FINAL REPORT
SUCH-1

Standard soil biodegradation test on **Balloon**

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1 Identification of the test

Project number
SUCH-1

Conditions
The test was performed under screening conditions (in duplicate)

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Test item
Balloon

Reference item
Cellulose

Test duration
720 days

2 Introduction

2.1 Principle of test method

With this test method, biodegradation of a test item in soil can be followed. The test item can either be a pure chemical or a compost sample with residual test product.

The test item is directly mixed with soil and incubated in the dark at ambient room temperature. Biodegradation is taking place through microbial activity. During the aerobic biodegradation in soil a mixture of gases, principally carbon dioxide and water, is produced. The CO_2 is captured in KOH and the CO_2 production is regularly determined by titration, which allows calculating the cumulative CO_2 production. The percentage of biodegradation can be calculated as the percentage of solid carbon of the test item, which has been converted to gaseous, mineral C under the form of CO_2. Also the kinetics of the biodegradation can be established.

The test is considered valid if:

- The degree of biodegradation of the reference material is more than 60% at the plateau phase or at the end of the test;
- The deviation of the CO_2 production in the different replicates of the control reactors (with standard soil only) is less than 20% of the mean at the plateau phase or at the end of the test.

2.2 Standard followed

- ISO 17556 *Plastics - Determination of the ultimate aerobic biodegradability in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved* (2012).

3 Results

3.1 Test set-up, analyses of inoculum and test item

The aerobic biodegradation of test item Balloon was evaluated in a standard soil biodegradation test at an incubation temperature of 25°C ± 2°C. The test was performed in duplicate. Reference item cellulose was added as powder. In equal amounts, three different colours of balloon were used in the test i.e. blue, white and pink. Before start-up the balloons were cut into pieces < 2 mm. At start-up 2 g of reference or test item was added to 510 g of inoculum (= 500 g standard soil + 10 g nutrients solution). The control reactors contained only 510 g of inoculum. The total test duration was 720 days.

The standard soil was a mixture of 70% industrial quartz sand, 10% kaolinite clay, 16% natural soil and 4% mature compost (percentages on dry weight basis). The natural soil was collected from a sandy field in Lokeren and 2 types of forest in Moerbeke (all located in Belgium). The mixture consisted of 1/3 field soil and 2/3 forest soils (equally divided). Before use, the soils were sieved over a 2 mm screen to remove stones and other inert materials, recognizable roots and other plant debris, and thoroughly mixed. The mature compost was derived from the organic fraction of municipal solid waste. The waste was stabilized and aerated in a composting bin at the laboratory under controlled conditions for more than 20 weeks. Before use the compost was sieved through 5 mm. Finally, salts were added to the standard soil by means of nutrients solution to obtain the final inoculum (see Table 1).

Table 1. Nutrients solution

Chemical	Amount (g/l)
KH_2PO_4	4.8
$MgSO_4$	2.4
$NaNO_3$	9.6
Urea	4.8
NH_4Cl	9.6

The characteristics of the soil inoculum are given in Table 2. The inoculum should have a water content between 40% and 60% of the total water holding capacity and a pH between 6.0 and 8.0. A total solids content of 85.7% was obtained. This corresponds with a moisture content on dry matter of 16.7% or 65.9% of the total water holding capacity. The moisture content was somewhat higher than prescribed, but still an optimal moisture content was obtained on an empirical basis. Throughout the test the moisture content was followed up closely and adjusted when needed. The inoculum showed a slightly too high pH of 8.5, but still a good degradation of reference item cellulose was observed, confirming the good quality of the inoculum. At the end of the test the pH of the different test series was within the prescribed range (Table 5). Finally, a C/N ratio of 7 was measured, ensuring a sufficient nitrogen presence.

