FINAL REPORT
LC-1/2

Pilot-scale composting + sieving test for measurement of disintegration of **Pouch 120*200 without vent valve (thickness: 112 µm (body), 1.36 mm (double zipper lock), 1.33 mm (single zipper lock))**

Author: Kwok Kuen Chow
Sponsor: BN Packaging Co., Ltd
Room 1002, #2 Building
Twin City Zone 1
Sanshui District
Foshan City
Guangdong Province 528199
CHINA

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1 Identification of the test

1.1 General information

Project number

LC-1/2

Sponsor

BN Packaging Co., Ltd
Room 1002, #2 Building
Twin City Zone 1
Sanshui District
Foshan City
Guangdong Province 528199
CHINA

Sponsor contact

Mrs. Angel Liu
sales@bnpak.com
Phone: +86-18688249735

Testing facility

OWS nv
Dok-Noord 5
9000 Gent
BELGIUM
Phone: +32 9 233 02 04
Fax: +32 9 233 28 25
bruno.dewilde@ows.be
kwokkuen.chow@ows.be

Test item

Pouch 120*200

- Thickness: 112 µm ± 2 µm (body)
- Thickness: 1.36 mm ± 0.01 mm (double zipper lock)
- Thickness: 1.33 mm ± 0.01 mm (single zipper lock)

1.2 Study personnel

Study Director:	Kwok Kuen Chow
Replacement Study Director:	Nike Mortier
Study Director Quality Assurance:	Steven Verstichel

1.3 Study schedule

Study initiation date:	April 23rd, 2019
Experimental starting date:	April 23rd, 2019
Starting date of incubation:	April 25th, 2019
Completion date of incubation:	July 18th, 2019
Experimental completion date:	September 16th, 2019
Study completion date:	January 13th, 2020
Revision date:	September 30th, 2022

1.4 Archiving

All raw data and records necessary to reconstruct the study and demonstrate adherence to the study plan will be maintained in the archives of OWS nv. These records include notebooks, study plan, study report, samples of test item and specimens. They will be stored in a file coded:

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The training records of personnel are stored in the maps 'Organisation and Personnel'. These files are stored per person and administered by the Lab Quality Manager and the Assistant Lab Quality Manager.

After seven (7) years, all data and records will be destroyed or returned to the sponsor after agreement in writing by the involved Sponsor and the Study Director. In case no written agreement of the sponsor can be obtained after seven years, the data and records will be destroyed.

2 Confidentiality statement

The Testing Facility will treat strictly confidential all relevant information on the test item disclosed by the Sponsor as well as all results obtained in executing the Test.

Bruno De Wilde
Managing Director

3 GLP compliance statement

The test was performed in accordance with the OECD principles of Good Laboratory Practices (GLP).

p.p. Nike Mortier

Kwok Kuen Chow
Study Director

4 Quality assurance audit statement

The results reported are in accordance with the study plan and raw data.

A quality control was executed on ..Jan-17-2020

This quality control ensures that the final report is complete and accurately reflects the conduct and raw data of the study.

Steven Verstichel
Study Director QA

5 Summary and conclusions

Zipper bag Pouch 120*200 (thickness: 112 µm (body), 1.36 mm (double zipper lock) and 1.33 mm (single zipper lock)) was evaluated for disintegration in a pilot-scale aerobic composting test according to ISO 16929 (2013). Test item Pouch 120*200 is a zipper bag with vent valve, but the valve is not taken into account for the calculation of the disintegration. Pouch 120*200 (length: 20.3 cm, height: 11.9 cm) was cut into 2 pieces and added in a 1% concentration to biowaste. The control vessel consisted of pure biowaste. The test was performed in duplicate and lasted 12 weeks. At the end of the composting test, the compost was sieved and disintegration was evaluated.

The composting test was done under optimum composting conditions. The operational parameters showed that the test was valid. In both bins the temperature remained above 60°C for at least 1 week and above 40°C during the entire test. Furthermore, the temperature did not exceed the 75°C limit, except once after 5 days of composting with maximum values of 78.3°C and 77.6°C in the control and test bin, respectively. However, immediate action was undertaken and the temperature decreased. A pH of 5.6 was found for the biowaste at start, but after 1.7 weeks the pH had already increased till above 8.6 for all test series and remained above 8.5 during the further test period. The oxygen concentration stayed well above 10%, which guaranteed good aeration conditions.

The disintegration of Pouch 120*200 (thickness: 112 µm (body), 1.36 mm (double zipper lock) and 1.33 mm (single zipper lock)) proceeded very well. Only the vent valves remained largely intact at the end of the test. After 3 weeks of composting, the pieces of the body started to tear and fall apart into smaller pieces. Notably, many of the vents had come loose and could be found separately in the test bin. One week later, the fragmented pieces had reduced in size and presence in the test bin. The vents were still completely intact and assembled. After 6 weeks of composting, fragments of the body of the pouches with an average size of approximately 2 cm × 3 cm were found in the test bin. Two weeks later, the bodies of the pouches and the zippers had disappeared and only the vents remained. Most of the vents remained assembled, while some had split into their components. At the end of the test only the vents (especially the cover and the valve) were retrieved, while no pieces of the body or zipper of the bag were found.

At the end of the composting test (after 12 weeks), the contents of the test bin were used for sieving, sorting, further isolation and analyses. Disintegration is defined as a size reduction to < 2 mm. The contents of the bin were sieved over 10 mm, 5 mm and 2 mm, after which a homogeneous sample of all compost fractions > 2 mm was manually selected and a mass balance was performed. No pieces of the body or zipper of the pouch were found in the different sieving fractions (2 - 5 mm, 5 - 10 mm, > 10 mm). According to the European norm EN 13432 *Requirements for packaging recoverable through composting and biodegradation - Test scheme and evaluation criteria for the final acceptance of packaging* (2000), the American standard ASTM D6868 *Standard Specification for Labeling of End Items that Incorporate Plastics and Polymers as Coatings or Additives with Paper and Other Substrates Designed to be Aerobically Composted in Municipal or Industrial Facilities* (2019) and the international standard ISO 18606 *Packaging and the environment - Organic recycling* (2013) less than 10% of the material may remain present in the > 2 mm fraction after 12 weeks of composting. As can be seen from Table 1, 100% disintegration was obtained for Pouch 120*200 without vent valve (thickness: 112 µm (body), 1.36 mm (double zipper lock), 1.33 mm (single zipper lock)), which is above the 90% pass level.

*Table 1. Disintegration of Pouch 120*200*

Pouch 120*200	Thickness (µm)	Disintegration (%)	Requirement (%)
Body	112	**100**	> 90
Double zipper lock	1,361		
Single zipper lock	1,331		

The quality of the composts to which 1% Pouch 120*200 was added at start of the composting cycle was equally good compared to the control composts. No volatile fatty acids were found in the test and control composts and they all showed a Rottegrad of V, which demonstrates that the composts were stable and mature. An average pH of 8.7 and 9.0 was measured for the control and the test composts, respectively. A similar average salt content was obtained for the test composts (2240 µS/cm) and the control composts (2230 µS/cm). At the end of the test the NH_4^+-N content had decreased till low levels for all series and the NO_x^--N content had increased. After 12 weeks an average NO_x^--N content of 112 mg NO_x^--N/l and 10.2 mg NO_x^--N/l was obtained for the control and test composts, respectively. This indicates that the nitrification process had started and was proceeding well, albeit slightly delayed for the test composts. Average densities of 0.527 kg/l and 0.581 kg/l were found for the control and test composts, respectively. The C/N ratio varied between 9 and 14. A high average volatile solids degradation was observed for all series, indicating that the composting process has proceeded well for all composts.

In conclusion it can be stated that Pouch 120*200 without valve vent (thickness: 112 µm (body), 1.36 mm (double zipper lock), 1.33 mm (single zipper lock)) does fulfil the 90% disintegration requirement stipulated by EN 13432 (2000), ASTM D6868 (2019) and ISO 18606 (2013). Moreover, no negative effect on the composting process and on the (physico-chemical) quality of the produced compost was observed when adding Pouch 120*200 in a 1% concentration at start of the composting.

6 Introduction

6.1 Purpose and principle of test method

The composting bin test simulates as closely as possible a real and complete composting process in pilot-scale composting bins of 200 l. The test item is mixed with the organic fraction of fresh, pre-treated municipal solid waste (biowaste) and introduced in an insulated composting bin after which composting spontaneously starts. Like in full-scale composting, inoculation and temperature increase happen spontaneously. The composting process is directed through aeration and moisture content. The temperature and exhaust gas composition are regularly monitored. The composting process is continued till fully stabilized compost is obtained (3 months).

At the end of the composting process, the compost is sieved by means of a vibrating sieve over 2 mm, 5 mm and 10 mm. Disintegration is evaluated very precisely by manual selection. If possible, a mass balance is calculated on the basis of wet and dry weight. The compost obtained at the end of the composting process can be used for further measurements such as chemical and physical analyses and ecotoxicity tests.

The test is considered valid only if:

- The maximum temperature during composting is above 60°C and remains below 75°C;
- The daily temperature remains above 60°C during at least 1 week and above 40°C during at least 4 weeks;
- The pH increases to above 7.0 during the test and does not fall below 5.0;
- After 12 weeks the blank compost has Rottegrad IV - V and a volatile fatty acids content of less than 500 mg/kg.

More details about the test procedure are given in the study plan.

