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TEST REPORT N: **RPT-SSCCP- 210375**

**DEGREE OF DISINTEGRATION AND ECOTOXIC EFFECT OF THE COMPOST RESULTING FROM THE
DISINTEGRATION**

Date of report: 23/03/2021

Your reference: Acceptance of our offer R-SSCCP-200429 dated 10/09/2020

SAMPLE IDENTIFICATION:

S-SSCCP-2001835: Sample of paper tray named BIOPAP(®) LC 426 LOTTO: 192723

S-SSCCP-2001858: Sample of paper tray named BIOPAP(®) LC 346 LOTTO: 192645

Sampling, transport and delivery due to Customer.

Received date: 11/09/2020

Date of test start: 18/09/2020

Date of test end: 22/03/2021

Head of the Sector

Patrizia Sadocco

Head of the Area

Patrizia Sadocco

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1. INTRODUCTION

1.1 Principle of the assessment

The purpose of this test is to evaluate the disintegration of the sample in a composting bin in the presence of a freshly prepared biowaste. The composting process is monitored regularly and conducted until the compost is fully stabilized (3 months). At the end of the process the compost is sieved and the disintegration of the sample is carefully measured. The ecotoxic effect of the compost obtained from this process is then evaluated to ensure the absence of any toxic effect on higher plants.

1.2 Test methods

- ISO 16929:2019 "Plastic – Determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test".
- Determination of Ecotoxic Effect of compost according to EN 13432:2000 Annex E/AC:2005 "Determination of ecotoxic effects to higher plants".

2. TEST SCHEDULE

- | | |
|-------------------------------------|------------|
| - Experimental starting date: | 18/09/2020 |
| - Start of the disintegration test: | 03/12/2020 |
| - End of the disintegration test: | 25/02/2021 |
| - Disintegration test duration: | 12 weeks |
| - Start of ecotoxic effects test: | 02/03/2021 |
| - End of ecotoxic effects test: | 18/03/2020 |
| - Experimental completion date: | 22/03/2021 |

3. DETERMINATION OF THE DEGREE OF DISINTEGRATION UNDER DEFINED COMPOSTING CONDITIONS

The test is conducted according to ISO 16929:2019 "Plastic – Determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test".

3.1 Materials and methods

3.1.1 Test item

For the evaluation of the disintegration:

- Name: "BIOPAP(®) LC 426 LOTTO: 192723"
- Sample description: paper tray 32 cm x 25.5 cm dimensions
- Colour: white
- Grammage : 417.8 g/m² coefficient of variation 0.7% (¹)
- Dry weight: 94.2%
- Volatile solids: 99.4%

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For the evaluation of the ecotoxicity:

- Name: "BIOPAP(®) LC 346 LOTTO: 192645"
- Sample description: paper tray, 25 cm x 19 cm dimensions
- Colour: white
- Grammage: 342.0 g/m² coefficient of variation 0.8% ⁽¹⁾
- Dry weight: 92.8%
- Volatile solids : 99.5%

⁽¹⁾ The grammage of the sample was determined according to UNI EN ISO 536:2020 "Grammage of paper and board"

on the left "BIOPAP(®) LC 426" on the right: "BIOPAP(®) LC 346" as received	"BIOPAP(®) LC 426" cut in specimens for disintegration test	"BIOPAP(®) LC 346" milled for ecotoxicity test
		

Fig. 1. Sample pictures.

3.1.2 Analytical methods

Weight measurements

During the test 4 different balances were used:

- Orma BCA 200S (max. 200g, d=0.1 mg), to measure dry weight and volatile solids;
- Radwag PS 8100 (max. 8100g, d=0.01 g) to weight the milled samples;
- Orma TKW30 (max. 30000g, d=0.5 g) to weight the waste mixtures.

Dry weight - humidity weight evaluation

The dry weight is determined in oven at 105±2°C, 2g of sample are treated overnight, then the sample is cooled in essicator and weighted according to UNI 10780:1998 "Compost- Classificazione, requisiti e modalità di impiego".

Evaluation of volatile solids

The volatile solids are determined on the dry sample at 550°C for at least 4h till complete disappearing of black particles as required to UNI ISO 1762:2015 "Carta, cartone e paste - Determinazione del residuo (cenere) dopo incenerimento a 525 °C" adopting the different temperature requested by EN 13432:2000/AC:2005.