Table 2. Characteristics of the inoculum

Characteristics	Inoculum
Total solids (TS, %)	85.7
Moisture content (%)	14.3
Volatile solids (VS, % on TS)	4.0
Ash content (% on TS)	96.0
pH	8.5
Electrical conductivity (EC, µS/cm)	984
Total water holding capacity (WHC_{tot}, % on TS)	25.3
Total N (g/kg TS)	2.8
C/N	7

According to ISO 17556 (2012) the test is considered valid when the deviation of the CO_2 production of the control reactors is less than 20% of the mean at the plateau phase or at the end of the test. After 720 days (end of test) a value of 1.1% was obtained, demonstrating the good precision of the test.

The reference and test item were analyzed for total solids (TS), volatile solids (VS) and total organic carbon content (TOC) (see Table 3).

Table 3. Total solids (TS), volatile solids (VS) and total organic carbon (TOC) content of the reference and test item

Test item	TS (%)	VS (% on TS)	TOC (%)
Cellulose	97.4	99.7	41.8
Balloon	99.9	94.5	81.0

3.2 Biodegradation percentages

Table 4 shows the net CO_2 production and the biodegradation percentage of the reference and test item after 720 days (end of test). The evolution of the average biodegradation percentage of cellulose and test item is given in Figure 1. The biodegradation percentage of the different replicates is shown in Figures 2 and 3.

Table 4. Net CO_2 production and biodegradation at the end of the test (720 days)

Test series	Net CO_2 (mg/g test item)	Biodegradation (%)		
		AVG	SD	REL
Cellulose	1299	84.9	2.5	100.0
Balloon	2206	74.3	6.0	87.5

With AVG = average, SD = standard deviation and REL = relative biodegradation

The biodegradation of reference item cellulose started almost immediately and proceeded at a good rate. After 27 days cellulose was already degraded by 60.6%. From then on biodegradation rate gradually slowed down and after 120 days a biodegradation of 79.6% ± 1.1% was reached. Biodegradation levelled off and at the end of the test (720 days) a plateau in biodegradation was reached at a level of 84.9% ± 2.5%. The test is considered valid when the degree of biodegradation of the reference material cellulose is more than 60% at the plateau phase or at the end of the test. This requirement was clearly fulfilled.

The biodegradation of test item Balloon started very slowly, but after 41 days biodegradation accelerated. Test item Balloon continued to degrade at a moderate rate and after 328 days a biodegradation of 53.5% was reached. Biodegradation gradually slowed down and at the end of the test (720 days) an absolute biodegradation of 74.3% ± 6.0% was reached. On a relative basis, compared to suitable reference substrate cellulose, a biodegradation of 87.5% was calculated. At the end of the test biodegradation of test item Balloon was still proceeding, especially for replicate RN 6 that was lagging a bit behind.

In general, for a material to be considered completely biodegradable, a minimum degradation of 90% absolute or relative to cellulose is required. The 90% limit must be reached within 2 years and the test should be executed at ambient temperature. From these results it can be concluded that test item Balloon did not meet this requirement within 720 days of testing under the given aerobic conditions, but biodegradation was very advanced.

