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Compostability of pre-oxidized LDPE film containing P-Life prodegradant additive

(two appendixes)

Summary

P-Life 15 µm thick blown film was pre-oxidized in 10 days at 65 °C. The pre-oxidized material was then subjected for the subsequent test of biodegradability in a compost environment at 58 °C. The test of biodegradability was performed in accordance with ISO 14855-1:2005 "Determination of the ultimate aerobic biodegradability and disintegrability of plastic materials under controlled composting conditions — Method by analysis of evolved carbon dioxide". The result of the biodegradability test performed on the oxidised material was 43 % after 607 days in the compost environment and still increasing.

Commission

The commission included testing of LDPE film containing P-Life pro-degradant system. The goal was to evaluate bio-degradability of the thermally oxidized material under optimal composting conditions. The temperature and the period for thermal degradation were decided from the previous Arrhenius test. An appropriate amount of the film was pre-aged at 65 °C for 10 days, for the subsequent tests of biodegradability. The pre-aged material was characterized with regard to molecular weight by size exclusion chromatography (SEC). Biodegradability of the pre-aged material was followed using measurements of the amount carbon dioxide evolved.

Material

The test material was 15 µm thick blown film produced in May 2008, consisted of 99,7 % PELD and 0,3 % P-Life prodegradant additive designated SMC 2522. The material was sent by the commissioner and arrived at SP on May 26th 2008. The material was kept in a storage space at 3-5 °C until the tests.

Biodegradability

The ultimate biodegradability of the degraded material was examined under optimal conditions simulating an industrial compost, using the test procedure in accordance with ISO 14855. Method by analysis of evolved carbon dioxide was applied using Maihak S710 analyser equipped with Multor NDIR detector and measurements each 14 hours and 40 minutes. Through the composting vessels carbon dioxide free air was flown at the rate of 300 ml/min. Three parallel composting vessels (6 l desiccators) were used from the beginning for the oxidized material. However, after about two months one vessel was broken thus only two parallel vessels were used.

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Cellulose (Merck, microcrystalline powder for thin layer chromatography, Avicel, 11,0 g, containing 44,4 % C) was used as positive reference material in three vessels. The cellulose was added three times at three different occasions because of the rapid bioassimilation compared to the total test duration. Each time the cellulose was totally converted to carbon dioxide.

Three vessels were also used as blanks, containing the compost mixture but no test material.

The biodegradability tests were performed by Linda Eriksson and Catrin Lindblad.

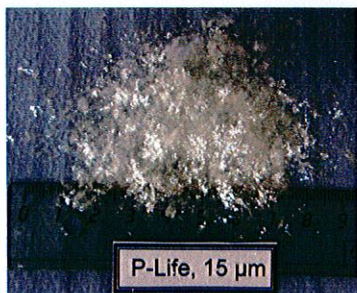


Image: Grinded material that was used in the biodegradability tests

Compost environment

Each vessel contained 1239 g of compost mixture from which about two thirds was water. The mixture consisted of 50 % mature compost and 50 % (by volume) expanded Vermiculite of concrete type as structural material (details about analyses are summarised in appendix 2). The test was performed in accordance with ISO 14855-1 along with the document of interpretation KMp T330-200A1 (see appendix 3) at 58 ± 2 °C. Before adding the test material, the compost mixture was pre-incubated at 58 ± 2 °C for 5 days in order to reduce the compost activity enough for the IR detector to be able to measure the development of carbon dioxide (measuring limit 5 000 ppm).

The mature compost was received from Sobacken industrial composting plant in Borås, Sweden the 8th of September 2008 and was according to the producer more than 3 months old.

The biodegradability test was conducted during the period 18th September 2008 until 25th May 2010.