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Evaluation of pH

The compost pH is measured by pH-meter HACH LANGE SensION+ PH3, after calibration. The determination is carried out according to UNI EN ISO 14855-1:2013, paragraph 8, on a compost suspension in deionized water at 1:5 ratio after mixing.

Total nitrogen

The evaluation of total nitrogen content of the compost is carried out by Kjeldahl method according to UNI 10780:1998 "Compost- Classificazione, requisiti e modalità di impiego" by applying modifications indicated by "Distillatore Velp Scientifica UDK 149" instrumentation.

1 g of sample is digested (mineralization: Velp Scientifica DK8 Heating Digester) in the presence of di K_2SO_4 , Se, concentrated H_2SO_4 for 30' at 420°C. After cooling the digested fraction is distilled in the presence of NaOH and boric acid. The trapped nitrogen by boric acid is titrated with standard HCl. The result is expressed as %.

Ammonium nitrogen

The ammonium nitrogen determination is carried out according to the method reported in UNI 10780:1998 "Compost- Classificazione, requisiti e modalità di impiego" by applying the modifications indicated by "Distillatore Velp Scientifica UDK 149" instrumentation.

5 g of sample is suspended in KCl for 1h at 1:10 ratio. After centrifugation the solution is diluted (ten times) and added by indicator and MgO. The solution is then distilled in the presence of NaOH boric acid. The captured nitrogen is titrated with standard HCl. The result is expressed as mg/kg.

Nitric nitrogen

The determination of nitric nitrogen is carried out according to the method reported in UNI 10780:1998 "Compost- Classificazione, requisiti e modalità di impiego" by applying the modifications indicated by "Distillatore Velp Scientifica UDK 149" instrumentation.

5 g of sample is suspended in KCl for 1h at 1:10 ratio. After centrifugation the solution is diluted (ten times) and added by Devarda Lega. The solution is then distilled in the presence of NaOH boric acid. The captured nitrogen by boric acid is titrated with standard HCl. The result is expressed as mg/kg.

3.1.3 Preparation of the biowaste

All the organic material components used to prepare the biowaste are reduced to a particle size <50 mm. The components are mixed and water is added to obtain an homogeneous mixture with a final humidity value of about 50%

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Table 1. Composition of fresh biowaste

MATERIALS	% of the total dry weight
Bulking agent (conifer bark)	15.00
Feed for Rabbits	46.00
Sawdust	12.00
Compost	12.05
Fruit, vegetable and cereals	14.80
Urea	0.15

Table 2. Characteristics of the final biowaste mixture and bulking agent.

Parameter	Biowaste	Bulking Agent
Humidity (%)	56.6	41.0
Volatile solids (%)	86.0	98.1
pH	6.8	6.0
NH ₄ -N (mg/Kg)	1790	2532
NO _x -N (mg/Kg)	4285	1266
N tot	1.8	0.5
C/N ratio	23.9	89.4

3.1.4 Set-up of the composting process

Sample preparation

Two fractions of sample are prepared as follows:

- the sample of paper tray "BIOPAP(®) LC 426" for the disintegration test was cut into pieces 10 cm x 10 cm dimensions.
- the sample of paper tray "BIOPAP(®) LC 346" for the composting process needed to determine ecotoxic effect was shredded to a thin fluff.

Composting bins preparation

To perform the test, plastic bins of about 60 litres capacity are used. The following bins were filled:

- Control bins. 2 bins each containing at least 30 kg of biowaste.
- Sample bins. 2 bins each containing at least 30 kg of biowaste with 11% sample concentration respect wet biowaste, obtained by adding: 10% sample "BIOPAP(®) LC 346" milled + 1% sample "BIOPAP(®) LC 426" cut in specimens 10 cm x 10 cm dimensions, in each bin. The two fractions of the sample are well mixed with the biowaste inside the bin.

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Table 3. Wet weight content of the composting bins.

Composting bin	Biowaste (kg)	Sample
Blank_1	30.0	-
Blank_2	30.0	-
BIOPAP(®) LC 346 and BIOPAP(®) LC 426_1	30.0	3.01 kg of milled sample + 301.2 g of sample cut in pieces
BIOPAP(®) LC 346 and BIOPAP(®) LC 426_2	30.0	3.05 kg of milled sample + 302.5 g of sample cut in pieces

3.1.5 Analysis and process control

The main process parameters monitored during the tests are the following:

- The temperature is detected at regular intervals with probes (every day for the first 8 weeks and at least three times a week for the remaining weeks).
- Periodically the composting material is turned and visually inspected (once a week for the first four weeks and at least every 2 weeks for the remaining period) the bins are added by water to restore humidity losses due to evaporation, the water content is maintained around 50-60% during the test, keeping attention that no free standing water is present at the bottom of the reactor after turning of composting material. During turning operations the smell and appearance of the composting mixture are monitored as well as the appearance of the sample specimens.
- Sampling of composting material is performed to check pH at 4, 8, 12 weeks.
- The reactors are aerated daily for 4 hours by fluxing air to ensure that the oxygen content is > 10%.