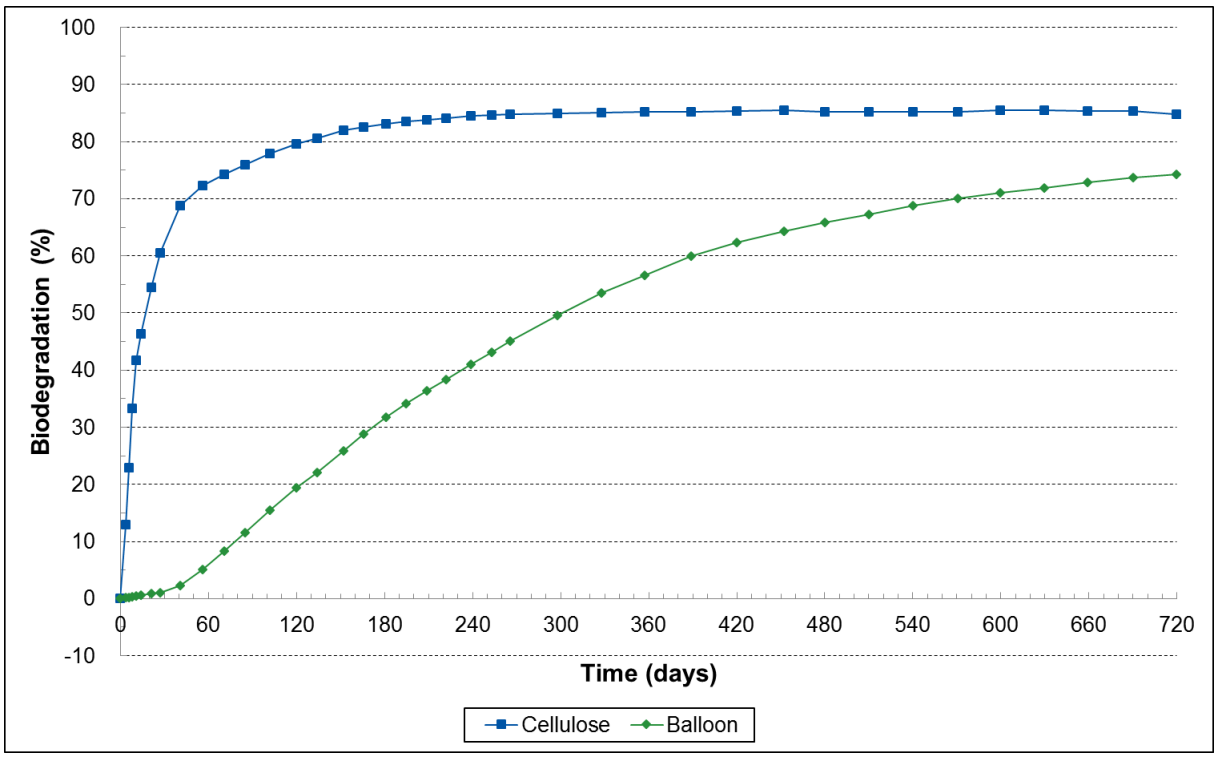

Figure 1. Evolution of the biodegradation percentage of reference and test item