Results

The molecular weight of the new and the pre-aged material is given in the table below.

| | Unaged | After 7 days at 65 °C | After 10 days at 65 °C |
|----|--------|-----------------------|------------------------|
| Mw | 131500 | 23000 | 8800 |
| Mn | 18300 | 2500 | 1700 |

The carbon content in the test material after 10 days at 65 °C was 79 %. The accumulated amount of CO₂ expressed as the percentage of the maximum theoretical value is presented in the Figure below.

The result of the biodegradability test after 607 days in the compost mixture at 58 °C was 43 % of the maximum theoretical value and still increasing. Details about analyses are summarised in appendix 1.

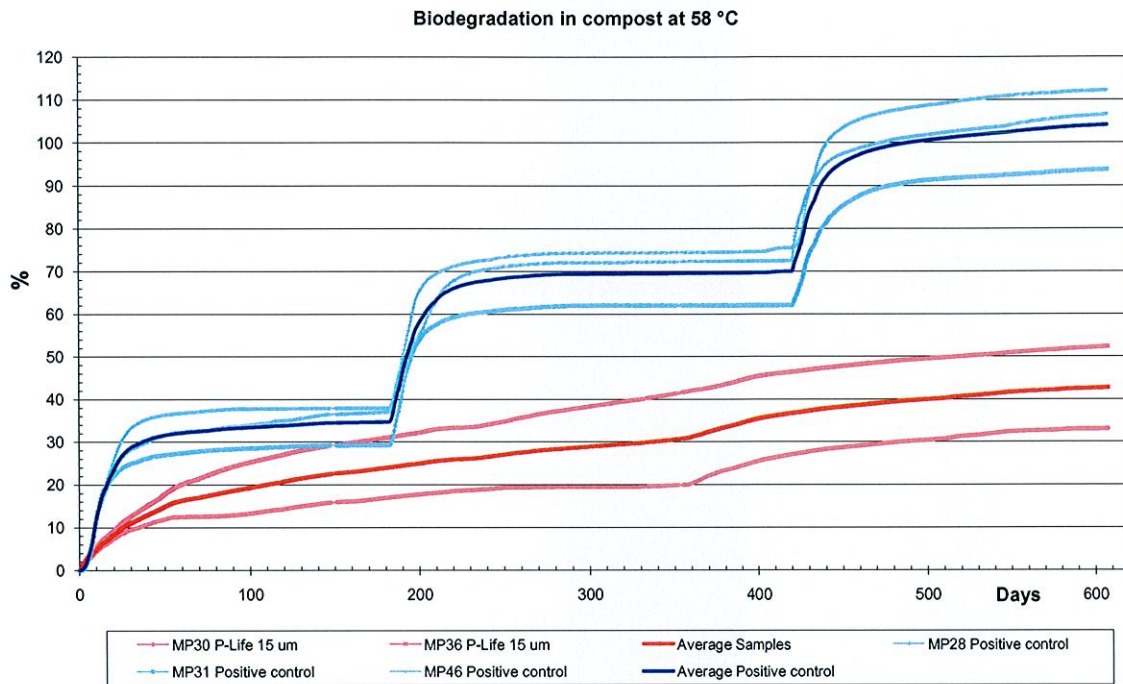


Figure. Amount of CO₂ expressed as percentage of the maximum theoretical value. The blue curves represent biodegradability of the microcrystalline cellulose.

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Technical specification for the biodegradability test in compost

Table 1. Results of analyses of the compost mixture

| Analysis | Mature compost | Vermiculite | Compost mixture |
|------------------------------------|----------------|-------------|-----------------|
| pH | 7,8 | | 7,9 |
| Dry content [weight-%]* | 44,2 | | 34,7 |
| C, [weight-%]* | 7,4 | | 5,2 |
| H, [weight-%]* | 6,9 | | 7,8 |
| N, [weight-%]* | 0,6 | | 0,41 |
| C/N-quotient* | 12,3 | | 12,7 |
| C, dry sample [weight-%] | 16,8 | | 14,9 |
| H, dry sample [weight-%] | 1,6 | | 1,7 |
| N, dry sample [weight-%] | 1,3 | | 1,2 |
| Volatile matter, 550 °C [weight-%] | 19,8 | | 15,2 |
| Dry matter/vessel [g] | 304 | 135 | 439** |