3.2 Results

3.2.1 Results of process controls and monitoring during disintegration test

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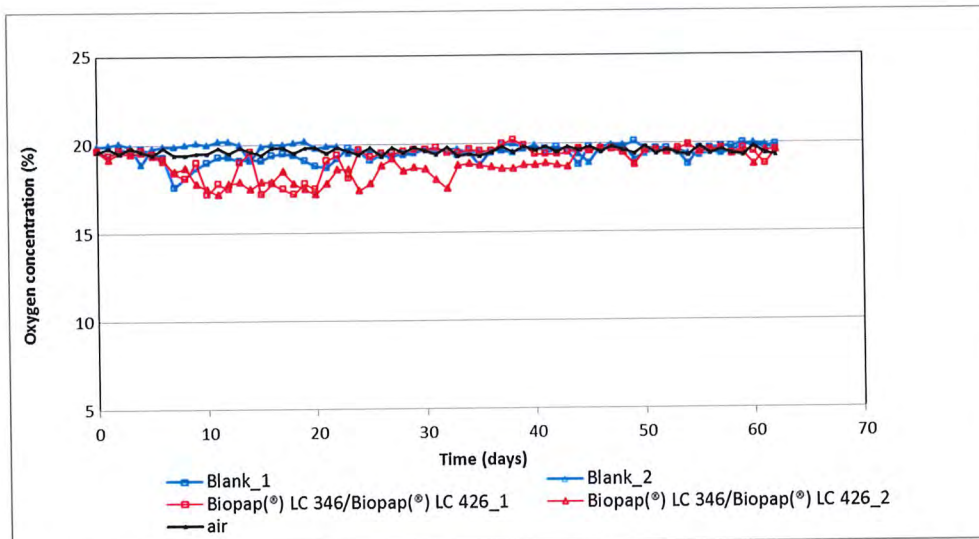


Fig. 2. Evolution of oxygen concentration in composting bins during the disintegration test.

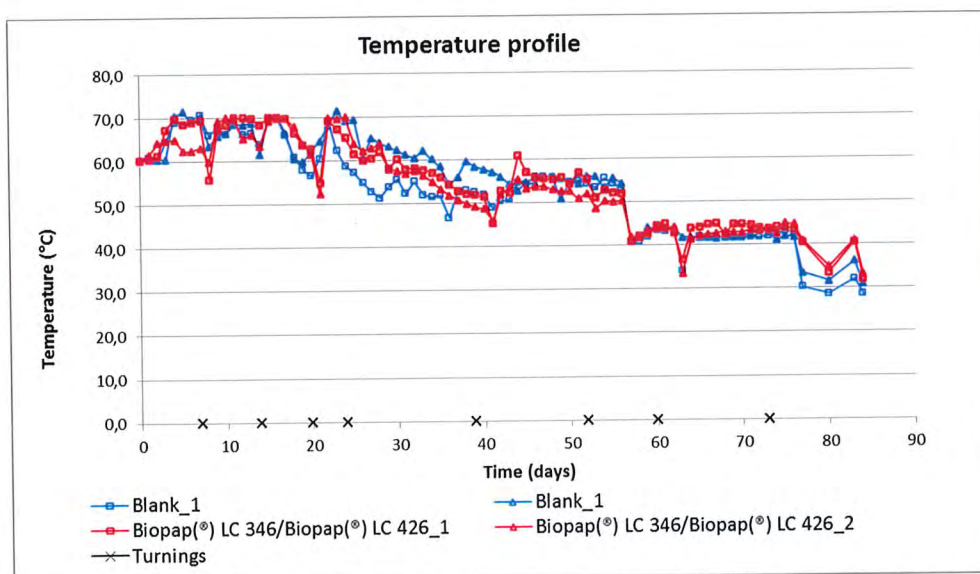


Fig. 3. Temperature profile in the composting bins during the disintegration test.