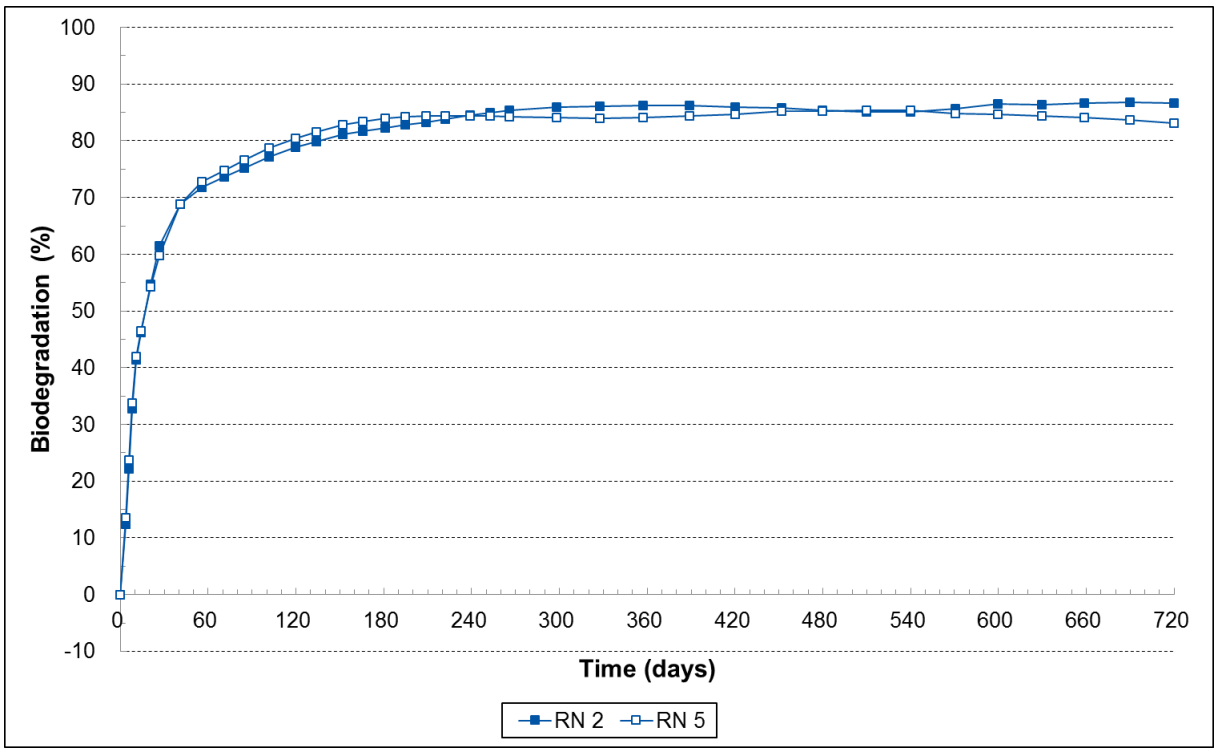

Figure 2. Evolution of the biodegradation percentage of the replicates of cellulose

