

FINAL REPORT MNI-1

High Solids Anaerobic Biodegradation and Disintegration test of **Undyed mink fur, Undyed fox fur, Dyed mink fur, Dyed fox fur and Fake fur**

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1 Identification of the test

Project number

MNI-1/1	High solids anaerobic biodegradation test of Undyed fox fur, Undyed mink fur, Dyed fox fur and Dyed mink fur
MNI-1/2	High solids anaerobic disintegration test of Undyed fox fur, Undyed mink fur, Dyed fox fur, Dyed mink fur and Fake fur
MNI-1/3	High solids anaerobic biodegradation test of Fake fur

Conditions

The test was performed under screening conditions

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Test items

Undyed mink fur
Undyed fox fur
Dyed mink fur
Dyed fox fur
Fake fur

Reference item

Cellulose

Test duration

30 days

2 Introduction

2.1 Principle of test method

The biodegradability of products in a sanitary landfill or in a solid state anaerobic digestion system is determined through high-rate dry anaerobic batch fermentation. This method simulates and accelerates the biodegradation process that takes place in a landfill because it is a stationary (no mixing) and dry fermentation under optimal conditions. The incubation temperature was $37^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

A small amount of test item is added to a large amount of highly active inoculum that has been stabilised prior to the start of the digestion period. Optimal conditions with regard to pH, nutrients, volatile fatty acids, etc. are provided and the mixture is left to ferment batch-wise. Likewise biodegradation is not influenced by other factors than those inherent to the test item itself.

During the anaerobic biodegradation of organic materials, a mixture of gases, principally methane and carbon dioxide, are the final decomposition products while some of the organic material will be assimilated for cell growth. The volume of the biogas produced is measured and the amount of CH_4 and CO_2 produced per weight unit of test item is calculated. If the carbon content of the test item is known the percentage of biodegradation can be calculated as the percentage of solid carbon of the test item that has been converted to gaseous, mineral C.

To evaluate the disintegration of a product in a sanitary landfill or in a solid state anaerobic digestion system, recognisable pieces of test item are added to a large amount of highly active inoculum and left to ferment batch-wise. At regular intervals (e.g. weekly) a piece of test item is removed from a test reactor and physical changes in the test item are observed.

2.2 Standard followed

The test was performed according to the following standard:

- ISO 15985 Plastics – Determination of the ultimate anaerobic biodegradation and disintegration under high-solids anaerobic-digestion conditions - Method by analysis of released biogas (2014)
- ASTM D5511 *Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions* (2018).

3 Results biodegradation tests (MNI-1/1 and MNI-1/3)

3.1 Test set up

A set of 18 equal vessels with a total volume of 2.5 l each was used. Each reactor was filled with 1000 g of inoculum and 15 g of reference or test item (except for the control reactors). The test items were cut into 2 cm by 2 cm pieces before adding them to the reactors, the cellulose was added as a microcrystalline powder.

The reactors were kept at 37°C ± 2°C in an incubator. The test set-up is given in Table 1.

Table 1. Test set up of the high solids anaerobic biodegradation test

RN	Test code	Test series	Inoculum (g)	Item (g)
1	MNI-1/1	Control	994.5	-
2	MNI-1/1	Cellulose	993.1	14.9
3	MNI-1/1	Undyed mink fur	1046.0	14.9
4	MNI-1/1	Undyed fox fur	996.1	15.6
5	MNI-1/1	Dyed mink fur	997.0	15.0
6	MNI-1/1	Dyed fox fur	996.8	14.8
7	MNI-1/1	Control	994.8	-
8	MNI-1/1	Cellulose	992.9	14.9
9	MNI-1/1	Undyed mink fur	997.4	14.9
10	MNI-1/1	Undyed fox fur	997.2	16.0
11	MNI-1/1	Dyed mink fur	998.1	15.0
12	MNI-1/1	Dyed fox fur	997.8	15.1
13	MNI-1/3	Control	997.7	-
14	MNI-1/3	Cellulose	995.0	14.9
15	MNI-1/3	Fake fur	994.8	15.1
16	MNI-1/3	Control	998.4	-
17	MNI-1/3	Cellulose	994.8	14.9
18	MNI-1/3	Fake fur	995.0	15.0

3.2 Analyses of inoculum, reference and test items

The inoculum was taken from a digester that has been operated during several months on the organic fraction of household waste. Before use, the inoculum was left to stabilize during 7 days. This post-fermentation was needed to reduce the biogas production rate. The characteristics of the inoculum are given in Table 2. It is recommended that the pH is between 7.5 and 8.5, the NH₄⁺-N content between 0.5 and 2.0 g/kg and the volatile fatty acids content < 1 g/kg. The quality of the inoculum was good as all of the recommendations were fulfilled for both tests.

The reference and test item were analysed for total solids (TS), volatile solids (VS) and total organic carbon content (TOC). The results are given in Table 3.

Table 2. Characteristics of the inoculum

Characteristics	Inoculum MNI-1/1	Inoculum MNI-1/3
Total solids (TS, %)	20.1	21.6
Volatile solids (VS, % on TS)	65.5	64.3
Ash content (% on TS)	34.5	35.7
pH	8.1	8.1
Volatile fatty acids (VFA, g/kg)	0.17	b.r.
NH ₄ ⁺ -N (g/kg)	1.52	1.14

b.r. = below reporting limit; reporting limit: VFA = 0.14 g/kg

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

Test item	TS (%)	VS (% on TS)	TOC (%)
Cellulose	97.0	100.0	42.7
Undyed mink fur	89.6	96.9	44.6
Undyed fox fur	91.9	94.9	44.8
Dyed mink fur	89.8	95.5	42.1
Dyed fox fur	91.5	98.8	44.9
Fake fur	99.2	98.9	64.0

3.3 Biogas production

The averages and standard deviations of the final gas readings, after 30 days, in NI (normalized litre. i.e. litre converted to standard conditions of temperature and pressure) are summarized in Table 4. The background activity of the inoculum was clearly lower compared to the reference reactors for both MNI-1/1 and MNI-1/3. This shows that the inoculum was stabilized sufficiently during the post-fermentation period. The low background activity improved the accuracy of the tests.