6.2 Standard followed

- ISO 16929 *Plastics – Determination of the Degree of Disintegration of Plastic Materials under Defined Composting Conditions in a Pilot-Scale Test* (2013)

7 Materials and methods

7.1 Test item

Name:	Pouch 120*200
Description:	PLA coated, kraft paper zipper bag with vent valve (Figure 1 and Figure 2)
Colour:	Brown
Dimensions:	Pouch height: 20.3 cm Pouch width: 11.9 cm
Thickness:	Body: 112 µm ± 2 µm Zipper lock: 1.36 mm ± 0.01 mm (double), 1.33 mm ± 0.01 mm (single)
Total solids (TS):	98.0%
Volatile solids (VS):	94.4% on TS
Sample preparation:	Cut into 2 pieces
Storage conditions:	Room temperature in the dark

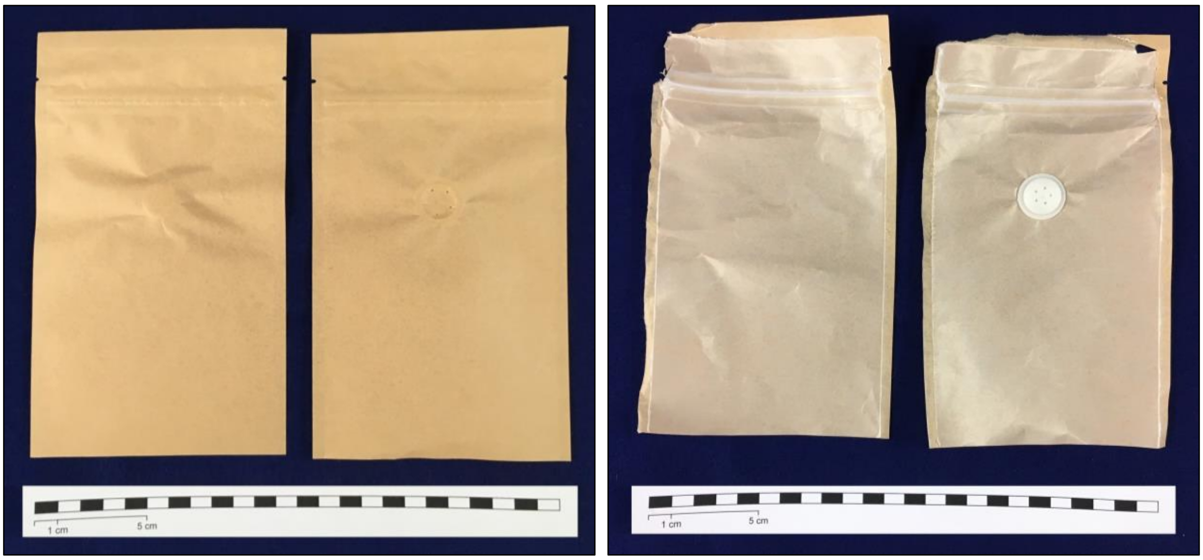

*Figure 1. Visual presentation of test material Pouch 120*200: outside (left) and inside (right)*

7.2 General procedure

The fresh biowaste is derived from the organic fraction of municipal solid waste after a source-separated collection. The test item is mixed with the biowaste, which is used as carrier matrix, and composted in a pilot-scale composting unit (Figure 2). At the end of the composting test the compost is sieved and disintegration is evaluated. More details on the procedure for the particular test reported are given in the study plan.

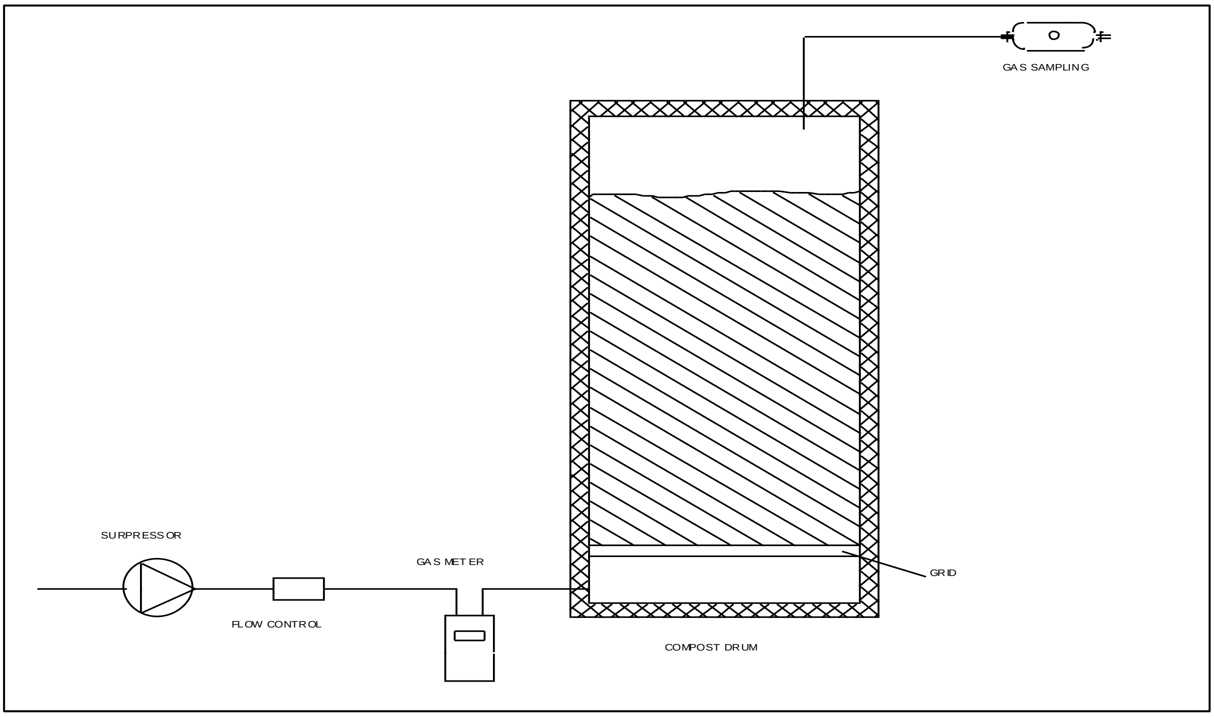

Figure 2. Set-up pilot-scale aerobic composting test

7.3 Analytical methods

Ammonium - nitrogen (NH_4^+-N)

This analysis is performed as described in 'M_054. Determination of ammonium nitrogen by a discrete analyser system and spectrophotometric detection'. The ammonium-N is determined in an aqueous extract (5 parts of demineralised water versus 1 part of sample; see M_057). Ammonia reacts with hypochlorite ions generated by the alkaline hydrolysis of sodium dichloroisocyanurate to form monochloramine. This reacts with salicylate ions in the presence of sodium nitroprusside at around pH 12.6 to form a blue compound. The absorbance of this compound is measured spectrophotometrically at the wavelength 660 nm and is related to the ammonia concentration by means of a calibration curve. The results are given in g per l wet weight.

Dry matter or total solids

The dry matter is determined by drying at 105°C for at least 14 hours and weighing, as described in 'M_009. Determination of moisture content'. The dry matter is given in percent on wet weight.

Gas composition

The gas analyses are performed on a PerkinElmer gas chromatograph with CTRI column as described in 'I_235. Manual TotalChrom software'. The gas chromatograph is calibrated with a standard gas mixture consisting of 10% O_2, 20% CO_2, 30% N_2 and 40% CH_4. Every day gas analyses were executed the gas chromatograph is validated. The results are given in per cent.

Nitrate and nitrite - nitrogen (NO_x^--N)

This analysis is done as described in 'M_055. Determination of total oxidized nitrogen by a discrete analyser system and spectrophotometric detection'. The determination is performed on an aqueous extract (5 parts of demineralised water versus 1 part of sample; see M_057). Nitrate is reduced to nitrite by hydrazine under alkaline conditions. The total nitrite ions are then reacted with sulphanilamide and N-1-napthylethylenediamine dihydrochloride under acidic conditions to form a pink azo-dye. The absorbance is measured at 540 nm and is related to the Total Oxidized Nitrogen (TON) concentration by means of a calibration curve. The results are given in mg per l wet weight.

pH

The pH is measured with a pH meter after calibration with standard buffer solutions (pH = 4.00, pH = 7.00 and pH = 10.00), as described in 'M_006. Determination of pH and electrical conductivity'. Before inserting the electrode, the sample is diluted with distilled water at a ratio of 5 to 1 (5 parts of demineralised water versus 1 part of sample) and thoroughly mixed, as described in 'M_057. Extraction of water and potassium chloride soluble nutrients and elements'.

Rottegrad

The 'Rottegrad' or maturity of the compost is determined by measuring the self-heating capacity of the compost. A precise volume of compost is placed in a 'Dewar' vessel after which the temperature is left to increase spontaneously. The maximum temperature reached is a measure of the stability. More details on the test procedure are given in the 'M_001. Determination of rotting degree – Self-heating test in a Dewar vessel'.

Salt content (electrical conductivity, EC)

The salt content is measured with a conductivity meter after calibration in a 0.01 M KCl and 0.1 M KCl solution, as described in 'M_006. Determination of pH and electrical conductivity'. Before inserting the electrode, the sample is diluted with distilled water at a ratio of 5 to 1 (5 parts of distilled water versus 1 part of sample) and thoroughly mixed, as described in ‘M_057. Extraction of water and potassium chloride soluble nutrients and elements’. The results are given in µS/cm.

Thickness

After an acclimatization period of 24 hours at 23°C and 50% relative humidity, 10 points are measured on the test item. The measurement is executed on a universal bench micrometre (accuracy of 0.1 µm) according to ISO 4593 *Plastics – Film and sheeting – Determination of thickness by mechanical scanning* (1993). An external laboratory executes the analysis.

Total nitrogen (N)

This analysis is done as described in ‘M_039. Determination of total organic carbon and total nitrogen – Method by total carbon, total nitrogen and inorganic carbon combustion’. By combusting the sample at 950°C – 1200°C and adding a controlled extra dose of oxygen for a short time, the nitrogen components will oxidize to nitrogen oxides (NOx). In the presence of a CuO catalyst and a copper reducer the nitrogen oxides are converted to N2. The formed N2 is measured by a Thermal Conductivity Detector (TCD). The results are given in g per kg total solids.

Volatile fatty acids (VFA)

The volatile fatty acids are determined as described in ‘M_035. Determination of volatile fatty acids by gas chromatography and flame ionization detector’. The sample is diluted with distilled water at a ratio of 5 to 1 (5 parts of distilled water versus 1 part of sample) and thoroughly mixed, as described in ‘M_057. Preparation of extracts and solutions’ and centrifuged to remove the suspended solids. Afterwards ether is added and the acids are extracted by centrifugation. The actual analysis is done by gas chromatography. The gas chromatograph is a Clarus 480. The column used is a FFAP of 30 m. The carrier gas is H_2. A mixture with precise concentrations of eight reference volatile fatty acids is used for calibration while 2-methyl-caproic acid is used as an internal standard. The results are given in g per l wet weight.

Volatile solids - ash

The volatile solids and ash contents are determined by heating the dried sample at 550°C for at least 4 hours and weighing, as described in ‘M_010. Determination of organic matter and carbon content’. The results are given in percent on dry matter.

Volumetric density

The volumetric density is determined by filling a 1 l cylinder and measuring the weight after compression with a 650 g plunger for 180 s. This is repeated three times. The exact procedure is described in ‘M_011. Determination of volumetric density’.