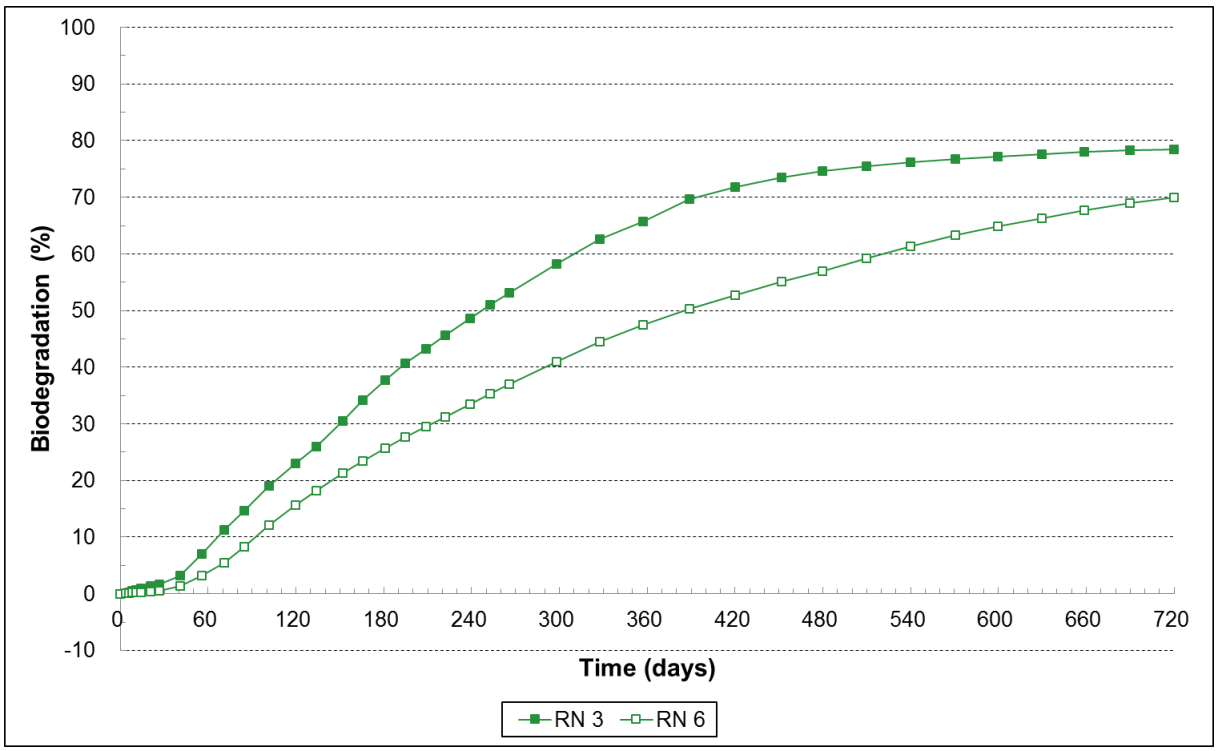

Figure 3. Evolution of the biodegradation percentage of the replicates of Balloon

3.3 Visual perceptions and analyses at end of test

At the end of the soil biodegradability test the different test series were examined for total solids (TS), volatile solids (VS) and pH (see Table 5). These results show that good moisture conditions were maintained during the test.

During the incubation, at each time of shaking, the reactors were inspected visually for several aspects such as moisture content, structure of the mixture, development of fungi and visual appearance of the test item. Good structure and moisture conditions were maintained throughout the test. No fungal growth was observed throughout the test. After 571 days test item Balloon became difficult to discern from the soil inoculum.

Table 5. pH, TS and VS content at the end of the test (720 days)

Test series	TS (%)	VS (% on TS)	pH
Control	88.4	3.1	7.7
Cellulose	88.4	3.0	7.7
Balloon	86.4	3.1	7.7

3.4 Disintegration

To follow up the disintegration of test item Balloon an extra reactor was included in the test. At start-up three 2.5 cm x 2.5 cm pieces of Balloon (one of each colour) and three balloon lips (one of each colour) were added to 510 g inoculum. After 6 weeks of incubation the test item remained intact, but red and yellow discoloration was observed on the white piece of sample (Figure 4). The first sign of disintegration was observed after 6 months of testing (Figure 5). The 2.5 cm x 2.5 cm pieces of test item started to show small tears. During the following months, more and more tears started to form and the 2.5 cm x 2.5 cm pieces started to break into smaller fragments (Figure 6). After 1 year of incubation only the lip of test item Balloon remained intact, while the softer pieces of sample have disintegrated further (Figure 7). At the end of the test (2 years) only the thick, rolled-up balloon ends could easily be recovered from

the soil (Figure 8). These lips remained more or less intact, but lost their elasticity and became brittle. Only small fragments of the softer pieces remained visible in the soil (Figure 9).

Figure 4. Visual presentation of Balloon after 6 weeks of incubation in soil

Figure 5. Detailed view of signs of disintegration of Balloon after 6 months of incubation

