | 13,49 | 17,71 | 15,57 | 16,44 | 13,19 | 21,85 |
| 49,00 | 30,17 | 36,14 | 24,20 | 27,11 | 61,12 | 50,88 | 11,31 | 33,54 |
| 77,00 | 43,57 | 51,43 | 33,81 | 39,73 | 75,97 | 78,57 | 43,14 | 64,00 |
| 98,00 | 58,20 | 68,46 | 43,74 | 51,44 | 82,25 | 91,38 | 69,10 | 81,21 |
| 109,00 | 62,01 | 74,20 | 50,10 | 58,06 | 84,11 | 97,09 | 76,85 | 88,05 |
| 120,00 | 69,78 | 79,93 | 55,53 | 66,22 | 83,40 | 98,64 | 86,27 | 94,90 |
| 132,00 | 72,22 | 81,32 | 58,94 | 69,00 | 82,54 | 100,72 | 96,32 | 105,93 |
| 151,00 | 75,26 | 83,06 | 64,83 | 74,54 | 82,54 | 101,76 | 104,28 | 109,46 |
| 187,00 | 81,36 | 88,97 | 75,69 | 82,55 | 85,97 | 103,84 | 115,59 | 115,64 |
| 214,00 | 84,25 | 91,92 | 79,88 | 86,71 | 85,25 | 104,70 | 125,01 | 118,07 |
| 259,00 | 87,76 | 98,70 | 89,65 | 95,48 | 86,54 | 106,60 | 136,11 | 124,47 |

Table 6: average data for all samples in sublittoral simulation (performance parameters).

| Material | Biodegradation (%) average | Deviation (%) |
|---|---|---|
| RIC1511c2 | 93,23 | 5,87 |
| RIC1532 | 92,57 | 3,15 |
| PHB | 96,57 | 10,39 |
| Filter paper positive control | 130,29 | 4,47 |

[1] SOP20a is the Novamont's Standard Operating Procedure to be followed to put in practice the test method under development for determining Aerobic Biodegradation of Plastics Buried in Sandy Marine Sediment under Controlled Laboratory Conditions.

Figure 3: average data for all samples in sublittoral simulation.




The samples of MaterBi of third generation identified as RIC1511c2 e RIC1532 (same materials as above) , reached a level of biodegradation respectively of 93.2% and of 92.6% in 259 days of test at the interface water/sediment according to the internal standard method Sop 20b[2]; the temperature during the whole test was kept at 28°C in aerobic conditions. The reference materials showed an aerobic average biodegradation of 96.6% (polyhydroxybutyrate) and 130.3% (cellulose filter paper) under the same conditions. (see comments on Priming effect reported on the laboratory tests and at chapter 6.2) (Annex 5b).

# 5. VERIFICATION RESULTS

## 5.1 Performance parameters and operational parameters

The two materials are identified by an alphanumeric code: Mater Bi AF03A0 (called RIC1511c2 in test results) and Mater-Bi AF05S0 (called RIC1532 in test results).

All the performance and operational parameters verified are listed in the table 7.

Additional parameters have been added in the table 7 relative to absolute aerobic biodegradation of control materials under the same conditions: cellulose and PHB.

Table 7: Parameter definition table

| *PARAMETER* | *RESULTS* | *TIMING* | *TEST OR MEASUREMENT METHOD(S)* |
|---|---|---|---|
| ***Performance parameters:*** | | | |
| *Aerobic Biodegradation* | *76.4%* | *195 days* | *SOP 20a* |

[2] SOP20b is the Novamont's Standard Operating Procedure to be followed to put in practice ISO DIS 19679