Weight determination

During the test 3 types of balances are used. A Sartorius AC 210 S with internal calibration (max. 200 g; d = 0.1 mg) for the determination of dry and volatile matter. A Sartorius CP 12001 S (max. 12100 g, d = 0.1 g), Sartorius CPA 12001 S (max. 12100 g, d = 0.1 g), Sartorius AX6202 (max. 6200 g, d = 0.01 g), Acculab ATL-224 (max. 220 g; d = 0.1 mg) or Sartorius AX224 (max. 220 g; d = 0.1 mg) is used for weighing of the test item. A Robbe Low Profile balance (max. 300 kg; d = 50 g) was used for weighing of the biowaste and the compost bins.

8 Results

8.1 Thickness of test item

The result of the thickness measurements on the body and zipper lock of Pouch 120*200 is given in Table 2. The measured thickness of the test item is taken into account for the disintegration result obtained in this study.

Table 2. Thickness of test item

Pouch 120*200	Measured thickness (µm) (AVG ± SD)	Minimum thickness (µm)	Maximum thickness (µm)
Body*	112 ± 2	109	116
Double zipper lock**	1,361 ± 5	1,361	1,377
Single zipper lock**	1,331 ± 9	1,318	1,345

With AVG = average and SD = standard deviation
**Performed in accordance with ISO 534 (2011)*
***Performed in accordance with ISO 4593 (1993)*

8.2 Test conditions and set-up

Two composting bins with a total volume of 200 l each were started. One control bin (LC-1/2-01), divided into two compartments, which contained only biowaste and one test bin (LC-1/2-02). The test bin was also divided into 2 compartments, which contained each biowaste and 1% Pouch 120*200 (height: 20.3 cm, length: 11.9 cm), cut into 2 pieces. Test item Pouch 120*200 is a zipper bag with vent valve, but the valve is not taken into account for the calculation of the disintegration. Each compartment has a volume of at least 80 l, which is well above the prescribed minimal volume of 35 l. The exact test set-up is given in Table 3. Moreover, the test material was placed in slide frames in order to evaluate the disintegration visually. The biowaste consisted of VGF (Vegetable, Garden and Fruit waste) to which 11% extra structural material was added in order to obtain optimal composting conditions. At start-up, all vessels were filled to the top of the bin.

Table 3. Test set-up

Composition	Control bin LC-1/2-01		Test bin LC-1/2-02	
	C1	C2	C1	C2
VGF (kg)	36.8	36.8	36.8	36.8
Structural material (kg)	4	4	4	4
Pouch 120*200 (kg)	-	-	0.41	0.41
% Pouch 120*200 on biowaste	-	-	1.0	1.0

8.3 Analyses biowaste

The fresh biowaste was derived from the separately collected organic fraction of municipal solid waste, which was obtained from the biowaste composting plant of Erembodegem, Belgium. The characteristics of VGF and structural material are given in Table 4. Table 5 shows the characteristics of the mixtures in the composting bins.

The biowaste at start (= VGF + structural material) should have a moisture content and a volatile solids content on total solids (TS) of more than 50% and a pH above 5. From Tables 4 and 5 it can be seen that these requirements were fulfilled. The biowaste contained a moisture content of 63.6% and a volatile solids content of 61.7% on TS. At start-up a pH of 5.6 was measured. Furthermore, the C/N ratio of the biowaste at start should preferably be between 20 and 30. A somewhat lower C/N ratio of 16 and 17 was obtained for the biowaste and biowaste with 1% test material. The somewhat lower C/N ratio of the biowaste did not really hinder the test. A low C/N ratio results from a high level of nitrogen in the biowaste (e.g. due to many proteins). This can lead to NH_3 emission (and odour) and eventually slow or difficult nitrification towards the end of the composting cycle. It must be noted that mainly a high C/N ratio can be disadvantageous for the composting process, as this is indicative for N deficiency.

Table 4. Characteristics of VGF and structural material

Characteristics	VGF	Structural material
Total solids (TS, %)	33.7	61.2
Moisture content (%)	66.3	38.8
Volatile solids (VS, % on TS)	56.0	90.7
Ash content (% on TS)	44.0	9.3
pH	5.6	-
Electrical conductivity (EC, µS/cm)	3120	-
Volatile fatty acids (VFA, g/l)	3.5	-
NO_x^--N (mg/l)	b.r.	-
NH_4^+-N (mg/l)	813	-
Total N (g/kg TS)	21.3	6.1
C/N	13	74

b.r. = below reporting limit
Reporting limit: NO_X^--N = 10 mg/l

Table 5. Characteristics of the biowaste and biowaste with test item

Characteristics	Biowaste (= VGF + structural material)	Biowaste + 1% Pouch 120*200
Total solids (TS, %)	36.4	37.0
Moisture content (%)	63.6	63.0
Volatile solids (VS, % on TS)	61.7	62.6
Ash content (% on TS)	38.3	37.4
Total N (g/kg TS)	18.8	18.3
C/N	16	17

8.4 Temperature profile and analyses exhaust air

Figure 4 shows the temperature evolution during the composting test. According to ISO 16929 (2013) the test is considered valid if in the composting bins the maximum temperature during composting is above 60°C and remains below 75°C during the first week and below 65°C thereafter in order to ensure that the microbial diversity is not reduced. Furthermore, the daily temperature should remain above 60°C during at least 1 week and above 40°C during at least 4 consecutive weeks.

These requirements were largely fulfilled. After start-up the temperature increased almost immediately till above 60°C for both bins and the temperature did not exceed the 75°C limit, except once after 5 days of composting with maximum values of 78.3°C and 77.6°C in the control and test bin, respectively. However, action was undertaken and the temperature decreased. The temperature remained above 60°C during at least 1 week of composting in both bins, but exceeded the 65°C limit a few times during the second week of composting with maximum values of 67.1°C and 71.0°C in the control and test bin, respectively. Taken into account that the composting process proceeded well during this test, sufficient microbial diversity was guaranteed in spite of the fact that the temperature shortly exceeded the 65°C limit after the first week. After 11 days of composting both bins were placed in an incubation room at 45°C to ensure high temperatures. Moreover, after 2.7 weeks of composting biowaste of the same composting age was added to the contents of the bins (separated by a net) in order to maintain optimal composting conditions. This resulted in a significant temperature increase in the control bin. Elevated temperatures during the composting process were also caused by the turning of the contents of the bins, during which air channels and fungal flakes were broken up and moisture, microbiota and substrate were divided evenly. As such optimal composting conditions were re-established, resulting in a higher activity and a temperature increase. The temperature remained above 40°C during the entire test period.

Figure 5 shows the CO_2 production rate during the composting test (individual measurements at regular points in time), which is representative for biological activity. After start-up a high activity was measured for the control and test bin, after which the CO_2 production gradually decreased. At the end of the test a low activity was found for all test series, indicating that the composting process was completed. The oxygen concentration of the exhaust air is given in Figure 6. The oxygen concentration remained above 10% during the test, which guaranteed good aeration conditions.