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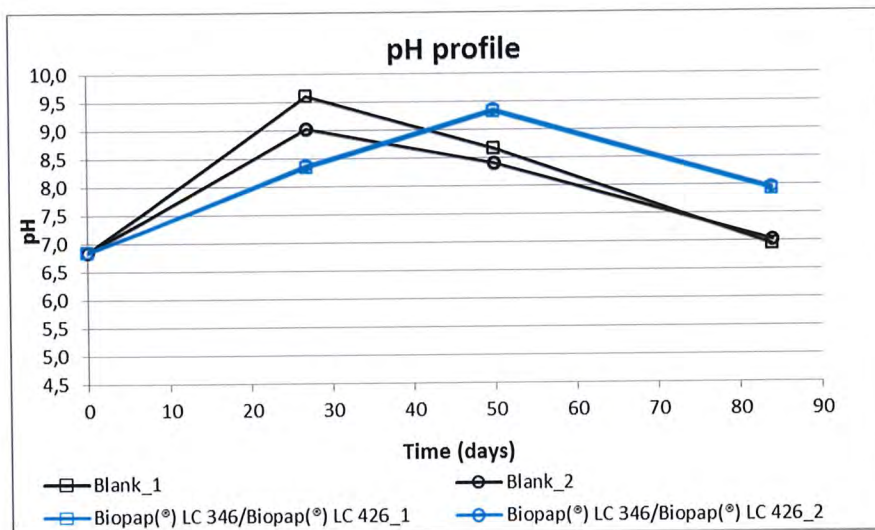


Fig. 4. pH profile in the composting bins during the disintegration test.

Odour and visual observations

In order to monitor the progress of the disintegration during the mixing and humidity recovery, the bins were monitored for the odour developed and visual appearance of the mixture and of the sample under test. The odour and visual inspections evidenced a regular aerobic composting process during the overall 12 testing weeks. No deviations were registered during the test and therefore no corrective operations were adopted. The monitoring operations are summarised as follows.

Odour:

During the different process phases the following odour evolution for Blank bins and Sample bins was observed:

- First 20 days: starting of fermentation processes with rancid odour, slight ammonia for the blank bins and significant ammonia odour for the sample bins,
- From 21 to 40 days: the ammonia odour increased with time and became strong,
- From 41 to 60 days: ammonia smell decreased and was substituted by fresh soil smell,
- From 61 to 84 days: presence of typical odour of mature compost by the end of the test

Visual aspect:

The visual aspect of the organic waste in the bins evolved starting from an initial darkening associated with a volume reduction that was much more visible in the sample reactor than in the Blank.

From the second week the growth of white moulds was noted, which increased up to cover major part of the biowaste. At the same time the smell of ammonia appeared.

The biowaste in the sample reactors showed a significant volume reduction starting from the third week, meantime the sample mixing with the biowaste increased by disintegration and darkening. The milled fraction of the sample was completely disappeared after about 50 days of test.

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The sample cut in specimens was covered by mould growth and became brown within the first month of test. During the second month of test the specimens disintegration increased and in the third month only few residues were visible.

Below pictures of the sample during the composting process are reported (Figs. 5 and 6).

Sample specimens at the beginning of the test and biowaste	Disintegration of the sample specimens after 14 days
	
Disintegration of the sample specimens after 27 days	Disintegration of the sample specimens after 50 days
	
Final compost at the end of the test	
	

Fig. 5. Photo of the sample during the test.

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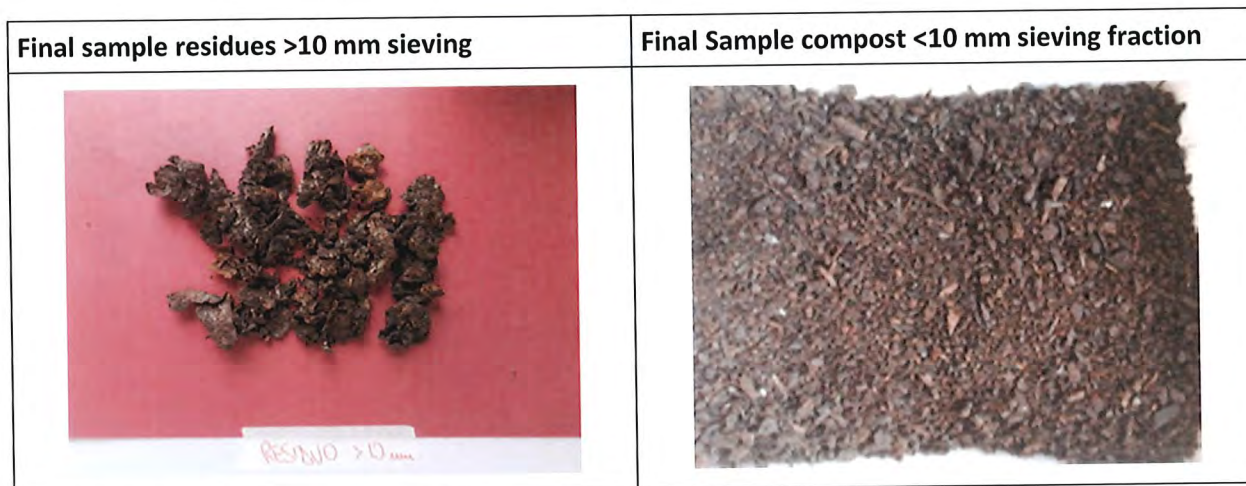