Table 4. Average and standard deviation of the final gas production (NI) after 30 days

Test code	Test series	Biogas production (NI)	
		Average	Standard deviation
MNI-1/1	Control	6.8	0.3
MNI-1/1	Cellulose	17.0	0.4
MNI-1/1	Undyed mink fur	10.2	0.6
MNI-1/1	Undyed fox fur	8.6	0.7
MNI-1/1	Dyed mink fur	9.0	0.1
MNI-1/1	Dyed fox fur	7.6	0.2
MNI-1/3	Control	7.1	0.1
MNI-1/3	Cellulose	17.1	0.2
MNI-1/3	Fake fur	7.1	0.0

Table 5 shows the biogas composition after 30 days of testing. The gas compositions were within a normal range for all reactors. The composition of the biogas has no influence on the biodegradation percentage, but gives an idea on the fermentation process. A high CO₂ concentration and a low CH₄ content could indicate a bad fermentation. As can be seen from Table 5 this was certainly not the case for the test items.

Table 5. Average biogas composition (%) at the end of the test of the different test series

Test code	Test series	CO ₂ content (%)	CH ₄ content (%)
MNI-1/1	Control	40.1	59.9
MNI-1/1	Cellulose	46.6	53.4
MNI-1/1	Undyed mink fur	39.3	60.7
MNI-1/1	Undyed fox fur	39.3	60.7
MNI-1/1	Dyed mink fur	39.6	60.4
MNI-1/1	Dyed fox fur	39.1	60.9
MNI-1/3	Control	40.4	59.6
MNI-1/3	Cellulose	47.3	52.7
MNI-1/3	Fake fur	39.5	60.5

3.4 Biodegradation percentages

Table 6 shows the biodegradation percentages of reference and test items after 30 days. They are calculated as the amount of carbon in the sample that was converted to carbon in the biogas (methane and carbon dioxide).

Table 6. Biodegradation percentages at the end of the test (30 days)

Test series	Average C_{input} (g)	Average $C_{gaseous}$ (g)	Biodegradation (%)		
			AVG	SD	REL
Cellulose*	6.4	5.4	85.7	2.0	100.0
Undyed mink fur	6.6	1.7	25.8	2.7	30.2
Undyed fox fur	7.1	1.0	13.6	4.9	15.9
Dyed mink fur	6.3	1.2	18.3	0.4	21.3
Dyed fox fur	6.7	0.4	6.6	2.0	7.7
Fake fur	9.6	0.0	0.3	0.1	0.4

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

* Average of MNI-1/1 and MNI-1/3

The values in Table 6 do not include the amount of carbon which was originally present in the test or reference item and which in the course of the digestion has been converted to biomass carbon. Some of the carbon that is biodegraded is indeed used for the building of new bacterial biomass. For anaerobic digestion the biomass yield factor is between 10% and 30%. This means that for 1 g of carbon consumed, between 10% and 30% is used for new cell biomass while 70% to 90% is converted to gaseous, mineral carbon under the form of CH₄ or CO₂.

Figure 1 shows the evolution of the average biodegradation percentages of the reference and test items. Figure 2 to Figure 7 show the evolution of the biodegradation percentage of all the replicates of cellulose (4 replicates – 2 for MNI-1/1 and 2 for MNI-1/3) and the test items.

The test is considered valid if after 15 days (according to ISO 15985) the biodegradation percentage of the reference item is more than 70% and if the standard deviation of the biodegradation percentage of the reference item is less than 20% at the end of the test. After a lag phase of about one day, biodegradation of cellulose started at a high rate in both MNI-1/1 and MNI-1/3. After about 6 days, a biodegradation percentage of already 79.7% was reached. Then biodegradation slowed down to reach a plateau. The final biodegradation (after 30 days) was 85.7% ± 2.0%, meaning that all requirements for a valid test were fulfilled.

Biodegradation of the Undyed mink fur, Undyed fox fur, Dyed mink fur and Dyed fox fur started immediately. The undyed mink fur shows the highest biodegradation (25.8%), followed by the dyed mink fur (18.3%), the undyed fox fur (13.6%) and the dyed fox fur (6.6%). Biodegradation reached a plateau for all test items. This means that these test items are partially biodegradable under mesophilic, high solids anaerobic conditions.

Biodegradation of the fake fur never started. At the end of the test a biodegradation percentage of 0.3% ± 0.1% was measured. The slightly positive result is considered due to natural variations in the biogas production of the inoculum. This means that the fake fur is not biodegradable under mesophilic, high solids anaerobic conditions.

To put the biodegradation of the (real) fur samples into perspective it is interesting to know the average biodegradation percentage of some 'natural products'. This data is included in Table 7.