| | | | |
|---|---|---|---|
| *in eulittoral conditions for **Mater-Bi AF03A0** in absolute term within two years (%);* | | | |
| *Aerobic Biodegradation in eulittoral conditions for **Mater-Bi AF05S0** in absolute term within two years (%);* | *110.8%* | *195 days* | *SOP 20a* |
| *Aerobic Biodegradation in sublittoral conditions for **Mater-Bi AF03A0** in absolute terms within two years (%);* | *93.2%* | *259 days* | *SOP 20b* |
| *Aerobic Biodegradation in sublittoral conditions for **Mater-Bi AF05S0** in absolute terms within two years (%);* | *92.6%* | *259 days* | *SOP 20b* |
| ***Operational parameters:*** | | | |
| *Temperature (°C)* | *28 °C* | *For 195 days and 259 days* | *SOP 20a*<br>*SOP 20b* |
| *CO2 evolution both in grams and percentage of the theoretical generation (average of samples) at defined intervals for material to be tested.* | ***Mater-Bi AF03A0** CO2 in grams for eulittoral conditions 623.304 mg CO2. In absolute %: 76.4%.*<br><br>***Mater-Bi AF03A0** CO2 in grams for sublittoral conditions 90.948 mg CO2. In absolute %: 93.2%.*<br><br>***Mater-Bi AF05S0** CO2 in grams for eulittoral conditions 689.436 mg CO2. In absolute %: 110.8%.*<br><br>***Mater-Bi AF05S0** CO2 in grams for sublittoral conditions 92.73 mg CO2. In absolute %: 92.6%.* | *For 195 days and 259 days* | *See above* |
| *CO2 evolution both in grams and percentage of the theoretical generation (average of samples) at defined intervals for reference* | ***Cellulose:** CO2 in grams for eulittoral conditions 572.616 mg CO2. In absolute %: 77.3%.* | *For 195 days and 259 days.* | *See above* |

| | | | |
|---|---|---|---|
| *biodegradable material* | ***Cellulose:** CO2 in grams for sublittoral conditions 93.258 mg CO2. In absolute %: 130.3%.* | | |
| *CO2 evolution both in grams of the theoretical generation (average of samples) at defined intervals for reference blank.* | ***Blank**: CO2 in grams for eulittoral conditions 450.186 mg CO2.*<br><br>***Blank** CO2 in grams for sublittoral conditions 53.196 mg CO2.* | *For 195 days and 259 days* | *See above* |
| *Minimum required duration of test for getting the desired biodegradation percentage* | *195 days for eulittoral condition, 259 for sublittoral condition.* | | *See above* |
| ***Additional parameters:*** | | | |
| *Aerobic Biodegradation in eulittoral conditions for **Cellulose** in absolute terms within two years (%);* | *77,3%* | *195 days* | *SOP 20a* |
| *Aerobic Biodegradation in eulittoral conditions for **PHB** (polyhydroxybutyrate) in absolute terms within two years (%);* | *163,4%* | *195 days* | *SOP 20a* |
| *Aerobic Biodegradation in sublittoral conditions for **Cellulose** in absolute terms within two years (%);* | *130,3%* | *259 days* | *SOP 20b* |
| *Aerobic Biodegradation in sublittoral conditions for **PHB** (polyhydroxybutyrate) in absolute terms within two years (%);* | *96,6%* | *259 days* | *SOP 20b* |

## 5.2 Performance claim

**Two Grades** of bio-based polymers (**Mater Bi of Third Generation, Mater Bi AF03A0** (called RIC1511c2 in test results) and **Mater-Bi AF05S0** (called RIC1532 in test results), have been evaluated.
The performances of the two grades, according to the test results, are :

***The Mater Bi AF03A0 has a 93,2% of biodegradation in absolute terms after 259 days under marine sublittoral conditions.***

***The Mater Bi AF05S0 has a 92,6% of biodegradation in absolute terms after 259 days under marine sublittoral conditions.***

***The Mater Bi AF03A0 has a 76,4% of biodegradation in absolute terms after 195 days under marine eulittoral conditions.***

***The Mater Bi AF05S0 has a 110,8% of biodegradation in absolute terms after 195 days under marine eulittoral conditions.***

- Note 1 The tests were performed in two marine conditions: sublittoral and eulittoral zone (fully explained in par. 3.1). One of two tests, because of the results affected by "priming effects", has been further investigated during the audit carried out at the test laboratory of Novamont .

# 6. CONCLUSIONS AND CONSIDERATIONS

## 6.1 Conclusions

According to the experts evaluation (Ref. Certiquality audit report, Annex 8) the tests under eulittoral conditions, which were temporarily excluded in the verification protocol awaiting for a deeper investigation, have been accepted and included in the reviewed claim. In conclusion no additional test is required.