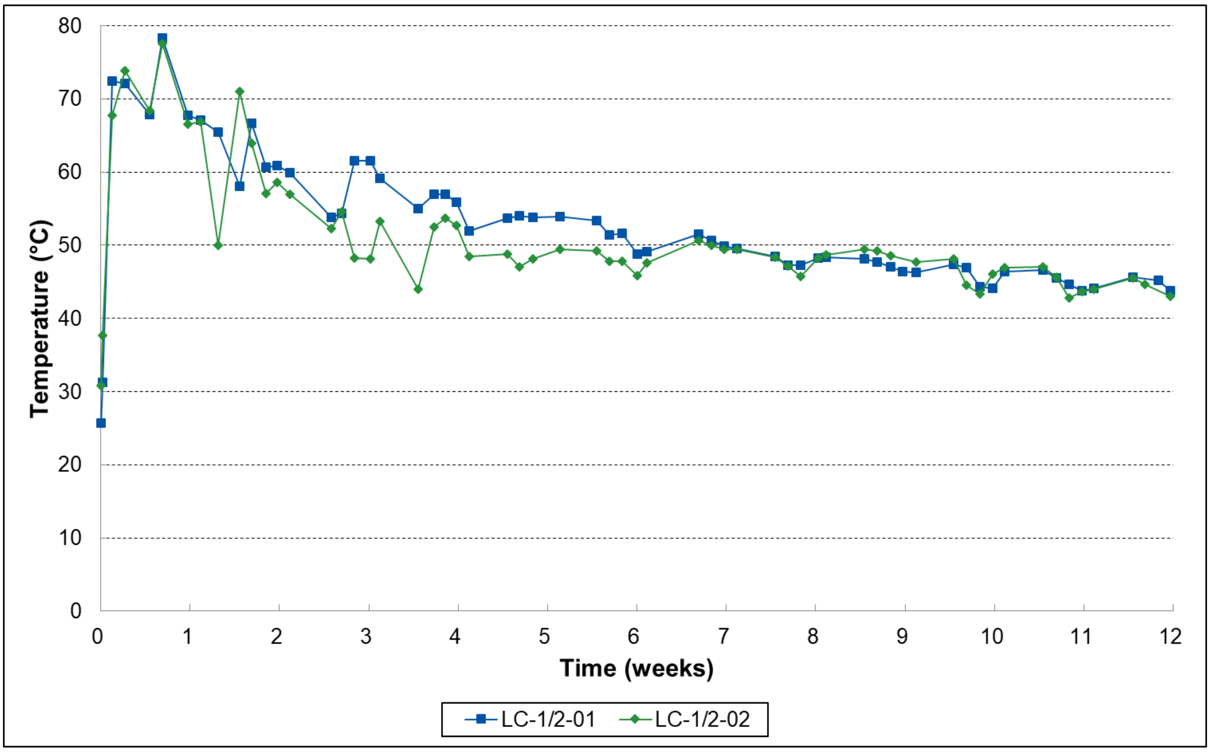

Figure 4. Temperature evolution during the composting test

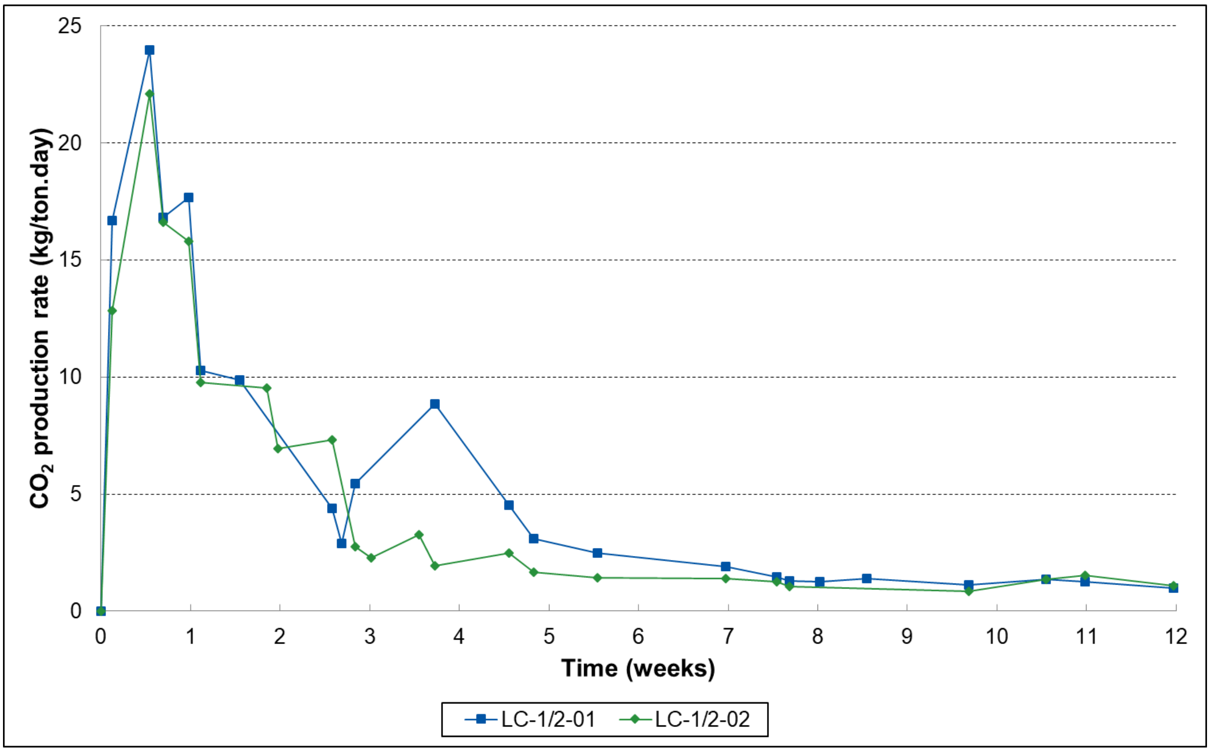

Figure 5. CO_2 production rate during the composting test (individual measurements at regular points in time)

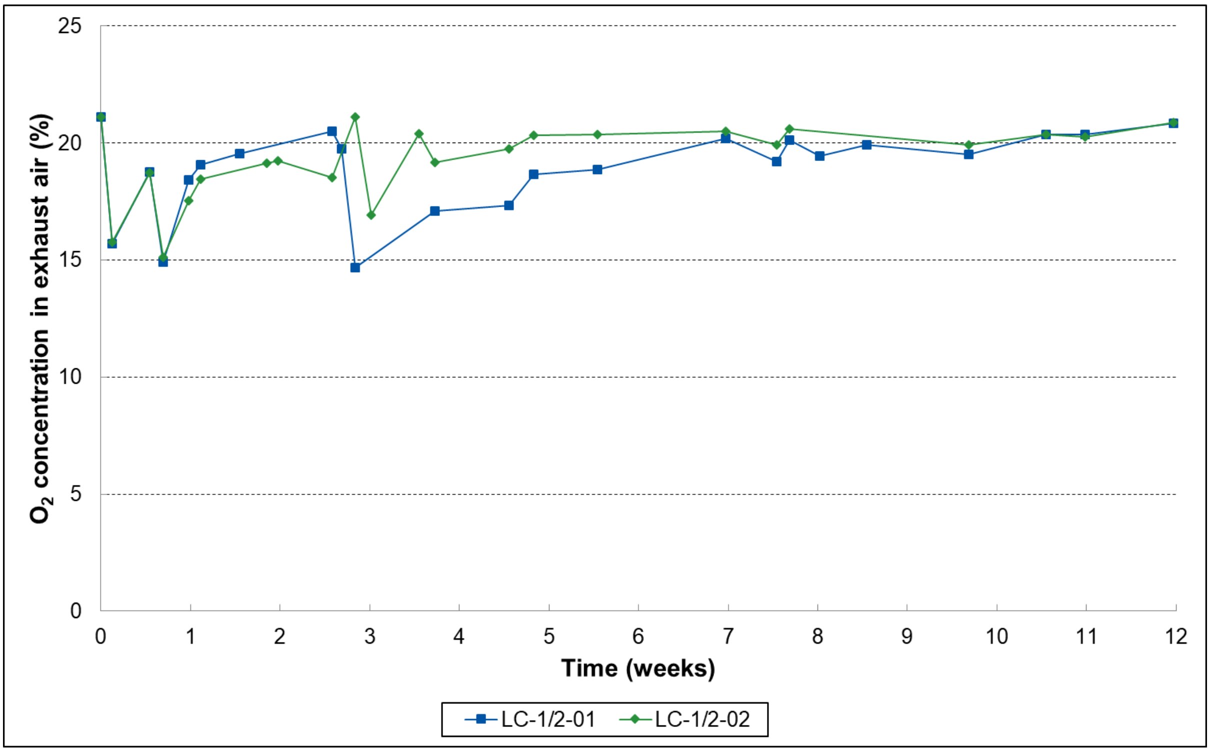

Figure 6. O_2 concentration in the exhaust air during the composting test

8.5 Evolution of pH, NH_4^+-N and NO_x^- -N

Figure 7 shows the evolution of the pH during the composting cycle, while Figure 8 and 9 give the trend in NH_4^+-N, respectively NO_x^- -N for the different bins.

According to the international standard ISO 16929 (2013) the pH should increase till a value above 7 during composting and not fall below 5. The biowaste at start showed a pH of 5.6. After 1.7 weeks of composting the pH had already increased till above 8.6. During the further test period the pH remained above 8.5. At the end of the test (after 12 weeks) an average pH of 8.7 and 9.0 was measured for the control and the test composts with 1% Pouch 120*200, respectively.

The biowaste at start contained an ammonium content of 813 mg NH_4^+-N/l. Immediately after start-up, the ammonium content decreased and after 9.7 weeks of composting low ammonium levels (< 20 mg NH_4^+-N/l) were obtained for the LC-1/2-01 C2 control replicate and both test replicates. These low ammonium levels were maintained till the end of the test, at which point control replicate LC-1/2-01 C1 had reached an equally low value.

After 5.9 weeks of composting an increase in NO_x^- -N concentration was observed for control replicate LC-1/2-01 C2. Two weeks later, a significant increase was also noticed for test replicate LC-1/2-02 C1. After 9.7 weeks of composting, an increase was observed for all replicates. At the end of the test an average NO_x^- -N content of 112 mg NO_x^- -N/l and 10.2 mg NO_x^- -N/l was measured for the control and test composts, respectively.

At the end of the test low NH_4^+-N levels were obtained, while the NO_x^- -N content had increased. This indicates that the nitrification process had started and was proceeding well.