Table 7. Average biodegradation percentage of natural products

Product	Average biodegradation (%)
Craft paper (bleached and lignin removed)	66
Newspaper	30
Cardboard	44
Grass (mostly roadside)	50
Straw (from wheat)	36
Leaves (oak, poplar, willow)	31

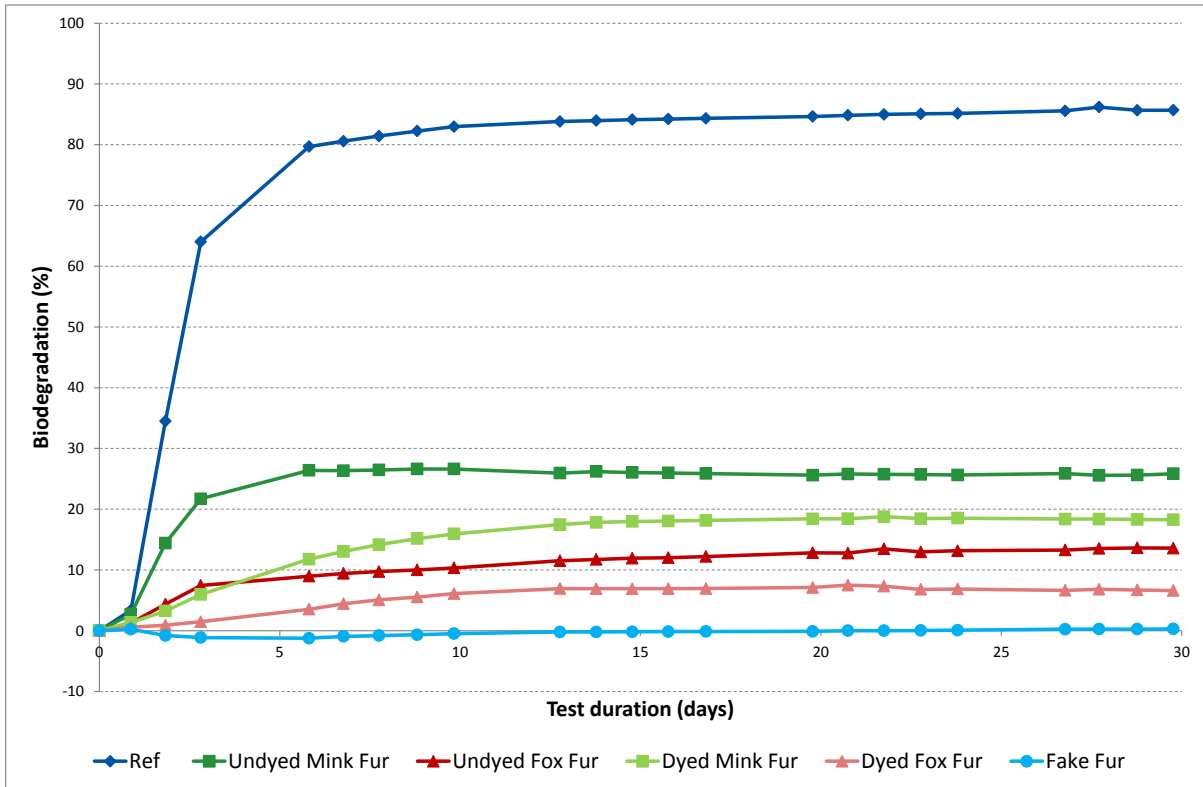


Figure 1. Evolution of the average biodegradation percentage of reference item and test items

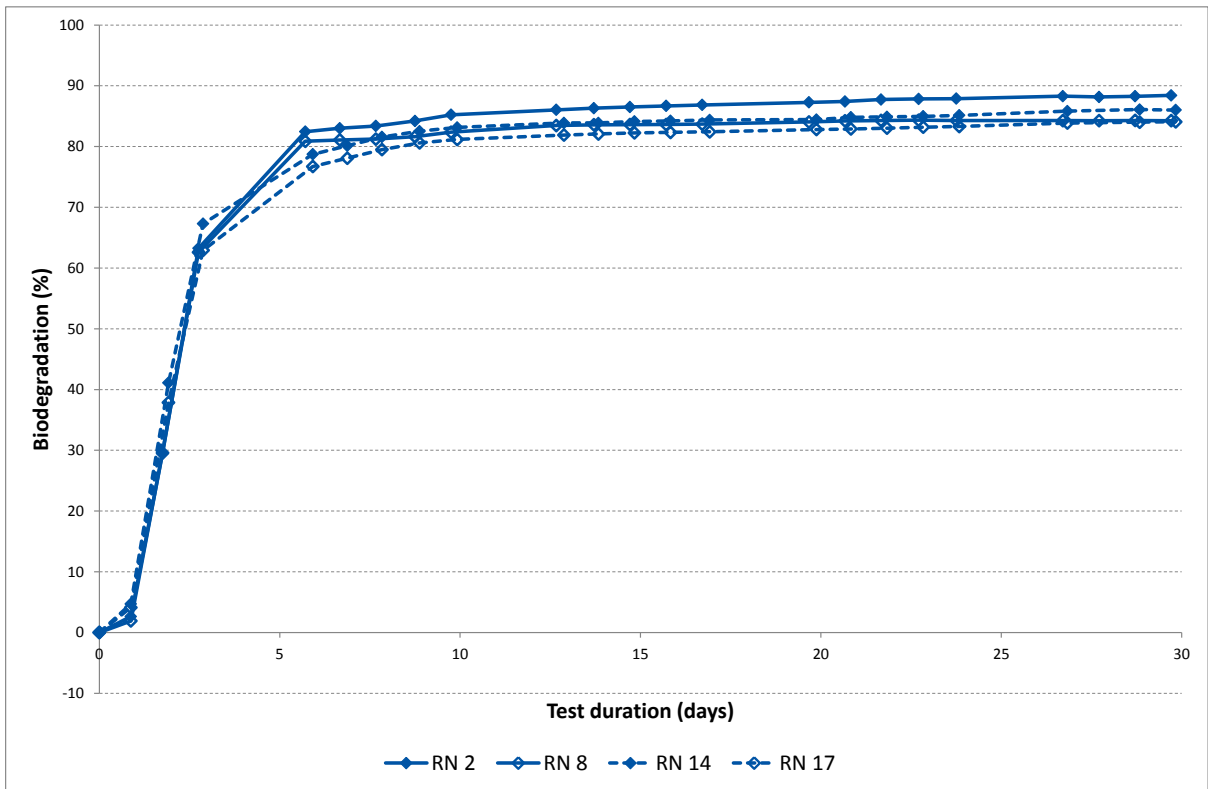


Figure 2. Evolution of the biodegradation percentage of the replicates of cellulose