Fig. 6. Photo of the sample residues and sample compost at the end of the test.

Table 4. Variation of volatile solids of composting biowaste.

Composting bins	Volatile Solids (%)			
	Days			
	0	27	50	84
Blank_1	86.0	73.9	68.7	65.2
Blank_2	86.0	71.3	70.0	63.2
BIOPAP(®) LC 346 and BIOPAP(®) LC 426_1	86.0	84.8	70.5	69.4
BIOPAP(®) LC 346 and BIOPAP(®) LC 426_2	86.0	80.7	72.6	67.1

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Table 5. Chemical characterization of the compost obtained at the end of the test.

	Blank_1	Blank_2	BIOPAP(®) LC 346 and BIOPAP(®) LC 426_1	BIOPAP(®) LC 346 and BIOPAP(®) LC 426_2
Humidity (%)	61.4	60.7	61.3	62.1
Volatile solids (%)	65.2	63.2	69.4	67.1
pH	6.9	7.0	7.9	8.0
NH ₄ -N (mg/Kg)	2166	2117	2036	1499
NO _x -N (mg/Kg)	3932	3691	4097	4934
N tot (mg/Kg)	3.3	3.2	3.7	3.6
C/N ratio	10.0	9.8	9.3	9.3

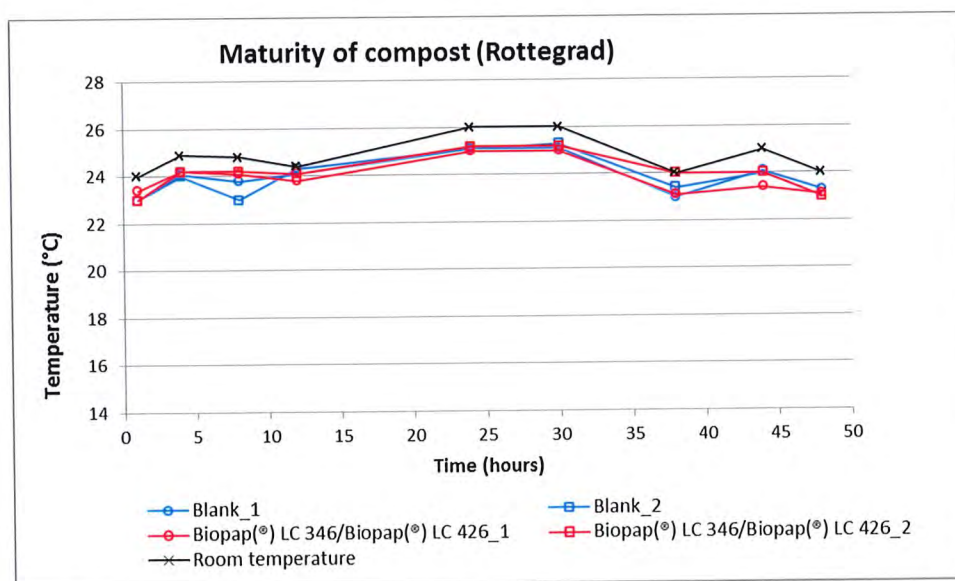


Fig. 7. Temperature profile of the final compost at room temperature.

At the end of the test the compost obtained from the composting process was mature since the maximum temperature measured during the self-heating test remained below 30° C: Rottegrad = V.