Figure 6. Visual presentation of Balloon after 9 months of incubation in soil

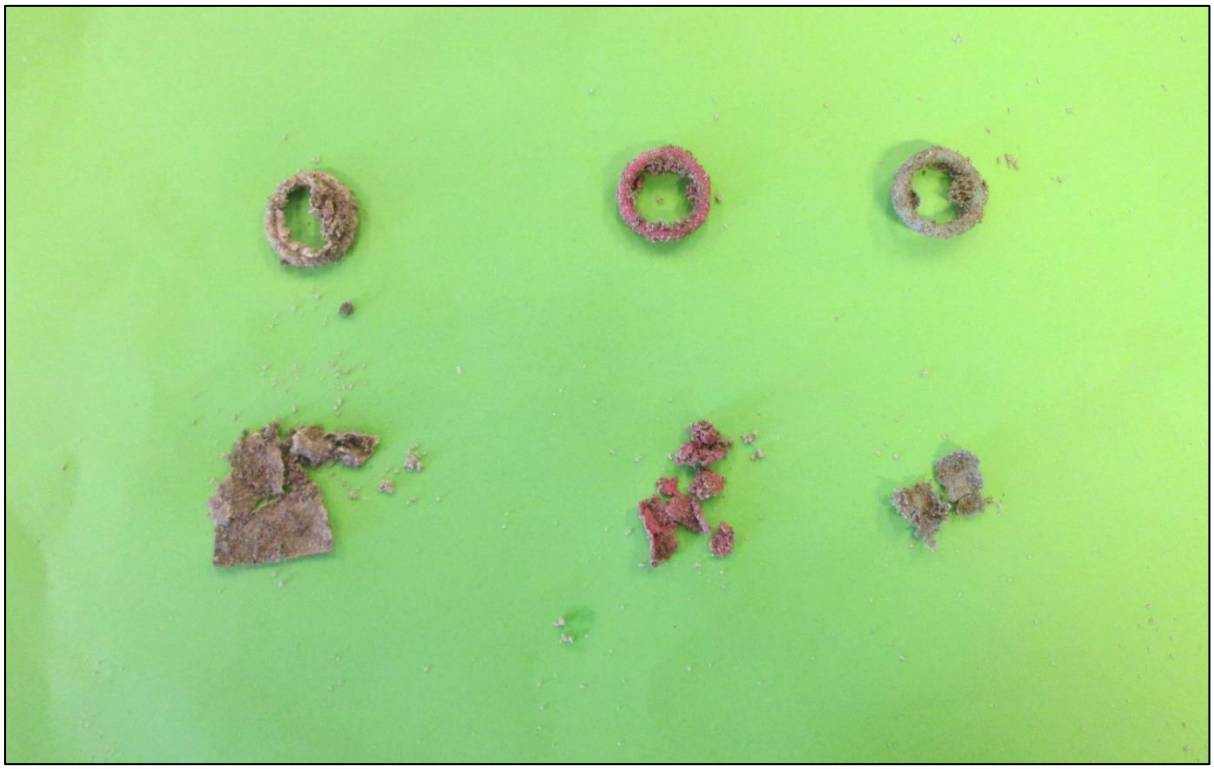

Figure 7. Visual presentation of Balloon after 1 year of incubation in soil

Figure 8. Visual presentation of Balloon after 2 years of incubation in soil

Figure 9. Visual presentation of the soil after 2 years of incubation

4 Summary and conclusions

The aerobic biodegradation of Balloon was evaluated in a standard soil biodegradation test according to ISO 17556 (2012). The incubation temperature was continuously kept at 25°C ± 2°C. The total test duration was 720 days.

According to ISO 17556 (2012) the test is considered valid when the standard deviation of the CO_2 production of the control reactors is less than 20% of the mean at the plateau phase or at the end of the test. After 720 days a value of 1.1% was obtained. Furthermore the degree of biodegradation of the reference material (cellulose) should be more than 60% at the plateau phase or at the end of the test. After 720 days a plateau in biodegradation was reached at a level of 84.9% ± 2.5%. Both requirements were clearly fulfilled.

The biodegradation of test item Balloon started very slowly, but accelerated after approximately six weeks. From that moment on the test item continued to degrade well for the remainder of the test. After 720 days (end of test) an absolute biodegradation of 74.3% ± 6.0% was reached, or 87.5% relative to reference substrate cellulose. At the end of the test biodegradation of test item Balloon was still proceeding.

In general, for a material to be considered completely biodegradable, a minimum degradation of 90% absolute or relative to cellulose is required. The 90% limit must be reached within 2 years and the test should be executed at ambient temperature. From these results it can be concluded that test item Balloon did not meet this requirement within 720 days of testing under the given aerobic conditions, but biodegradation was very advanced.

The disintegration of three 2.5 cm x 2.5 cm pieces of Balloon and three balloon lips was evaluated in an extra reactor with 510 g inoculum. Over the course of 2 years the pieces of Balloon disintegrated well and only small fragments could be recovered at the end of the test. The thick Balloon lips became brittle and lost their elasticity, but remained more or less intact.

Gent, December 5th, 2017

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