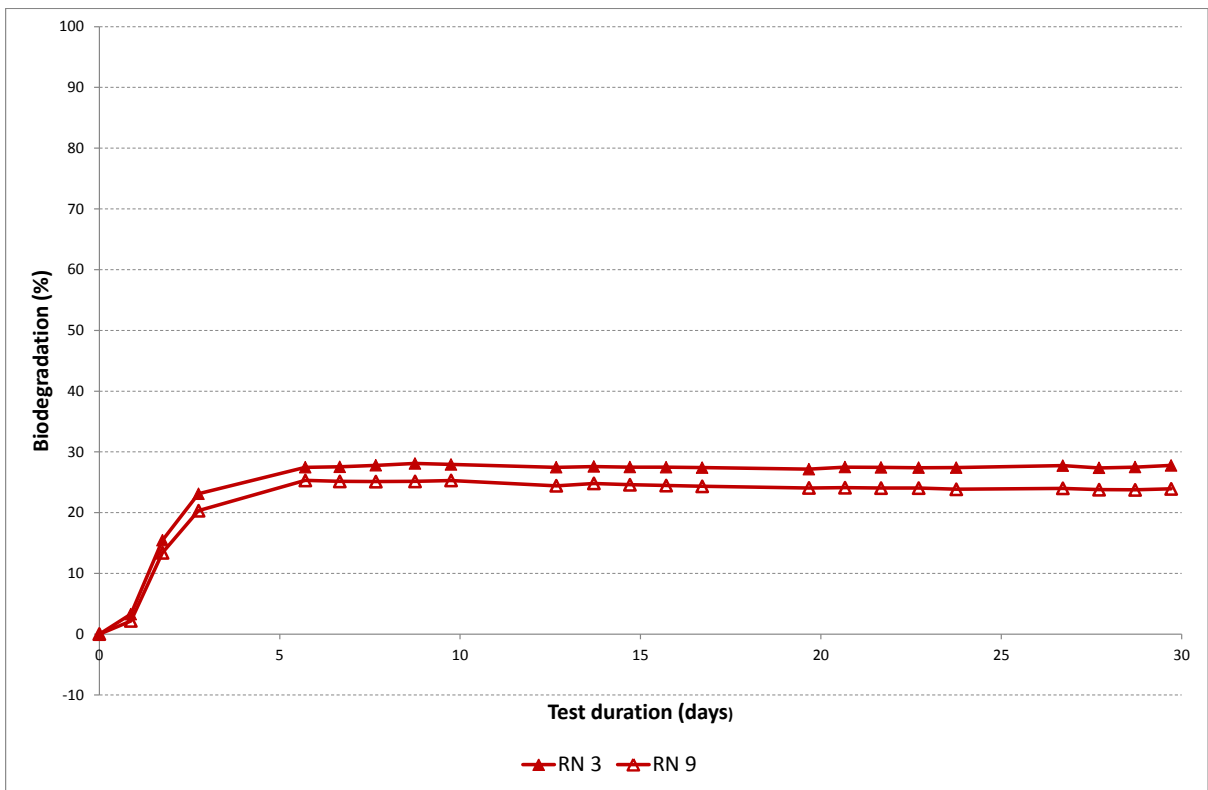


Figure 3. Evolution of the biodegradation percentage of the replicates of Undyed mink fur