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Table 6. Validation of the composting process (ISO 16929:2019 paragraph 8)

	YES	NO
Was the temperature between 60°C and 75°C during days 2-7?	X	
Was the temperature between 55 (±5)°C and 65°C (±5)°C during days 8-28?	X	
Was the temperature between 50 (±5)°C and 60°C (±5)°C during days 29-56?	X	
Was the temperature below 45°C during days 57-84?	X	
The pH increases to a value above 7 during the test and does not fall below 5?	X	
The biowaste compost of the blank control has a maturity (Rottegrad) of IV or V after 12 weeks?	X	
Validation of the composting process:	X	

All the results of the test validation were positive.

3.2.2 Results of the disintegration test

At the end of the 12 weeks of test all the bins were cooled.

The compost was sieved with sieves of 10 mm, 5 mm and 2 mm dimensions for isolating fractions of the residual sample. Some sample residues were recovered from the 10 mm sieving, the sample residues were cleaned from the compost, dried and weighted. All compost fractions except the fraction >10 mm, were then used for the determination of the quality of the compost.

Table 7. Results of the sample "BIOPAP(®) LC 426" disintegration test.

Sample bin	Weight of sample residues (g)				Disintegration (%)	Average disintegration (%)	Disintegration limit (%)
	Initial	At the end					
		2-10 mm	>10 mm	Total			
BIOPAP(®) LC 426_1	301.2	0	13.2	13.2	95.6	95.9	≥90
BIOPAP(®) LC 426_2	302.5	0	11.7	11.7	96.1		

3.3 Summary and conclusions of the disintegration test

Process controls, monitoring and deviations

The odour and visual controls evidenced a regular aerobic composting process during the overall 12 testing weeks. At the end of the test the compost obtained from the composting process was mature since the maximum temperature measured during the self-heating test remained below 30° C: Rottegrad = V as requested by ISO 16929:2019 paragraph 8 point e).

No deviations were registered during the test and therefore no corrective operations were adopted.

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Disintegration test

The sample "BIOPAP(®) LC 426" with grammage equal to $417.8 \text{ g/m}^2 \pm 0.7\%$, disintegrated at 95.9% value therefore, it falls within the limits specified by the EN 13432:2000/AC:2005 with respect to the point A.3.1 for Disintegrability in the composting process.

4. ECOTOXIC EFFECT EVALUATION TO HIGHER PLANTS OF THE COMPOST RESULTING FROM THE DISINTEGRATION TEST

The test was conducted according to EN 13432:2000 Annex E/AC:2005 "Determination of ecotoxic effects to higher plants" on the compost obtained from the disintegration test carried out according to ISO 16929:2013.

4.1 Materials and methods

4.1.1 Materials analysed

The phytotoxic effect (germination of seeds and growth of the plants) was assessed on the following compost:

- Reference compost: compost obtained after exposure to the composting process for 12 weeks, without the presence of the sample.
- Sample compost: compost obtained after exposure to the composting process for 12 weeks, in the presence of the sample "BIOPAP(®) LC 346".

Reference compost and sample compost before the analysis were mixed with a reference substrate. The reference substrate was prepared by mixing vermiculite and peat at a 2 : 1 (w/w) ratio.

4.1.2 Experimental conditions

Set up of the test:

The phytotoxic effect tests were conducted with two different types of seeds: mung bean (*Vigna radiata*) (100 seeds per pot) and barley (*Hordeum vulgare*) (50 seeds per pot).

The sowing was conducted in pots filled with a vermiculite mixture/peat (2: 1 w/w) containing the reference compost or the sample compost, both analysed at 2 different concentrations: 25% and 50% (w/w). The tests were conducted in triplicate. At the end of the test the following parameters were evaluated: germination (number of plants germinated) and biomass (dry weight of the plants). The results obtained with the reference compost and the compost sample were compared at different concentrations. The percentage of germination and the percentage of growth obtained with the compost of the sample were calculated as a percentage compared to the values obtained with the corresponding reference compost.

Set up of pots:

Plastic containers of 500 ml volume were filled with 15g of vermiculite added by 50 ml of deionized water, then about 200g of testing mixture was added and the seeds were distributed on the surface, the seeds were covered by a thin layer of vermiculite/peat mixture (2:1 w/w). The pots were covered (to preserve humidity) and incubated in the dark till the seeds germination, therefore the covering were taken off and

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the pots were exposed to light/dark cycles with photoperiod of 16 hours for 14 days. The plants were watered according to the needs. At the end of the growth period, the plants were cut at the base emerging from the soil and the number of germination and plant dry weight were determined. The percentage of germination and the percentage of growth obtained with the sample compost are calculated respect the values obtained with the corresponding reference compost.