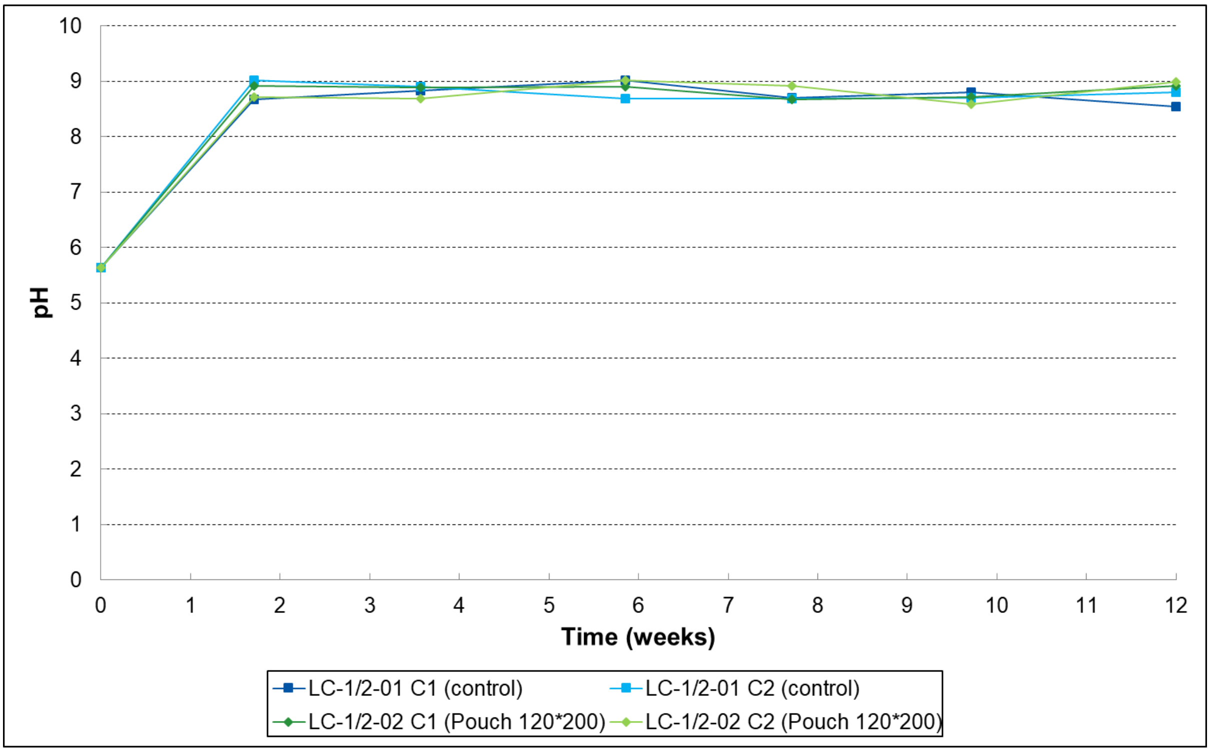

Figure 7. Evolution of pH during composting cycle

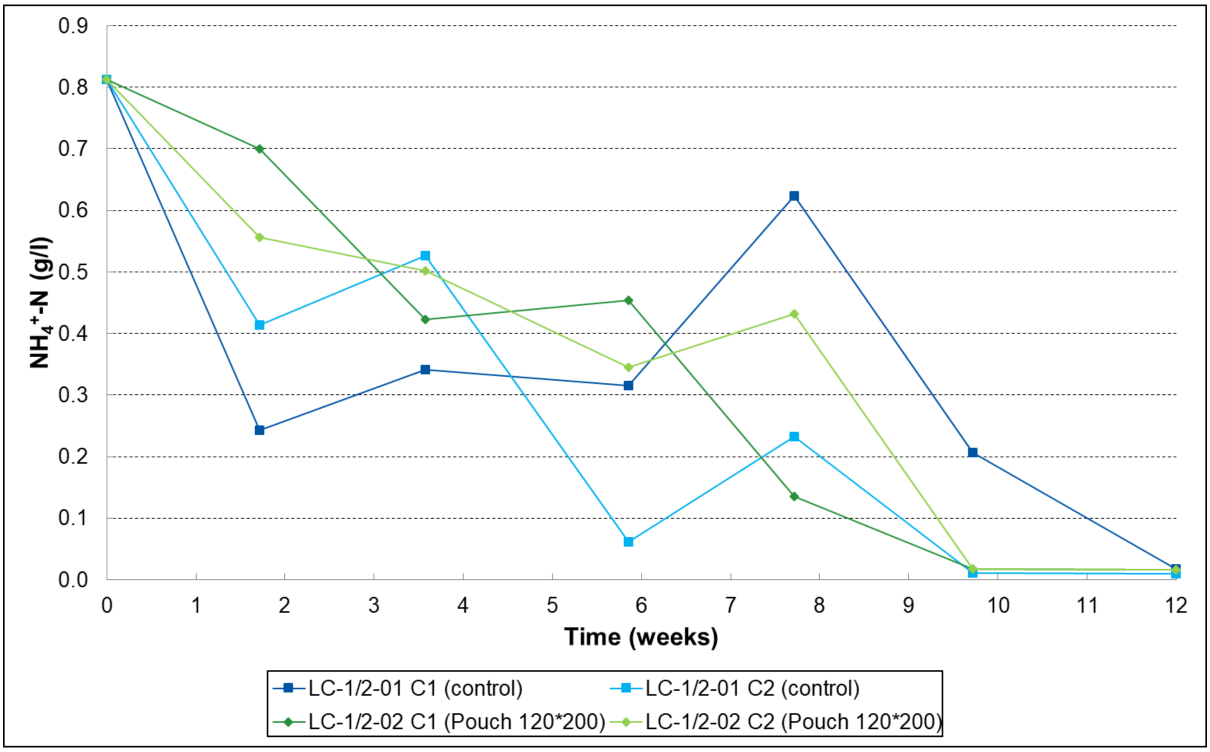

Figure 8. Trend of NH_4^+-N during composting cycle

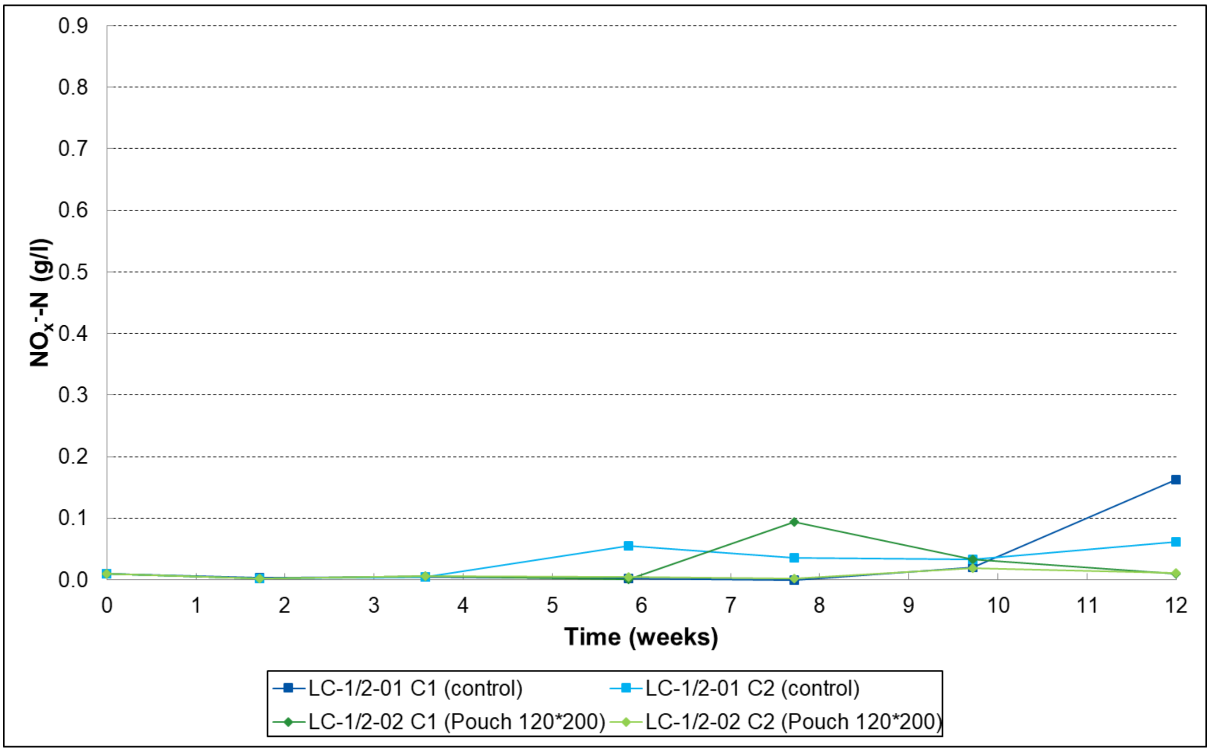

Figure 9. Trend of NO_x^--N during composting cycle

8.6 Visual perceptions

The mixtures in the composting bins were regularly turned by hand, during which the disintegration of the test material was carefully examined. A visual presentation of the disintegration of the body and seal of Pouch 120*200 (thickness: 112 µm (body), 1.36 mm (double zipper lock), 1.33 mm (single zipper lock)) in slide frames is shown in Annex 1 (Figure 19 and Figure 20). The disintegration of both body and seal in slide frames proceeded well. After 1 week of composting, discoloration and fungal growth were noticed on the materials. After 3 weeks the first holes started to appear and the test material started to feel weak. After 4 weeks these holes had mostly increased in size. Moreover, some of the zipper pieces had fallen out of the slide frames. After 6 weeks of composting, the body of the pouch appeared to have disappeared entirely from the slides, which was confirmed during the next turning of the contents of the bin after 8 weeks. The zipper is somewhat more resilient. After 6 weeks pieces of the material remained present but two weeks later only some tiny pieces were left in the borders of the slide frames. After 10 weeks of composting, the slides containing the seal of the pouch were also completely empty.

The disintegration of Pouch 120*200 (thickness: 112 µm (body), 1.36 mm (double zipper lock) and 1.33 mm (single zipper lock)), cut into 2 pieces, has proceeded very well. Only the vent valves remained largely intact at the end of the test. Figure 10 shows a visual presentation of the contents of a test bin with 1% Pouch 120*200 after 1 week of composting. The pouches were abundantly present in the bin and remained intact (Figure 10). One week later, a dark discolouration of the test material was noticed (Figure 11). After 3 weeks of composting, the pieces started to tear and fall apart into smaller pieces. Notably, many of the vents had come loose and could be found separately in the test bin (Figure 12). The disintegration continued and after 4 weeks of composting the fragmented pieces had reduced in size and presence in the test bin (Figure 13). The vents were still completely intact and assembled. After 6 weeks of composting, fragments of the body of the pouches with an average size of approximately 2 cm × 3 cm were found in the test bin. The vents were found separately and remained intact (Figure 14). Two weeks later, the bodies of the pouches appeared to have disappeared and only the assembled vents remained. Some of which were still covered with a piece of the body. Despite being whole, the plastic of the vents was brittle and broke easily while handling (Figure 15). At the end of the test no more pieces of the body of the bag or zipper could be found. Only the vent value remained present in the different sieving fractions (Figure 16).

Figure 17 gives a visual comparison of the < 10 mm compost fraction of control and Pouch 120*200 compost at the end of the test (after 12 weeks of composting). Pieces of the vent (such as the valve) distinguished the < 10 mm fraction of test composts from the control composts, but the pouch itself was completely disappeared.

*Figure 10. Visual presentation of the contents of a test bin with 1% Pouch 120*200 (thickness: 112 µm (body)), cut into 2 pieces, after 1 week of composting*

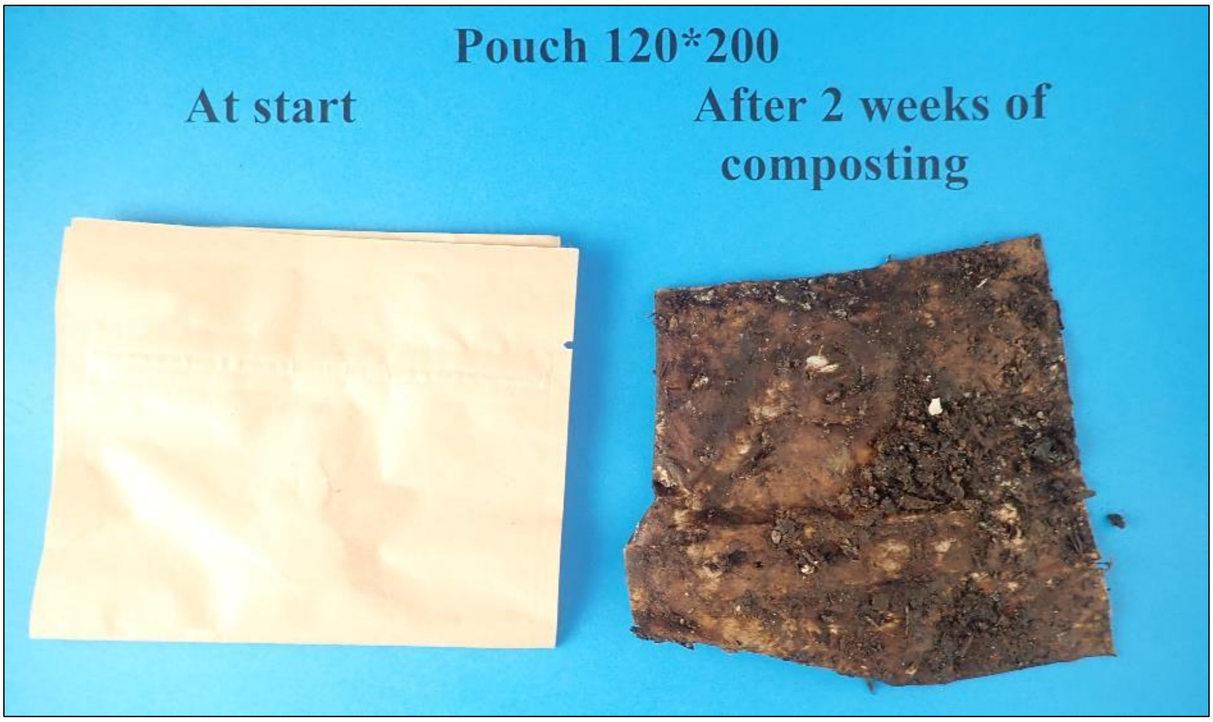

*Figure 11. Visual comparison between Pouch 120*200 (thickness: 112 µm (body)), cut into 2 pieces, at start and after 2 weeks of composting*