* the material condition as received

** the total weight including water was 1239 g

Table 2. Points of measuring and the sample weights at the beginning of the test

| Point of measuring | Total weight including vessel [kg] | Sample | Sample weight [g] |
|--------------------|------------------------------------|---------------------|-------------------|
| 30 | 4,5360 | P-Life 15 µm; D | 13,163 |
| 36 | 4,5765 | P-Life 15 µm; E | 12,098 |
| 41 | 4,5490 | P-Life 15 µm; F | 12,108 |
| 28 | 4,5380 | Positive control; D | 14,542 |
| 31 | 4,5660 | Positive control; E | 14,546 |
| 46 | 4,5180 | Positive control; F | 14,777 |
| 25 | 4,5245 | Blank control; D | - |
| 33 | 4,5595 | Blank control; E | - |
| 42 | 4,4940 | Blank control; F | - |

Appendix 2

Details about analyses and results of the biodegradability test after 180 and 607 days in compost mixture at 58 °C.

The sample No 41, "P-Life 15 µm; F" was broken by accident, which is the reason for the lack of results for this sample.

Table 1: Total weight of the vessels after 180 days of the test and pH after 180 and 607 days

| Sample | Total weight after 180 days [kg] | pH after 180 days | pH after 607 days |
|-------------------------------------|----------------------------------|-------------------|-------------------|
| P-Life 15 µm; D | 4,3390 | 6,6 | 6,4 |
| P-Life 15 µm; E | 4,3970 | 6,4 | 6,7 |
| P-Life 15 µm; F | - | - | - |
| Mean value: P-Life 15 µm | - | 6,5 | 6,6 |
| Positive control; D | 4,3515 | 7,5 | 6,9 |
| Positive control; E | 4,3800 | 6,5 | 7,2 |
| Positive control; F | 4,3760 | 6,7 | 6,9 |
| Mean value: Positive control | - | 6,9 | 7,0 |
| Blank; D | 4,3405 | 6,6 | 7,0 |
| Blank; E | 4,3725 | 6,7 | 6,6 |
| Blank; F | 4,3270 | 6,5 | 7,1 |
| Mean value; Blank | - | 6,6 | 6,9 |

Table 2: Calculated and measured amount of carbon dioxide
Biodegradability = (measured – blank)/theoretical

| Sample | Theoretical max CO ₂ [g] after | | Measured CO ₂ [g] after | | Biodegradability [%] after | |
|---------------------|---|-------------|------------------------------------|--------------|----------------------------|------------|
| | 180 days | 607 days | 180 days | 607 days | 180 days | 607 days |
| P-Life 15 µm; D | 35,23 | - | 41,0* | 80,1 | 17 | 33 |
| P-Life 15 µm; E | 35,04 | - | 45,9* | 86,9 | 31 | 52 |
| P-Life 15 µm; F | - | - | - | - | - | - |
| Mean value: | 35,1 | - | 43,44* | 83,5 | 24 | 43 |
| Positive control; D | 23,7 | 70,4 | 62,7* | 147,5 | 116 | 112 |
| Positive control; E | 23,7 | 72,6 | 56,3* | 134,7 | 90 | 94 |
| Positive control; F | 24,1 | 71,1 | 60,9* | 141,0 | 112 | 107 |
| Mean value: | 23,8 | 71,4 | 60,0* | 141,1 | 106 | 104 |
| Blank; D | - | - | 33,7 | 65,8 | - | - |
| Blank; E | - | - | 38,5 | 74,4 | - | - |
| Blank; F | - | - | 33,1 | 65,6 | - | - |
| Mean value; | - | - | 35,1 | 68,1 | - | - |

* Total amount including the contribution from the compost