For each seed the following pots were set up:

- 3 pots with vermiculite/peat mixture (2:1 w/w), as growth control;
- 3 pots with reference compost 25% w/w;
- 3 pots with reference compost 50% w/w;
- 3 pots with sample compost 25% w/w;
- 3 pots with sample compost 50% w/w;

For a total of 15 pots for each seed.

Table 8. Seeds used for ecotoxic effect test.

Plant	Brand	Germination index (%)
Mung bean (<i>Vigna radiata</i>)	Marca Bavicchi, linea GEO	98
Barley (<i>Hordeum vulgare</i>)	Marca Bavicchi, linea GEO	92

Environmental conditions:

- Temperature: $25 \pm 5^\circ\text{C}$;
- humidity: $70 \pm 25\%$;
- photoperiod: 16 h of light and 8 hours of dark;
- light intensity: 6500 ± 1000 lux;
- wavelength: solar lamp (visible spectra)

4.1.3 Analytical methods

Germination capacity of the seeds. A layer of cotton and a filter paper disk were piled in a Petri dish and added with 5 ml of deionized water. 20 seeds are distributed on top of the paper disk, and another paper disk previously humidified is put on top of the seeds. The Petri dish is covered with the lid and closed by a parafilm strip. The Petri dishes are incubated 4 days in the dark at room temperature. The germinated seeds are counted and the percentage of germination (germination capacity) is calculated respect the initial seed number.

Weight measurements. During the test 2 different balances were used:

- Orma BCA 200S (max. 200g, d=0.1 mg) to measure dry weight.
- Orma BC 1000 (max. 1000g, d=0.01 g) to weight compost samples and pots.

Dry weight – humidity weight evaluation. The dry weight is determined in oven at $105 \pm 2^\circ\text{C}$, 2g of sample are treated overnight, then the sample is cooled in essicator and weighted according to UNI 10780:1998 "Compost- Classificazione, requisiti e modalità di impiego".

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4.2 Results

Table 9. Ecotoxic effect test validity.

Requirements	YES	NO
Seed germination capacity > 70%	X	
No visible signs of phytotoxic effects (chlorosis, necrosis, leaf deformation; etc.) and morphological variations of the plant species characteristics.	X	
Average number of germinated seeds on the growth control pots > 90%.	X	
Are the requirements respected?	X	

Table 10. Germination and growth on the reference substrate Vermiculite/peat mixture (growth control) (SD= standard deviation)

	BARLEY						MUNG BEAN					
Replicate	Germination			Biomass of plants			Germination			Biomass of plants		
	N° of plants	Average	SD	Dry weight (g)	Average	SD	N° of plants	Average	SD	Dry weight (g)	Average	SD
1	43			0,3795			96			2,5982		
2	48			0,6088			82			2,0556		
3	45	45,3	2,5	0,7475	0,5786	0,1858	95	91,0	7,8	2,1946	2,2828	0,2818

Table 11. Germination and growth on the Reference Compost (SD= standard deviation)

		BARLEY						MUNG BEAN					
% compost	Replicate	Germination			Biomass of plants			Germination			Biomass of plants		
		N° of plants	Average	SD	Dry weight (g)	Average	SD	N° of plants	Average	SD	Dry weight (g)	Average	SD
25%	1	47			0,5422			93			3,4664		
25%	2	45			0,9255			91			2,5444		
25%	3	44	45,3	1,5	0,8884	0,7854	0,2114	90	91,3	1,5	2,4035	2,8048	0,5773
50%	1	47			0,9766			91			2,7199		
50%	2	47			0,9945			93			2,9984		
50%	3	49	47,7	1,2	1,0293	1,0001	0,0268	89	91,0	2,0	2,9926	2,9036	0,1591

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Table 12. Germination and growth on the Sample Compost "BIOPAP(®) LC 346" (SD= standard deviation)

% compost	Replicate	BARLEY						MUNG BEAN					
		Germination			Biomass of plants			Germination			Biomass of plants		
		N° of plants	Average	SD	Dry weight (g)	Average	SD	N° of plants	Average	SD	Dry weight (g)	Average	SD
25%	1	48	47,3	0,6	0,591	0,7748	0,1927	95	91,7	4,9	2,9929	3,0283	0,1408
25%	2	47			0,9753			94			2,9085		
25%	3	47			0,758			86			3,1834		
50%	1	47	47,0	1,0	0,9877	1,1174	0,1126	93	91,3	1,5	2,8788	2,6915	0,1627
50%	2	46			1,1741			90			2,6099		
50%	3	48			1,1903			91			2,5858		

Table 13. Percentage of germination and growth on the Sample Compost with "BIOPAP(®) LC 346" respect the values obtained with the Reference Compost.