*Figure 12. Visual presentation of the contents of a test bin with 1% Pouch 120*200 (thickness: 112 µm (body)), cut into 2 pieces, after 3 weeks of composting*

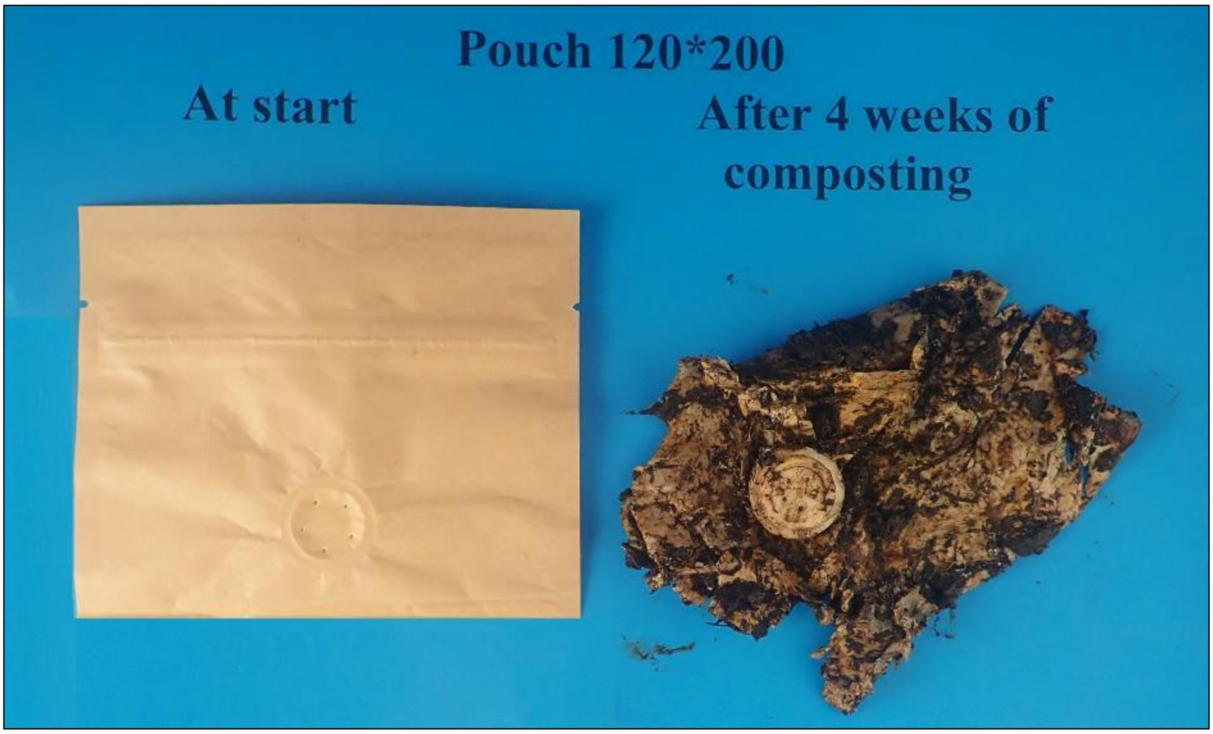

*Figure 13. Visual comparison between Pouch 120*200 (thickness: 112 µm (body)), cut into 2 pieces, at start and after 4 weeks of composting*

*Figure 14. Visual presentation of the contents of a test bin with 1% Pouch 120*200 (thickness: 112 µm (body)), cut into 2 pieces, after 6 weeks of composting*

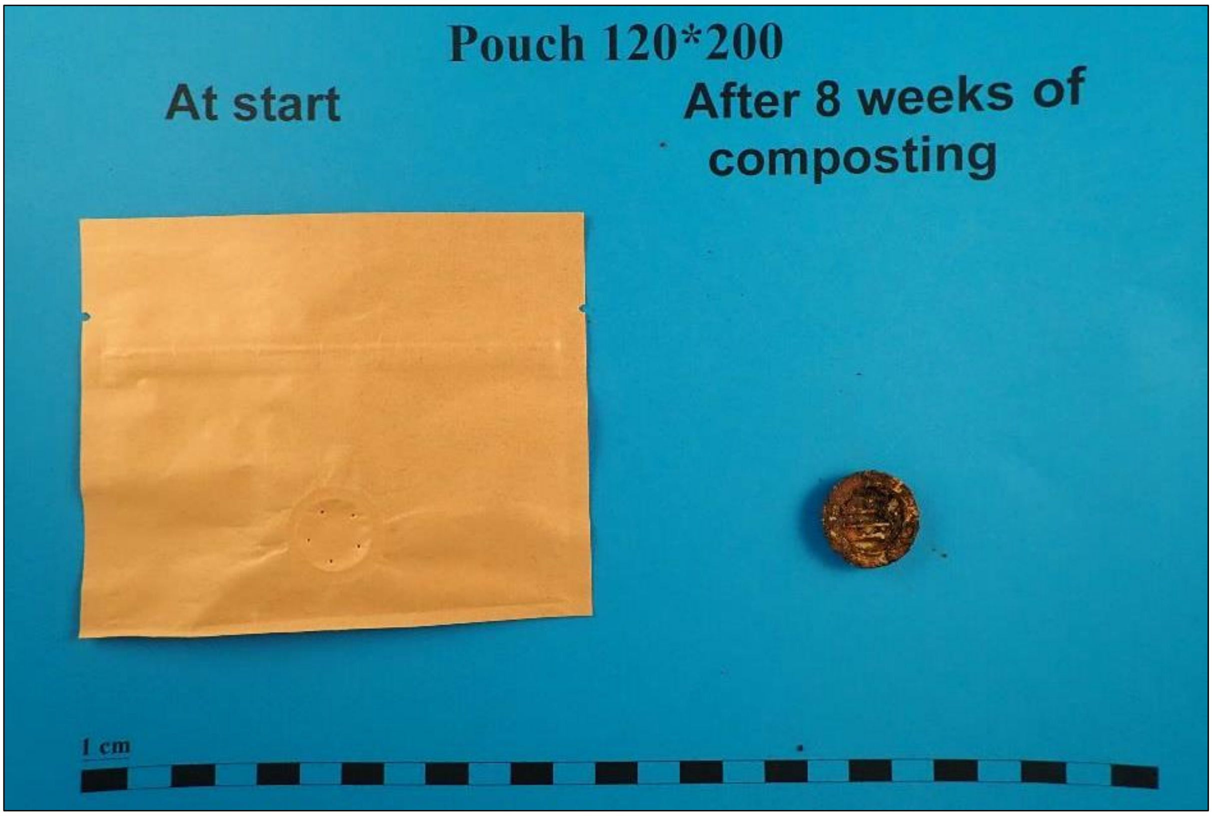

*Figure 15. Visual comparison between Pouch 120*200 (thickness: 112 µm (body)), cut into 2 pieces, at start and after 8 weeks of composting*

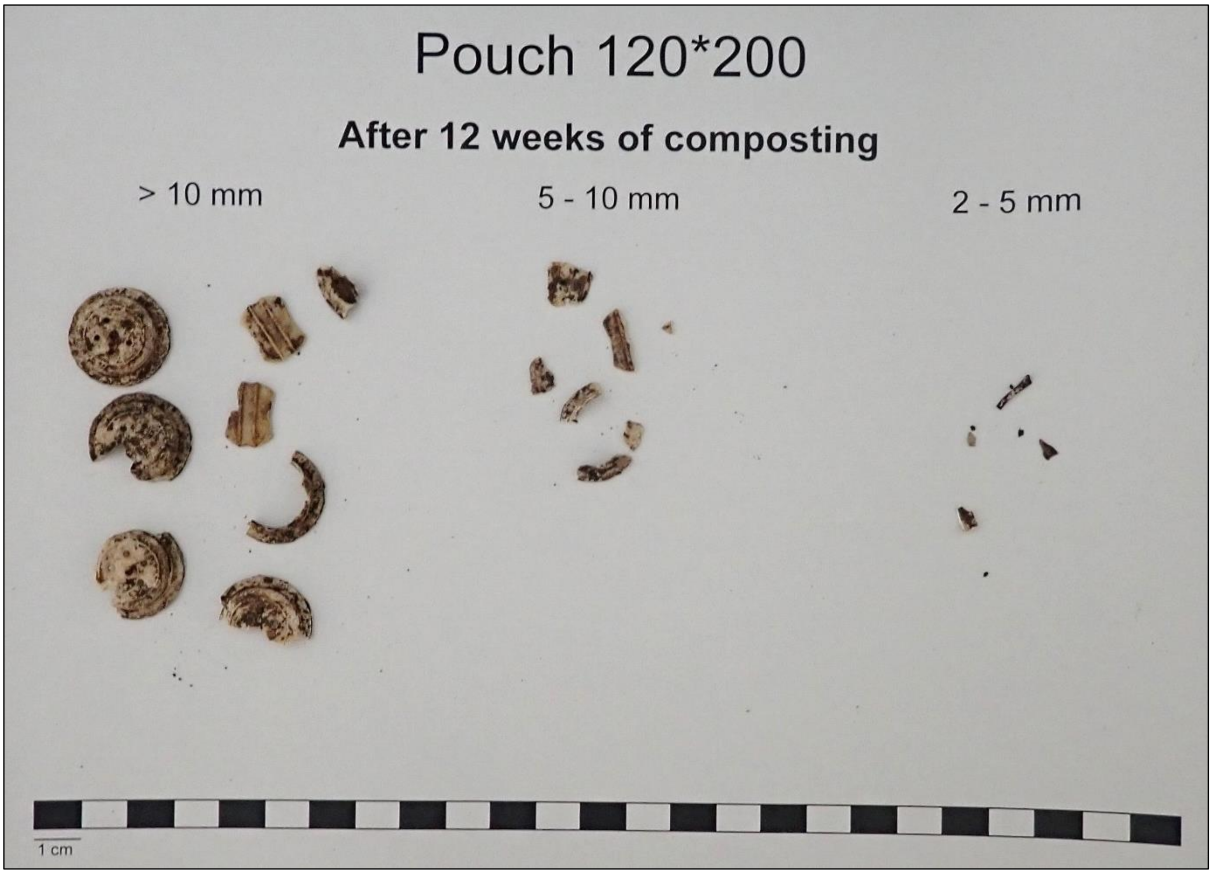

*Figure 16. Visual presentation of some of the retrieved pieces of Pouch 120*200 (thickness: 112 µm (body)) in the different sieving fractions at the end of the test*