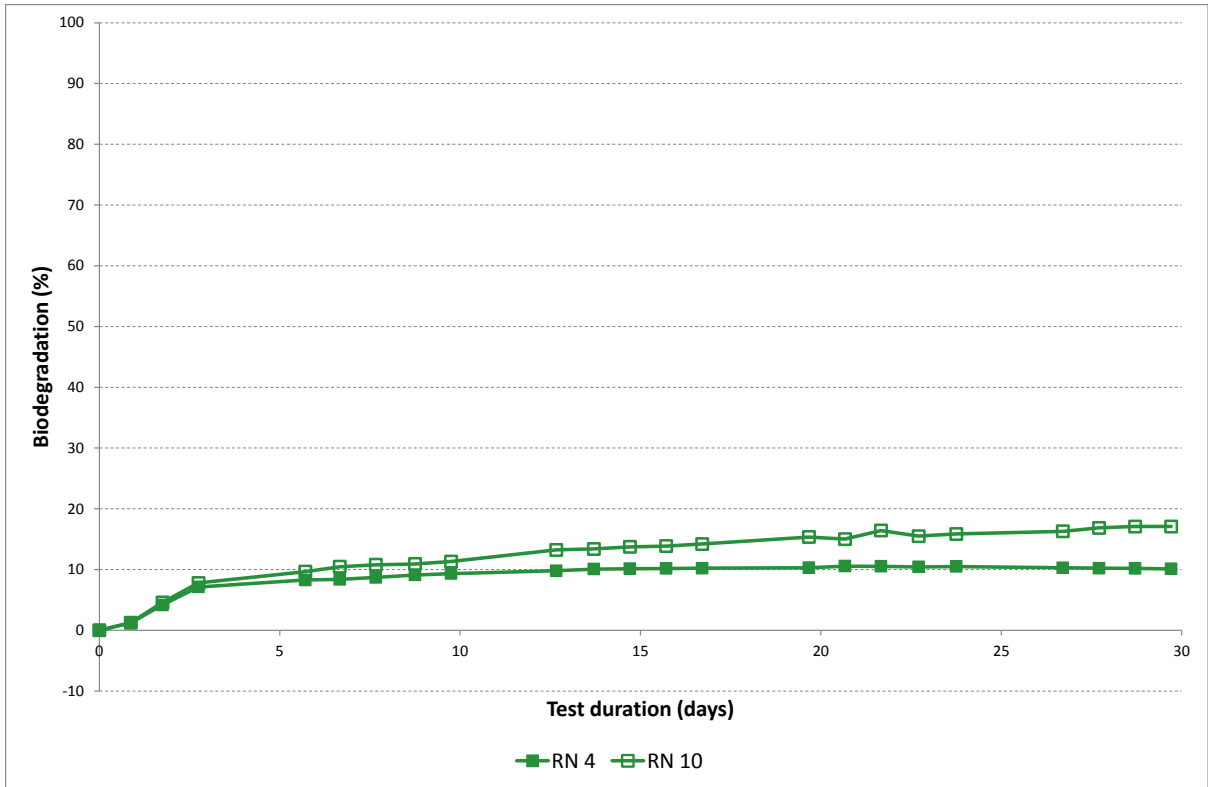


Figure 4. Evolution of the biodegradation percentage of the replicates of Undyed fox fur

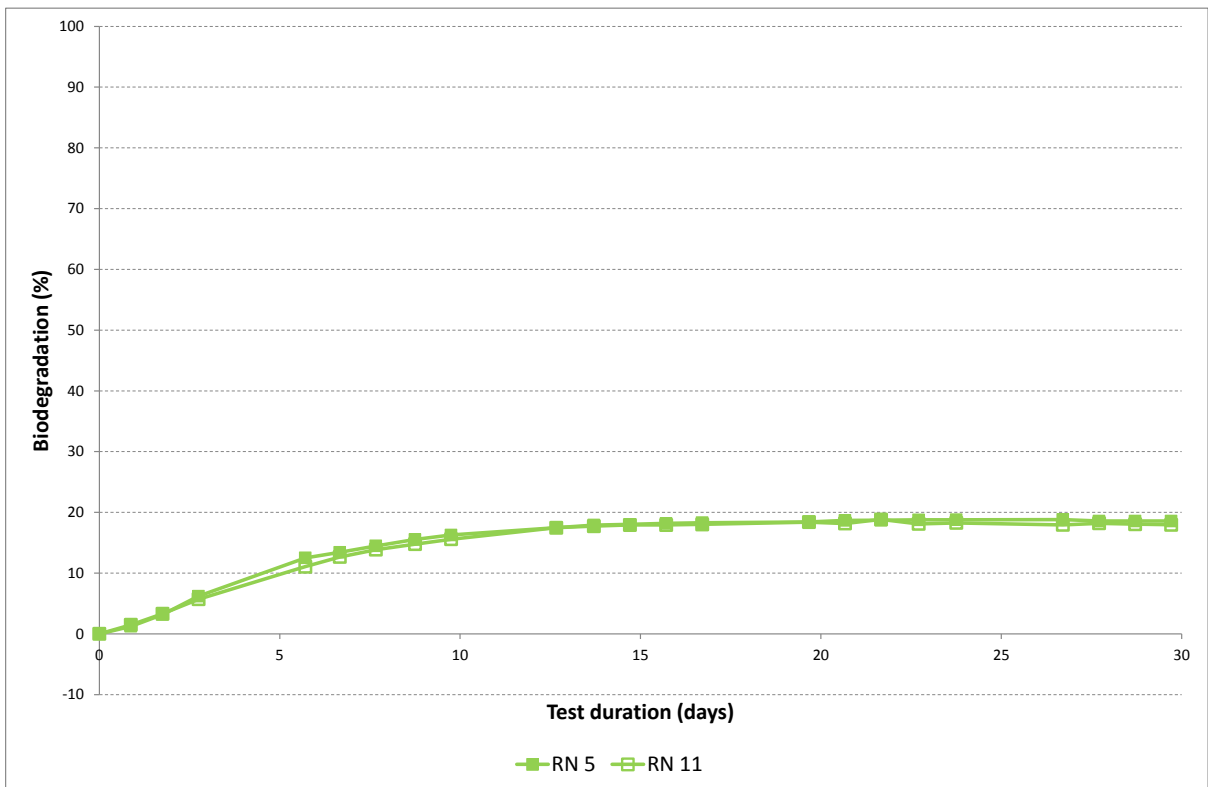


Figure 5. Evolution of the biodegradation percentage of the replicates of Dyed mink fur