% compost	BARLEY		MUNG BEAN	
	% Germination	% Grown	% Germination	% Grown
25%	104,4	98,7	100,4	108,0
50%	98,6	111,7	100,4	92,7

4.3 Summary and conclusions of ecotoxic test

The compost obtained from the disintegration test in the presence of the sample "BIOPAP(®) LC 346" has not determined an inhibiting effect on either the germination or growth of both plants analysed at the different tested compost concentrations.

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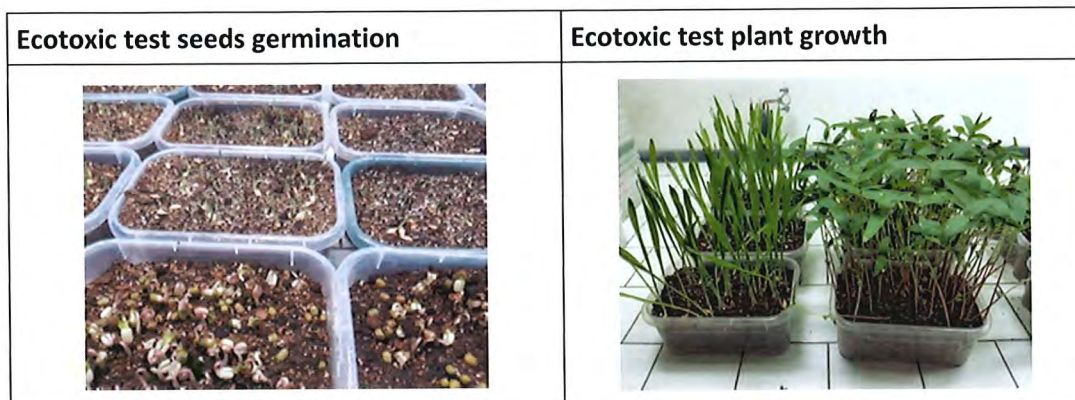


Fig. 8. Photos of ecotoxic test.

5. DETERMINATION OF HEAVY METALS AND OTHER TOXIC AND HAZARDOUS SUBSTANCES

5.1 Materials and methods

5.1.1 Test item

Sample named "BIOPAP(®) LC 346".

5.1.2 Analytical methods

Determination of Arsenic (As), Cadmium (Cd), Chrome (Cr), Mercury (Hg), Nickel (Ni), Lead (Pb), Copper (Cu), Selenium (Se), Zinc (Zn) e Molybdenum (Mo): UNI EN 15411:2011 Combustibili solidi secondari - Metodi per la determinazione del contenuto di microelementi.

Determination of Fluorine (F): UNI EN 15408:2011 Combustibili solidi secondari - Metodi per la determinazione del contenuto di zolfo (S), cloro (Cl), fluoro (F) e bromo (Br).

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5.2 Results

Table 14. Heavy metals and other toxic and hazardous substances and related limits specified in the standard norm EN 13432:2000 Annex A.1.2/AC:2005, table A.1.

Substance Method: UNI EN 15411:2011	"BIOPAP(®) LC 346" (mg/kg)	Limits EN 13432:2000 (mg/kg)
Chrome	3.1±0.6	50
Cobalt	<0.1	38 (a)
Nickel	3.9±0.8	25
Copper	0.2±0.1	50
Zinc	<0.1	150
Arsenic	<0.1	5
Selenium	<0.1	0.75
Molybdenum	<0.1	1
Cadmium	<0.1	0.5
Lead	<0.1	50
Mercury	<0.05	0.5
Fluorine Method: UNI EN 15408:2011	<10	100

The extended uncertainty values refer to a 95% confidence interval. Coverage factor k = 2.

(a) Canadian Certification Programme (CAN/BNQ 9011-911-1/2007).

5.3 Summary and conclusions of the determination of heavy metals and other toxic and hazardous substances

The concentration of heavy metals and other toxic and hazardous substances of sample "BIOPAP(®) LC 346" complies with the limits specified by the EN 13432:2000 Annex A.1.2/AC:2005 reported in table A.1.

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