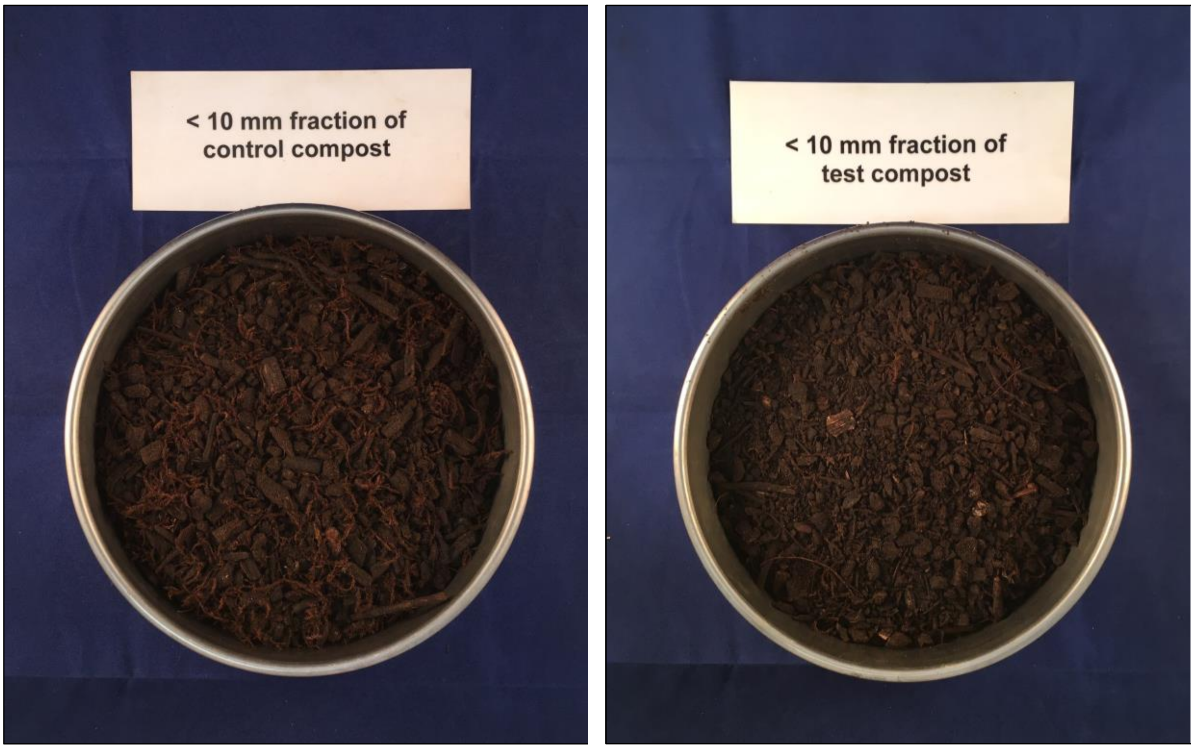

*Figure 17. Visual comparison between the < 10 mm fraction of control and Pouch 120*200 compost obtained after 12 weeks of composting*

8.7 Sieving - disintegration

At the end of the composting test (after 12 weeks), the contents of the test bin were used for sieving, sorting, further isolation, analyses and disintegration was calculated. Figure 18 shows the distribution in terms of percentage of the different compost fractions (< 2 mm, 2-5 mm, 5-10 mm, > 10 mm) on dry weight basis of the control and test composts. Disintegration is defined as a size reduction to < 2 mm. After carefully selecting all fractions (2-5 mm, 5-10 mm, > 10 mm) no pieces of the body or zipper of the pouch were found. Only a considerable amount of vent valve pieces could be retrieved. Therefore, it can be concluded that a disintegration percentage of 100% was obtained for Pouch 120*200 without vent valve. Consequently, the 90% pass level as required by the European norm EN 13432 *Requirements for packaging recoverable through composting and biodegradation - Test scheme and evaluation criteria for the final acceptance of packaging* (2000), the American standard ASTM D6868 *Standard Specification for Labeling of End Items that Incorporate Plastics and Polymers as Coatings or Additives with Paper and Other Substrates Designed to be Aerobically Composted in Municipal or Industrial Facilities* (2019) and the international standard ISO 18606 *Packaging and the environment - Organic recycling* (2013) was reached. From the results it can be concluded that test material Pouch 120*200 without vent valve (thickness: 112 µm (body), 1.36 mm (double zipper lock) and 1.33 mm (single zipper lock)) does fulfil the disintegration criterion in a pilot-scale composting test as prescribed by EN 13432 (2000), ASTM D6868 (2019) and ISO 18606 (2013).

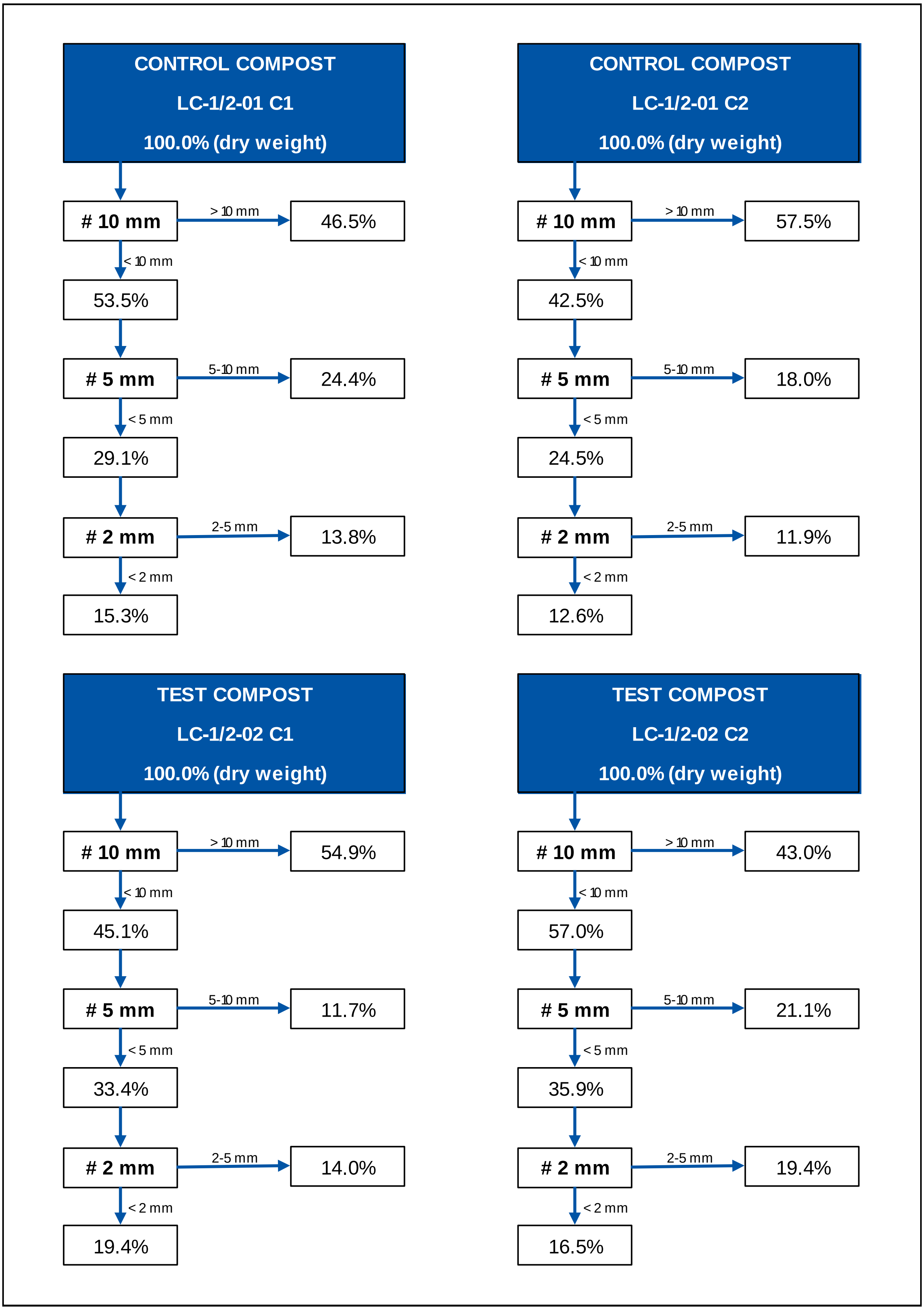

*Figure 18. Distribution in terms of percentage of the control and Pouch 120*200 compost fractions (on dry weight basis)*

8.8 Chemical analyses

The overall compost quality is determined by the analyses performed on the < 10 mm fraction (Table 6). The > 10 mm fraction, 5-10 mm, 2-5 mm and < 2 mm fraction of all composts were analysed for total solids and volatile solids content (Table 7).

To ensure a completion of the normal composting process, the blank biowaste control must have a Rottegrad of IV or V and volatile fatty acids content lower than 500 mg/kg at the end of the test. From Table 6 it can be seen that these requirements were fulfilled for control and test composts. The quality of the composts to which 1% Pouch 120*200 was added at start of the composting cycle was equally good compared to the control composts. No volatile fatty acids were found in the test and control composts and they all showed a Rottegrad of V, which demonstrates that the composts were stable and mature. An average pH of 8.7 and 9.0 was measured for the control and the test composts, respectively. A similar average salt content was obtained for the test composts (2240 µS/cm) and the control composts (2230 µS/cm). At the end of the test the NH_4^+-N content had decreased till low levels for all series and the NO_x^--N content had increased. After 12 weeks an average NO_x^--N content of 112 mg NO_x^--N/l and 10.2 mg NO_x^--N/l was obtained for the control and test composts, respectively. This indicates that the nitrification process had started and was proceeding well, albeit slightly delayed for the test composts. Average densities of 0.527 kg/l and 0.581 kg/l were found for the control and test composts, respectively. The C/N ratio varied between 9 and 14.

A higher average volatile solids degradation was observed for the test series when compared to the controls (Table 8). Moreover, a high volatile solids degradation was observed for all series, indicating that the composting process has proceeded well for all composts.