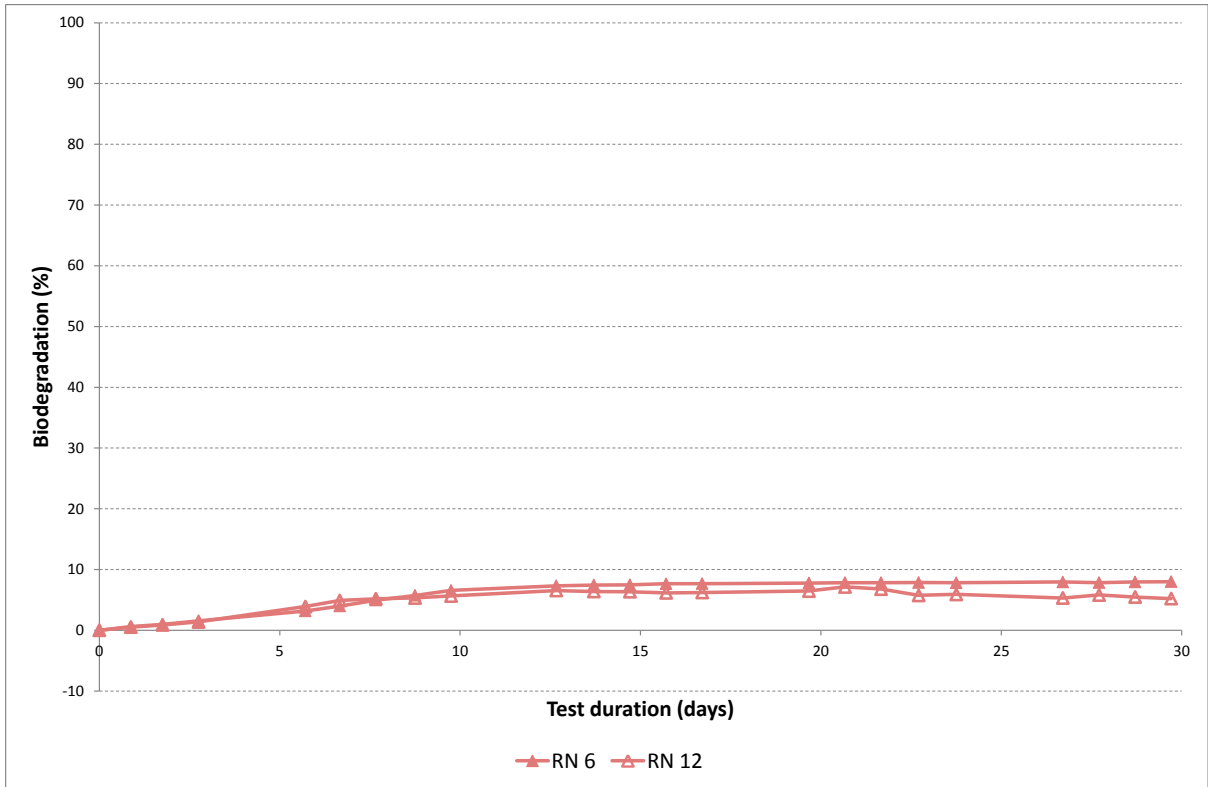


Figure 6. Evolution of the biodegradation percentage of the replicates of Dyed fox fur

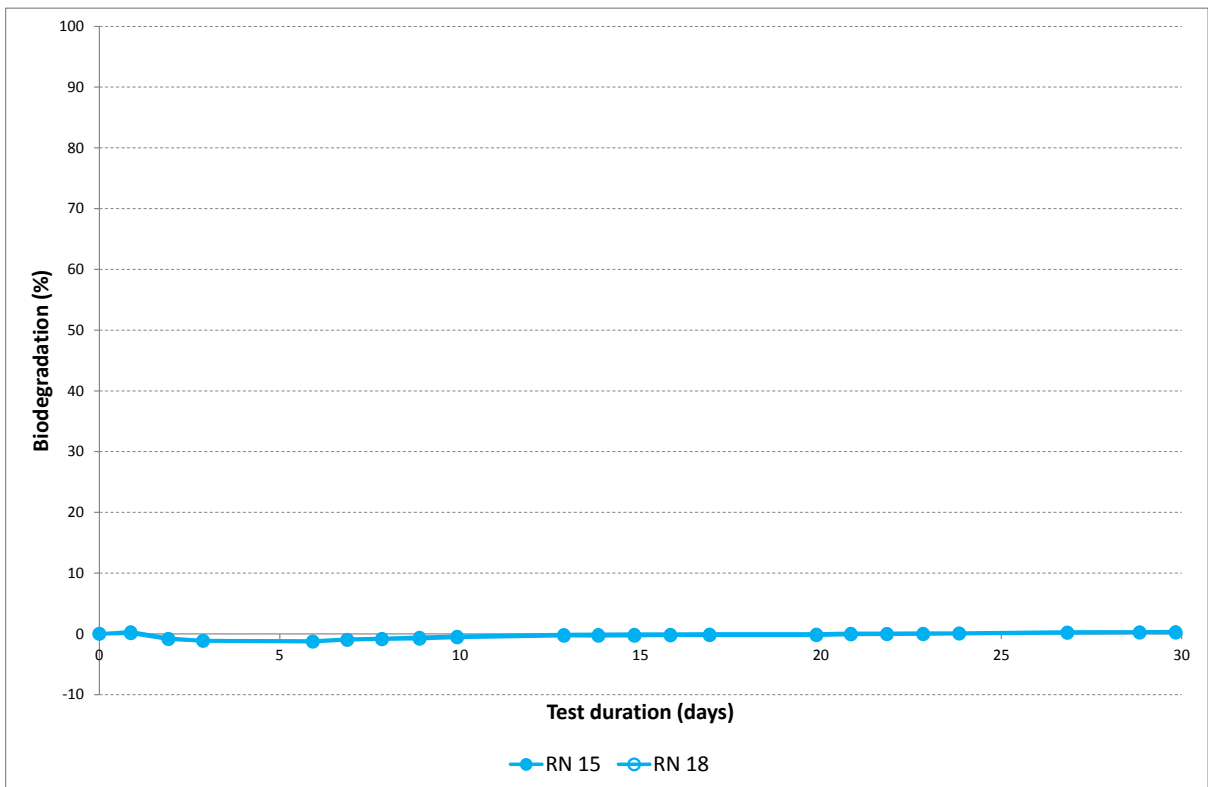


Figure 7. Evolution of the biodegradation percentage of the replicates of Fake fur

4 Results disintegration test (MNI-1/2)

4.1 Test set up

A series of 10 equal reactors with a volume of 2.5 l was used. The reactors were carefully filled with 1000 g of inoculum and 5 cm by 5 cm pieces of test items Undyed mink fur, Undyed fox fur, Dyed mink fur, Dyed fox fur and Fake fur. It was seen to that the pieces of test item are completely covered in inoculum. Each reactor contained 6 pieces of test item. The test was performed in duplicate at $37^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

Every week, a piece of test item was removed from the test reactors to assess the disintegration. The removed pieces were cleaned up as much as possible, photos were taken and visual observations were noted.