According to the performance parameters verified the final performance claim is:

**The materials Mater-Bi AF03A0 and Mater-Bi AF05S0, belonging to the family named "Mater-Bi of third generation", have been tested with a test method based on ISO DIS 19679. They showed an aerobic average biodegradation of 93.2% and 92.6% respectively, after 259 days of testing. The reference materials showed an aerobic average biodegradation of 96.6% (polyhydroxybutyrate) and 130.3% (cellulose filter paper) under the same conditions. The testing temperature was 28°C.**

**The materials Mater-Bi AF03A0 and Mater-Bi AF05S0, belonging to the family named "Mater-Bi of third generation", have been tested with a test method under development based on the exposure of plastic material to a wet marine sediment. They showed an aerobic average biodegradation of 76.4% and 110.8% respectively, after 195 days of testing. The reference materials showed a biodegradation of 163.4% (polyhydroxybutyrate) and 77.3% (cellulose filter paper) under the same conditions. The testing temperature was 28°C.**

## 6.2 Considerations about priming effects

The priming effect is a phenomenon where humified portion of soil or compost starts to degrade at an accelerated rate after substrate addition[3].

Priming effect is particularly evident with highly biodegradable substances. For example, glucose is a well-known and very powerful priming effect initiator.

The addition of a fast biodegradable matter to a natural matrix (it can be compost, soil, or as in this case, marine sediment) can cause the stimulation of the biodegradation of the matrix itself. This in turn causes an extra respiration that adds to the test material value bringing to values higher than 100%.

According to Shen and Bartha[4] "net $CO_2$ evolution that exceeds 100% of the added substrate carbon constitutes clear-cut evidence of a priming effect". Shen and Bartha compared CO2 net evolution with 14CO2 evolution. It turned out that substances with a $CO_2$ evolution with a plateau below 100% (and displaying a regular biodegradation course) were not affected by the priming effect while glucose that had a $CO_2$ evolution well above 100 and still growing, was affected by the priming effect.

To cope with the priming effect the practitioner has to decrease to a minimum the biodegradable organic matter present in the natural matrix, to use positive controls and cross-check data.

The two laboratory tests applied in this ETV consider the same marine environment i.e. the littoral zone. One test is carried out with wet sediment, the other one with sediment and a seawater column. Therefore, both tests make use of sediment and seawater sampled at the littoral zone and by a microbiological viewpoint are equivalent. Therefore, the results of the two tests can be considered together and cross-checked.

The biodegradation test under eulittoral conditions (wet sand) showed that filter paper (surely a biodegradable material, used as a positive control) had a biodegradation behavior identical to the RIC 1511c2 test material, while RIC 1532 was higher. Therefore both test materials can be considered biodegradable in comparison with cellulose. The biodegradation test under sublittoral conditions (sediment and a seawater column) showed biodegradation percentages higher than 90% both for 1511c2 and RIC 1532 test material, in line with the polyhydroxybutirrate (PHB) biodegradation. As known, PHB is a natural polymer considered to be a totally biodegradable material (similarly to cellulose) and used as a positive control. On the other hand, cellulose showed a much higher biodegradation, a clear sign of priming effect. The two test methods are disturbed by the priming effect but, considering together the results, both concur to showing that the test materials are as biodegradable as cellulose and as PHB, the two reference materials. Priming effect is a test noise that can be reduced by reducing the available organic carbon present in the natural matrices. In particular it is currently under discussion at the ISO working group to extend the preliminary oxidation phase up to one month.

The applied test methods are the current state-of-the-art. Very likely, in the next years the methods will be improved, as it happened with similar test method: most notably with the ISO 14855-1 Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions -- Method by analysis of evolved carbon dioxide -- Part 1: General method, originally published in 1999,

[3] Marja Tuomela, Annele Hatakka, Sari Karjomaa, Merja Itävaara 2002 Priming effect as determined by adding 14 C-glucose to modified controlled composting test. Biodegradation, 2002, Volume 13(2):131

[4] J Shen and R Bartha (1996) "Priming effect of substrate addition in soil-based biodegradation tests". Appl. Environ. Microbiol. vol. 62 no. 4 1428-1430

where in order to reduce the priming effect the possibility of using a mineral matrix instead of compost was established in the 2005 revision.