Table 6. Chemical analysis of the < 10 mm compost fraction after 12 weeks of composting

Parameter	Control composts		Test composts	
	LC-1/2 01 C1	LC-1/2 01 C2	LC-1/2 02 C1	LC-1/2 02 C2
Total solids (TS, %)	62.5	57.8	69.9	57.8
Volatile solids (VS, % on TS)	31.9	33.2	32.8	35.0
Ash (% on TS)	68.1	66.8	67.2	65.0
pH	8.5	8.8	8.9	9.0
Volatile fatty acids (g/l)	b.r.	b.r.	b.r.	b.r.
Total N (g/kg TS)	16.9	11.6	15.8	18.3
NH_4^+-N (mg/l)	17.2	10.0	16.2	16.0
NO_x^--N (mg/l)	163	61.4	b.r.	11
Electrical conductivity (EC, µS/cm)	2170	2280	2450	2020
Rottegrad	V	V	V	V
Density (kg/l)	0.512	0.543	0.588	0.573
C/N	9	14	10	10

b.r. = below reporting limit
Reporting limit: VFA = 0.3 g/l

Table 7. Dry matter and volatile solids content of different compost fractions after 12 weeks of composting

Parameter	Control composts		Test composts	
	LC-1/2-01 C1	LC-1/2-01 C2	LC-1/2-02 C1	LC-1/2-02 C2
> 10 mm fraction				
Total solids (TS, %)	58.9	56.9	66.7	54.2
Volatile solids (VS, % on TS)	45.3	40.5	33.9	47.4
Ash (% on TS)	54.7	59.5	66.1	52.6
5 - 10 mm fraction				
Total solids (TS, %)	55.7	53.2	66.2	55.4
Volatile solids (VS, % on TS)	44.9	38.1	38.8	39.0
Ash (% on TS)	55.1	61.9	61.2	61.0
2 - 5 mm fraction				
Total solids (TS, %)	60.3	58.6	67.7	58.9
Volatile solids (VS, % on TS)	41.8	35.6	31.6	33.9
Ash (% on TS)	58.2	64.4	68.4	66.1
< 2 mm fraction				
Total solids (TS, %)	69.2	63.7	66.7	66.1
Volatile solids (VS, % on TS)	25.3	26.2	33.8	25.8
Ash (% on TS)	74.7	73.8	66.2	74.2

Table 8. Volatile solids degradation for the different test series

Test series	Volatile solids degradation	
	Average %	%
Control composts	58.6	
LC-1/2-01 C1		56.8
LC-1/2-01 C2		60.4
Test composts	63.6	
LC-1/2-02 C1		64.3
LC-1/2-02 C2		62.9

ANNEX 1

Visual presentation of the disintegration by means of slide frames taken during the composting process of

Pouch 120*200 without vent valve
(thickness: 112 µm (body),
1.36 mm (double zipper lock),
1.33 mm (single zipper lock))

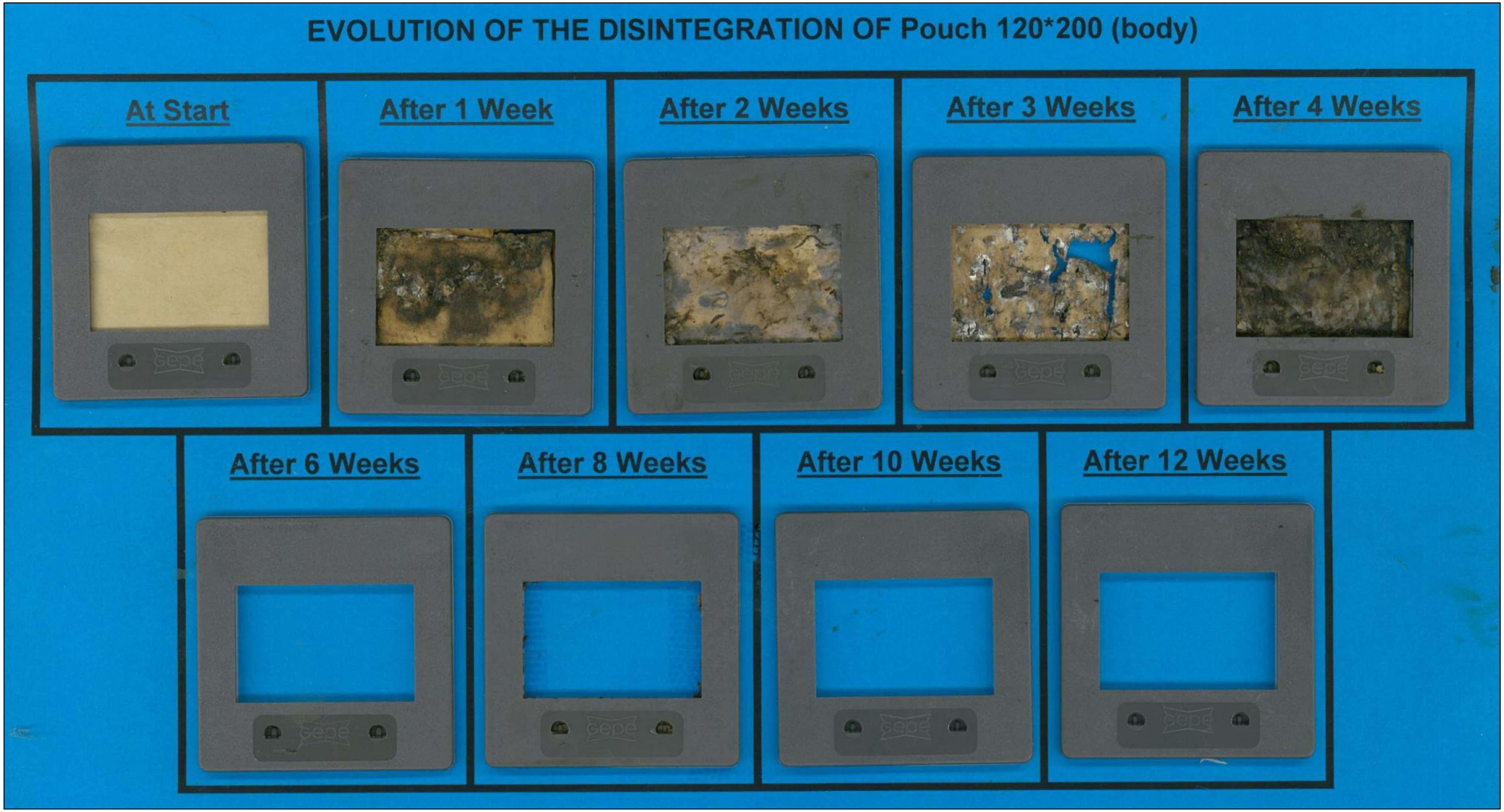

*Figure 19. Visual presentation of the evolution of the disintegration of the body of test item Pouch 120*200 (thickness: 112 µm) in slide frames*

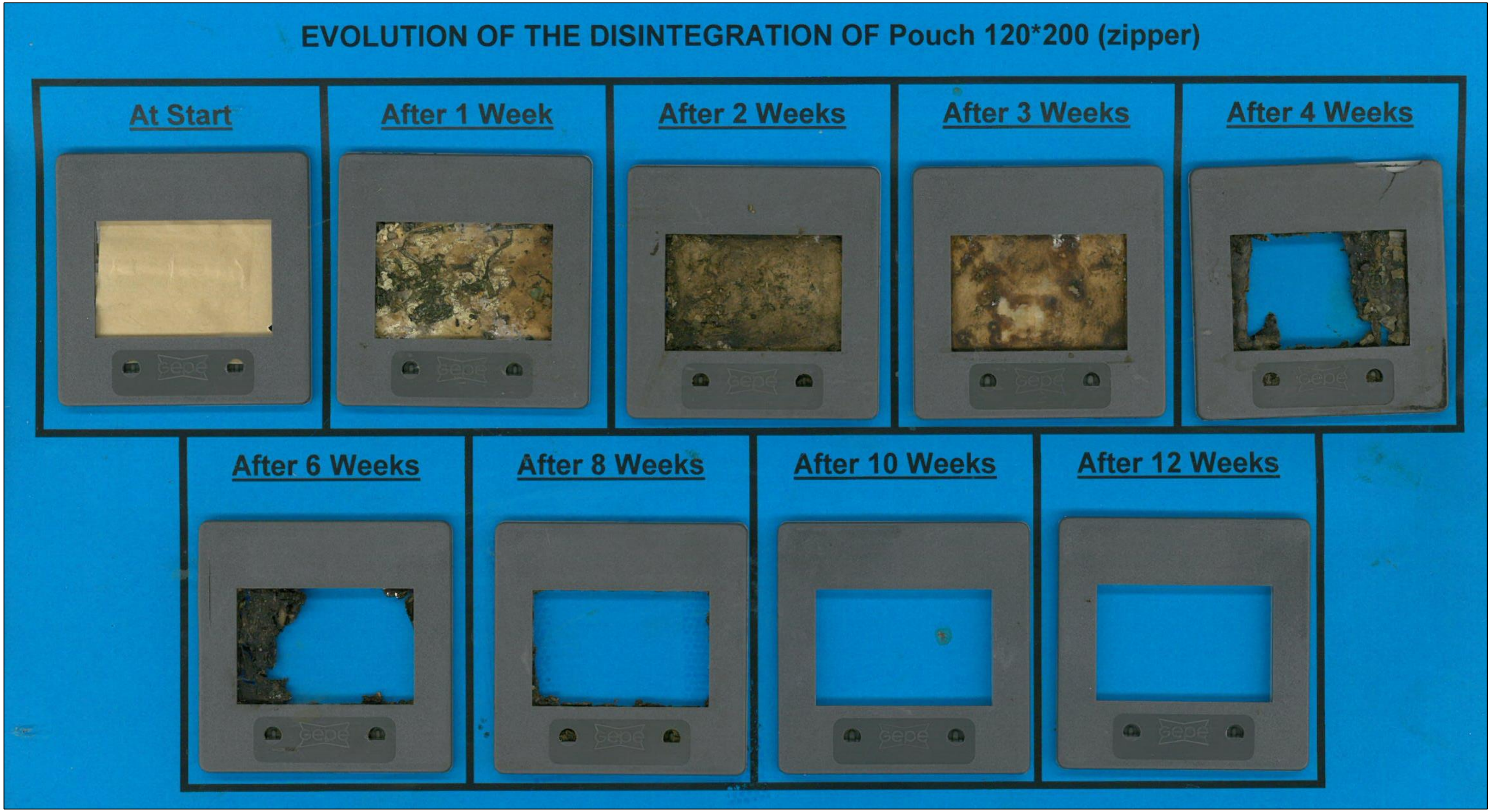

*Figure 20. Visual presentation of the evolution of the disintegration of the seal of test item Pouch 120*200 (thickness: 1.36 mm (double zipper lock), 1.33 mm (single zipper lock)) in slide frames*