4.2 Analyses of inoculum

The same inoculum as MNI-1/2 was used for this test. The characteristics of the inoculum are given in Table 2. The quality of the inoculum was good as all of the recommendations were fulfilled.

4.3 Disintegration

In Table 8 to Table 12 a photo and the observations can be found for each test item and every test week.


In general, it was observed that the Undyed mink fur, Undyed fox fur, Dyed mink fur and Dyed fox fur partially disintegrated: the skin fell apart and disappeared but the hairs remained. The fake fur did not show any disintegration, only discolouration.

Table 8. Visual representation of the disintegration of the undyed mink fur

 <p>MNI-1/2 Undyed Mink Fur at start</p> <p>5 cm</p>	Undyed mink fur at start
 <p>MNI-1/2 Undyed Mink Fur after 1 week of desintegration</p> <p>5 cm</p>	Undyed mink fur after 7 days Skin has become weak and lost strength. The piece fell apart easily. Hairs were clearly visible and seemed intact.

<p>MNI-1/2 Undyed Mink Fur after 2 weeks of desintegration</p>  <p>5 cm</p>	<p>Undyed mink fur after 14 days</p> <p>The pieces were falling apart completely. Some skin was still left, but all strength was gone. Hairs detached very easily.</p>
<p>MNI-1/2 Undyed Mink Fur after 3 weeks of desintegration</p>  <p>5 cm</p>	<p>Undyed mink fur after 21 days</p> <p>The pieces were falling apart completely. Some skin was still left, but all strength was gone. Hairs detached very easily.</p>
<p>MNI-1/2 Undyed Mink Fur after 4 weeks of desintegration</p>  <p>5 cm</p>	<p>Undyed mink fur at end (30 days)</p> <p>The pieces were falling apart completely. Some skin was still left, but all strength was gone. Hairs detached very easily.</p>

Table 9. Visual representation of the disintegration of the undyed fox fur

	<p>Undyed fox fur at start</p> <p>(Blue background used to obtain better photo – hairs were difficult to distinguish on white background)</p>
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<p>MNI-1/2 Undyed Fox Fur after 1 week of desintegration</p>  <p>5 cm</p>	<p>Undyed fox fur after 7 days</p> <p>Skin had become weak and lost strength. It was difficult to remove the digestate from the piece without causing the piece to fall apart. Hairs were clearly visible and seemed intact.</p>
<p>MNI-1/2 Undyed Fox Fur after 2 weeks of desintegration</p>  <p>5 cm</p>	<p>Undyed fox fur after 14 days</p> <p>The skin was almost completely gone. Due to the longer hair (compared to mink), the pieces still hold together better, possibly due to digestate 'gluing' them together.</p>
<p>MNI-1/2 Undyed Fox Fur after 3 weeks of desintegration</p>  <p>5 cm</p>	<p>Undyed fox fur after 21 days</p> <p>The skin was almost completely gone. Due to the longer hair (compared to mink), the pieces still hold together better, possibly due to digestate 'gluing' them together.</p>
<p>MNI-1/2 Undyed Fox Fur after 4 weeks of desintegration</p>  <p>5 cm</p>	<p>Undyed fox fur at end (30 days)</p> <p>The pieces were falling apart, some skin was still left, but all strength was gone. Hairs detach very easily. Due to the longer hair (compared to mink), the pieces still hold together better, possibly due to digestate 'gluing' them together.</p>

Table 10. Visual representation of the disintegration of the dyed mink fur

<p>MNI-1/2 Dyed Mink Fur at start</p> 	<p>Dyed mink fur at start</p>
<p>MNI-1/2 Dyed Mink Fur after 1 week of desintegration</p> 	<p>Dyed mink fur after 7 days</p> <p>Skin had become weak and lost strength. It was difficult to remove the digestate from the piece without causing the piece to fall apart. Hairs were clearly visible and seemed intact.</p>
<p>MNI-1/2 Dyed Mink Fur after 2 weeks of desintegration</p> 	<p>Dyed mink fur after 14 days</p> <p>The pieces were falling apart completely. Some skin seems to remain (very hard to distinguish from the black digestate), but all strength was gone. Hairs detached very easily.</p>
<p>MNI-1/2 Dyed Mink Fur after 3 weeks of desintegration</p> 	<p>Dyed mink fur after 21 days</p> <p>The pieces were falling apart completely, some skin seems to remain (very hard to distinguish from the black digestate), but all strength was gone. Hairs detached very easily.</p>


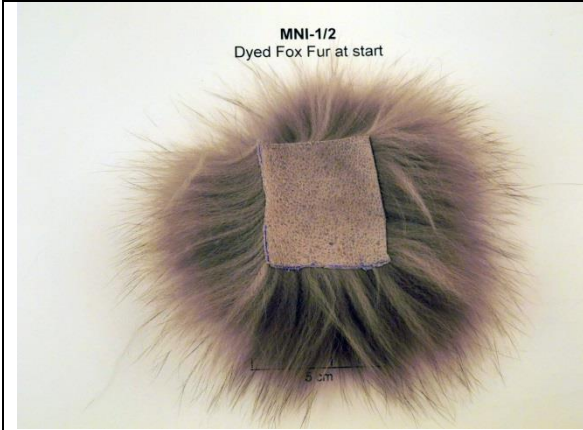


<p>MNI-1/2 Dyed Mink Fur after 4 weeks of desintegration</p> 	<p>Dyed mink fur at end (30 days)</p> <p>The pieces were falling apart completely, some skin seems to remain (very hard to distinguish from the black digestate), but all strength was gone. Hairs detached very easily.</p>
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Table 11. Visual representation of the disintegration of the dyed fox fur