## 7. QUALITY ASSURANCE OF VERIFICATION PROCESS

The whole verification process by Certiquality has been managed according Certiquality Quality Manual and related procedures/instructions.

The Verification Body Certiquality is accredited ISO 17021 which requires it to have a Quality assurance System in line with ISO 9001. The Italian Accreditation Body (ACCREDIA) verifies every year its conformity.

Both the internal and external experts are qualified auditors .

The overall process of verification was managed according Certiquality Quality Manual and related procedures/instructions.

Certiquality is accredited ISO 17021 which requires Certiquality to have a Quality assurance system in line with ISO 9001. The Italian Accreditation Body (ACCREDIA) verifies every year our conformity.

The staff and the experts responsible for quality assurance as well as the different quality assurance tasks are reported in Table 8.

Table 8: Quality assurance steps for verification process

| | **Verification Body: CERTIQUALITY** | | | | **Proposer: NOVAMONT** | | **External Expert** |
|---|---|---|---|---|---|---|---|
| ***TASKS/INITIALS*** | *PF* | *MM* | *SM* | *CG* | *FDI* | *MT* | *IC* |
| *Specific verification protocol* | *Author* | | *Review* | *Approve* | *Review* | | *Review* |
| *Test plan* | *Review* | *Review* | | | *Review + Approve* | *Author* | |
| *Test system at test site* | *Audit* | *Audit* | | | | | |
| *Test report* | *Review* | *Review* | | | *Review + Approve* | *Author* | |
| *Verification Report* | *Author* | | *Review* | *Approve* | *Review* | | *Review* |
| *Statement of verification* | *Author* | | *Review* | *Approve* | *Acceptance* | | *Review* |

The specific verification protocol and the verification report require internal (by verification body) and external review according to the EU ETV General Verification Protocol (European Commission, 2014). The internal review for specific verification protocol, verification report and Statement of verification will be carried out by Sabrina Melandri (SM), as Coordinator of Technology Area “Material, Resources and Waste”. The internal approval for specific verification protocol, verification report and Statement of verification will be carried out by Claudia Gistri (CG), as Project Manager of ETV. The external review will be carried out by Irma Cavallotti (IC).

The verification body reviewed the test plan and the test report. The review was done by Piero Franz (PF) and Marina Mari (MM).

A test system audit was conducted following general audit procedures by qualified auditors (Piero Franz for ETV procedures and Marina Mari for laboratory management system) from Certiquality.

During the verification process the proposer NOVAMONT representatives Maurizio Tosin (MT), as Lab Manager, and Francesco Degli Innocenti (FDI), as Ecology of Products and Environmental Communication Director were in charge of the following tasks:

- Review the specific verification protocol and the verification report

- Review and approve the test plan
- Review and approve the test report
- Accept the Statement of Verification.

# 8. REFERENCES

8.1 European Commission (2014): EU Environmental Technology Verification pilot Programme. General Verification
Protocol. Version 1.1 - July 7th, 2014.
8.2 Regulation 17020 ETV of Certiquality and internal procedures.
8.3 International Standardardization Organisation ISO/DIS 19679 (Draft)
8.4 International Standardardization Organisation ISO/DIS 18830 (Draft)

# 9. ANNEXES

ANNEX 1. QUICK SCAN REV.9
ANNEX 2. SPECIFIC VERIFICATION PROTOCOL rev. 07052015
ANNEX 3. a & b. PIANO DI PROVA (Eulittoral & Sublittoral)
ANNEX 4. COVER OF THE RING TEST FINAL REPORT ( ROUND ROBIN )
ANNEX 5. a & b LABORATORY TEST REPORTS (Ris551 ed3 and Ris552 ed3)
ANNEX 6. a & b . REPORTS OF ISTITUTO ITALIANO PLASTICI
ANNEX 7. CHECK LIST ISO 17025
ANNEX 8. AUDIT REPORT
ANNEX 9. PRIMING EFFECT LITERATURE - PAPERS
ANNEX 10. QUALIFICATION OF NOVAMONT BIODEGRADATION LABORATORY