<p>MNI-1/2 Dyed Fox Fur at start</p> 	<p>Dyed fox fur at start</p>
<p>MNI-1/2 Dyed Fox Fur after 1 week of desintegration</p> 	<p>Dyed fox fur after 7 days</p> <p>Skin has become weak and lost strength. It was difficult to remove the digestate from the piece without causing the piece to fall apart. Hairs were clearly visible and seemed intact.</p>
<p>MNI-1/2 Dyed Fox Fur after 2 weeks of desintegration</p> 	<p>Dyed fox fur after 14 days</p> <p>The skin was almost completely gone. Due to the longer hair (compared to mink), the pieces still hold together better, possibly due to digestate 'gluing' them together.</p>


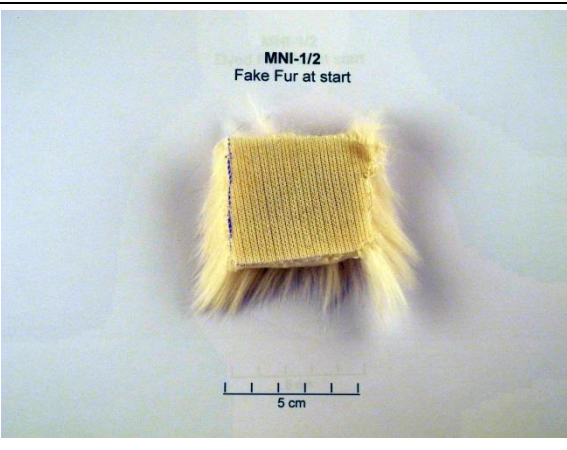
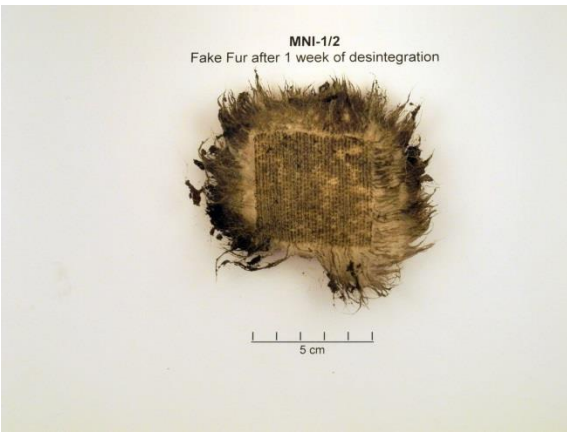



 <p>MNI-1/2 Dyed Fox Fur after 3 weeks of desintegration</p> <p>5 cm</p>	<p>Dyed fox fur after 21 days</p> <p>The skin was almost completely gone. Due to the longer hair (compared to mink), the pieces still hold together better, possibly due to digestate 'gluing' them together.</p>
 <p>MNI-1/2 Dyed Fox Fur after 4 weeks of desintegration</p> <p>5 cm</p>	<p>Dyed fox fur at end (30 days)</p> <p>The skin was almost completely gone and the sample falls to pieces. Due to the longer hair (compared to mink), the pieces still hold together better, possibly due to digestate 'gluing' them together.</p>

Table 12. Visual representation of the disintegration of the fake fur

 <p>MNI-1/2 Fake Fur at start</p> <p>5 cm</p>	<p>Fake fur at start</p>
 <p>MNI-1/2 Fake Fur after 1 week of desintegration</p> <p>5 cm</p>	<p>Fake fur after 7 days</p> <p>The sample looked intact, only discoloration due to the digestate was observed.</p>

 <p>MNI-1/2 Fake Fur after 2 weeks of desintegration</p>	<p>Fake fur after 14 days</p> <p>The sample looked intact, only discoloration due to the digestate was observed.</p>
 <p>MNI-1/2 Fake Fur after 3 weeks of desintegration</p>	<p>Fake fur after 21 days</p> <p>The sample looked intact, only discoloration due to the digestate was observed.</p>
 <p>MNI-1/2 Fake Fur after 4 weeks of desintegration</p>	<p>Fake fur at end (30 days)</p> <p>The sample looked intact, only discoloration due to the digestate was observed.</p>

5 Summary and conclusions

The biodegradation at $37^{\circ}\text{C} \pm 2^{\circ}\text{C}$ (mesophilic conditions) of test items Undyed mink fur, Undyed fox fur, Dyed mink fur, Dyed fox fur and Fake fur was tested together with cellulose as reference item in a high solids anaerobic digestion test, according to ISO 15985. The test was performed in duplicate and lasted 30 days. The biodegradation percentage is based on the net biogas production and carbon content of the test item. Furthermore, the disintegration of the test items was evaluated.

According to ISO 15985 the test is considered valid if a) the degree of biodegradation of the reference material is $>70\%$ after 15 days, and b) the deviation of the percentage of biodegradation for the reference item in the different vessels is less than 20% at the end of the test. After 15 days the biodegradation of cellulose was 84.1%. The final biodegradation (after 30 days) was $85.7\% \pm 2.0\%$, meaning that all requirements for a valid test were fulfilled.

Biodegradation of the Undyed mink fur, Undyed fox fur, Dyed mink fur and Dyed fox fur started immediately. The undyed mink fur shows the highest biodegradation so far (25.8%), followed by the dyed mink fur (18.3%), the undyed fox fur (13.6%) and the dyed fox fur (6.6%). Biodegradation reached a plateau for all test items. This means that these test items are partially biodegradable under mesophilic, high solids anaerobic conditions within 30 days.

Biodegradation of the fake fur never started. At the end of the test a biodegradation percentage of $0.3\% \pm 0.1\%$ was measured. The slightly positive result is considered due to natural variations in the biogas production of the inoculum. This means that the fake fur is not biodegradable under mesophilic, high solids anaerobic conditions.

In the disintegration test, it was observed that the Undyed mink fur, Undyed fox fur, Dyed mink fur and Dyed fox fur partially disintegrated: the skin fell apart and disappeared but the hairs remained. The fake fur did not show any disintegration, only discolouration.

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